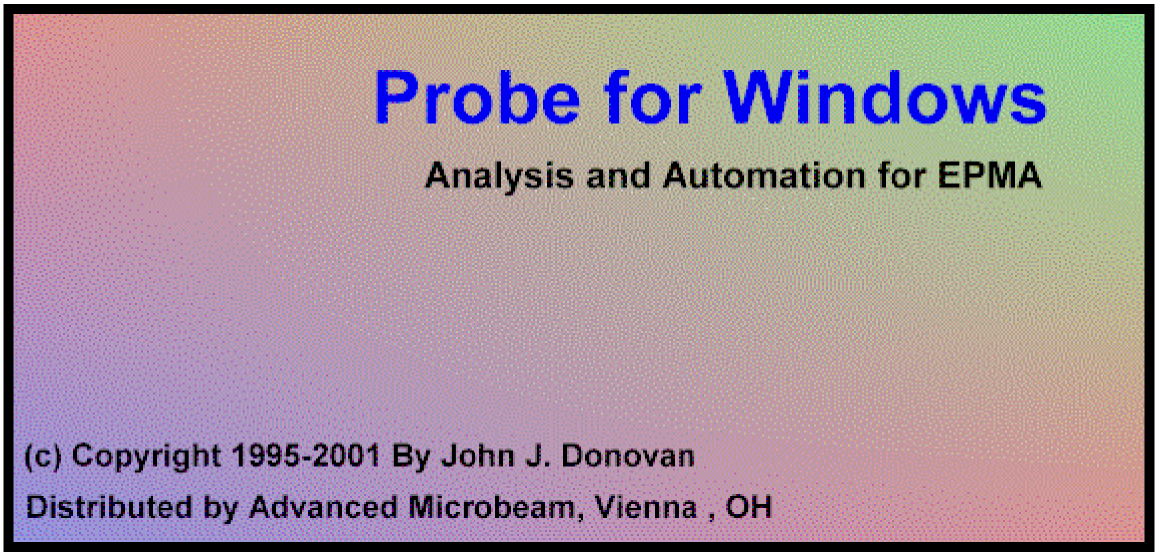
Probe for Windows 95/98/NT/2000 v. 5.39

User's Guide and Reference



Probe for Windows

Analysis and Automation for EPMA

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Distributed by Advanced Microbeam, Vienna , OH

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Introduction

Description

Probe for Windows NT is a complete 32 bit acquisition, automation and analysis package for WDS (wavelength dispersive) EPMA (Electron Probe Micro-Analysis) running under Windows NT (4.0 or higher), on the Intel Pentium, Pentium Pro or Pentium II platform. Probe for Windows can acquire, automate and analyze x-ray intensities on JEOL, ARL and Cameca microprobes with WDS spectrometers and stage motors.

Probe for Windows NT can also be run off-line to simply re-process previously acquired x-ray intensity data. Because Probe for Windows stores all standard, unknown and wavescan sample data and retains all experimental conditions in a single Microsoft Access relational database file (v. 3.5 Access DAO), it is extremely simple to transfer Probe for Windows NT intensity data to another computer for off-line reprocessing with another copy of Probe for Windows NT running in "demonstration" mode.

Probe for Windows is written primarily in Visual Basic 5.0 (Professional) for Windows and utilizes several external DLLs (Dynamic Link Libraries) for data access, automation and graphics written in C++ v. 4.0.

License

Probe for Windows NT has a flexible site license policy. You are allowed to use Probe for Windows on only one (1) computer for automation and data acquisition on an electron microprobe or SEM. However you may install Probe for Windows NT on as many computers as you require for the processing of off-line data from microprobe or SEM.

Probe for Windows NT requires specialized hardware for operation under the real-time interfaces, however no special hardware is required for operation in "demonstration" mode. Probe for Windows NT provides full functionality for quantitative analysis and graphical or ASCII file output in "demonstration" mode.

Special Thanks

The author would like to thank John T. Armstrong for his valuable contribution of the CITZAF quantitative matrix correction routines. Also Dan Snyder for help with the mathematical codes and Mark L. Rivers for his work on the quantitative interference and iterated MAN corrections and Dan Kremser and Tracy Tingle for extensive beta testing and John Friday and Brian Gaynor for help with the hardware interfacing code. Also Jim Eckert and Paul Carpenter for help with the serial interfacing. And most of all, my wife Barbara, for her patience and support throughout the development of this program.

Technical Support

Technical support for Probe for Windows is provided by Advanced MicroBeam, Inc. Please contact John Donovan or Don Lesher at (330) 394-1255 for any questions or difficulties you may have with Probe for Windows.

If an error message or software bug is seen, please note the exact error message and record as many details of the incident as you can to help us in determining a solution to your problem. Suggestions regarding new features or improvements to Probe for Windows are always welcome and every effort will be made to incorporate them in future releases. If you have an idea for a new feature, we would like to hear about it. Please write John Donovan at the address below or call anytime.

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Analysis Concepts

One significant difference between Probe for Windows and other microprobe analysis programs is that Probe for Windows treats all samples, regardless of whether they are standard or unknown samples, in exactly the same manner. This begins with the data acquisition; Probe for Windows can acquire a complete analysis for every standard sample, just as it would for an unknown sample.

For example, if in a particular run you are analyzing for Si, Al, Na and K, then by default, Probe for Windows can acquire data for all four elements on each standard. This means that even if a standard is only used to calibrate Si, the x-ray count data for the other three elements, Al, Na, K can also be acquired on that standard. Measuring all samples in the same manner under the same conditions can eliminate errors that may be introduced in the analysis from beam and sample interactions, especially when beam charge buildup, carbon buildup or volatile element effects are present in the samples.

Although the time needed to acquire standard data is not any longer for this method when the analysis is measuring only one element on each spectrometer, it is true that acquiring more than one element on a spectrometer will increase the time required for standard calibration. This might be considered a small price to pay for the increased accuracy and flexibility of measuring both standards and unknown for the same elements. However, to save time, program Probe for Windows does allow the acquisition of "quick" standards for those standards used in the primary calibration. Note that acquisition of these "quick" standards will restrict the use of some of the unique analysis features discussed below.

Aside from the obvious value in keeping the analysis conditions constant for all samples, having the complete data for each standard at hand, provides the analyst with several useful capabilities. Among these is a unique standard re-assignment feature. Since all elements in the run are acquired for all standards, any standard containing a particular element can be assigned as the calibration standard for that element at any time, even when processing off-line.

Another benefit to complete standard analysis is the ability to calculate each standard analysis as if it were an unknown. This means that every standard is also a secondary standard. In this way, one can easily determine the accuracy of each element in the run (and the overall accuracy of the run by noting the totals on each standard). If an element is present in a standard but is not assigned as the calibration standard for that element, then the program will calculate the relative error in the analysis based on the assigned standard. In this way one can check for agreement between various standards in the run. If the element is not present in the standard, the analysis provides a check on the accuracy of the off-peak or MAN background correction and also as a check for possible spectral interferences.

In fact, the quantitative interference correction in Probe for Windows uses this additional data to calculate the effect of an interfering element on an interfered channel. Again, since all elements are acquired for each standard and the composition of each standard is of course already known, the count to the concentration ratio of the interfering element can be calculated. This in turn can be used in the ZAF composition iteration to quantitatively calculate the interference correction in an unknown sample (Donovan, et. al., 1993).

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Installation

Platform Requirements

Probe for Windows requires a fairly fast computer platform for reasonable calculation and data access times. The following system requirements are the minimum and preferred hardware that can be used to run Probe for Windows NT.

Minimum

- Windows NT (v. 4.0, Service Pack 4)
- 200 Mhz Pentium CPU
- 2 GB hard disk with at least 100 MB free disk space
- 32 MB RAM
- mouse
- 3.5 floppy drive
- 17" color monitor (1024 x 768 x 256 colors)

Preferred

- Pentium II 300 MHz or faster CPU
- 8 GB hard disk with at least 200 MB free disk space
- 128 MB RAM
- mouse
- 3.5 floppy drive and Internet connection
- 21" color monitor (1280 x 1024 x 64K colors)

Installation

Be sure that you have at least 100 MB of free disk space on the installation hard drive. The Probe for Windows installation requires less than this, but the run time files require additional space for the creation of data and table files.

If attempting to perform a "clean" installation, be sure to remove any existing copies of Probe for Windows by running the Add/Remove Software applet from the Windows Control Panel. In addition, be certain to check that the USERDATA sub-directory is not present. Probe for Windows NT uses the presence of this directory to determine if it has already been installed or not.

If updating an already existing installation using the ZIP files, Probe for Windows will only replace the executable and certain internal data files in the C:\Program Files\Probe for Windows directories. Always backup your user/site specific files: STANDARD.MDB, PROBEWIN.INI, MOTORS.DAT, SCALERS.DAT, ELEMENTS.DAT, CRYSTALS.DAT and any other data files that you have created or modified since the original installation.

Installation Over The Network (FTP)

To install Probe for Windows NT from the network, first confirm that your computer is networked properly to the Internet by running Netscape or Internet Explorer.

Then using either Netscape, Explorer or other FTP software, connect to ftp://www.advancedmicrobeam.com and change to the pub/users/advancedmicrobeam directory.

Download the following file to a temporary directory on your hard disk:

PFW-32_Dist.ZIP

Using Pkunzip or compatible utility, extract the files from the .ZIP file. A password may be required to extract the installation files. Then, from the Windows Start button, click the Run menu and type in the text box "c:\temp\:setup" (or appropriate directory name containing the downloaded files) and hit <enter>.

The Probe for Windows setup program will run and prompt you for the installation directory. Accept the default "C:\Program Files\Probe for Windows" or type another directory name for the Probe for Windows installation. The setup program will confirm the installation when it is complete. If any errors messages are noted during the installation, please note the exact error message text and call or e-mail for technical support.

Upgrade Over The Network (FTP)

To upgrade Probe for Windows after the installation is complete, download the following file from the Advanced Microbeam FTP site:

PFW-32_Update.ZIP

Using Pkunzip or compatible utility, extract the files from the .ZIP file. Then copy the new files to the Probe for Windows folder (usually C:\Program Files\Probe for Windows).

Files Used by Probe for Windows NT:

The following files are installed by the Probe for Windows Setup program. The following section details where they are ultimately installed and what they are used for. Note that all files that are not installed into the C:\WINNT\SYSTEM32 directory are installed in the specified Probe for Windows installation directory (usually C:\Program Files\Probe for Windows).

Files installed to the Windows SYSTEM32 sub directory (usually C:\Windows\System for Win95/98 or C:\Winnt\System32 for WinNT/2000) :

MSVBVM50.dll	' Microsoft VB5 support file
StdOle2.tlb	' Microsoft OLE support file
OleAut32.dll	' Microsoft OLE support file
OlePro32.dll	' Microsoft OLE support file
AsycFilt.dll	' Microsoft support file
Ctl3d32.dll	' Microsoft support file
ComCat.dll	' Microsoft support file
Grid32.ocx	' Microsoft grid control
MFC40.dll	' Microsoft support file
MSVCRT40.dll	' Microsoft support file
COMDLG32.OCX	' Microsoft common dialog control
GRAPHS32.OCX	' Graphics Server graph control
Spin32.ocx	' Microsoft spin button control
Gauge32.ocx	' Microsoft gauge control
MSCOMM32.OCX	' Microsoft Communication control

DAO350.DLL	' Microsoft 3.5 DAO
MSJtEr35.dll	' Microsoft 3.5 DAO support file
MSJInt35.dll	' Microsoft 3.5 DAO support file
GSWDLL32.DLL	' Graphics Server support file
MSJet35.dll	' Microsoft 3.5 DAO support file
VBAJet32.dll	' Microsoft 3.5 DAO support file
VB5DB.dll	' Microsoft 3.5 DAO support file
MSRD2x35.dll	' Microsoft 3.5 DAO support file
MsRepl35.dll	' Microsoft 3.5 DAO support file
ODBCJt32.dll	' Microsoft 3.5 DAO support file
GSWAG32.DLL	' Graphics Server support file
GSW32.EXE	' Graphics Server support file
GSJPG32.DLL	' Graphics Server support file
GRAPHPPR.HLP	' Graphics Server support file
JoyStk32.OCX	' Joy Stick control

Files that are installed to the Probe for Windows application directory (usually C:\Program Files\Probe for Windows) :

EMPAPF.DAT	' empirical APF factors ASCII file
EMPMAC.DAT	' empirical MACs ASCII file
EMPFAC.DAT	' empirical alpha-factors ASCII file
EMPPHA.DAT	' empirical PHA ASCII file
XEDGE.DAT	' x-ray absorption edge data binary file
XFLUR.DAT	' x-ray fluorescence yield data binary file
XLINE.DAT	' x-ray emission line data binary file
ABSORB.DAT	' McMaster MAC coefficients ASCII file
CITZMU.DAT	' Cal-Tech (Heinrich) MACs data binary file
LINEMU.DAT	' Henke MACs data binary file
MCMASTER.DAT	' McMaster MACs data binary file
MAC30.DAT	' Caltech MACs data binary file
XRAY.MDB	' NIST x-ray database ASCII file
PROBEWIN.INI	' JEOL INI file (sample)
MOTORS.DAT	' JEOL MOTOR file (sample)
SCALERS.DAT	'JEOL SCALER file (sample)
CRYSTALS.DAT	' crystal data
ELEMENTS.DAT	' element data
STANDARD.MDB	' default standard composition database
STANDARD.EXE	' Standard database application
PROBEWIN.EXE	' Probe for Windows main application
PROBEWIN.HLP	' Probe for Windows help file
VERSION.TXT	' ASCII file of version changes
GRAPHPPR.HLP	' plot graphics window help file
INSTALL.WRI	' installation notes (Windows Write format)
PROBEHLP.INI	' context sensitive help INI file
STARTWIN.EXE	' Start utility application
JOYWIN.EXE	' Joystick utility application
TESTTC.EXE	' Counter-timer test utility application
USERWIN.EXE	' User database application
STAGE.EXE	' Stage utility application
COAT.EXE	' Coating utility applet
TECH1.DOC	' technical document (Word 97)
TECH2.DOC	' technical document (Word 97)
TECH3.DOC	' technical document (Word 97)
GRIDBB.BAS	' partial OLE script used for creating new scripts (Surfer 6)

GRIDCC.BAS	' partial OLE script used for creating new scripts (Surfer 7)
TEST3D.DAT	' sample polygon for polygon grid
JEOL1.WMF	' sample JEOL stage bitmap
CALCZAF.EXE	' ZAF/Phi-Rho-Z calculation application
DRIFT.EXE	' Standard intensity drift utility
LINES2.DAT	' line and edge energies used for creating MAC30.DAT
DEMO1.BMP	' Demo image for Image Digitize feature
MAC30.DAT	' MAC30 mass absorption coefficients

NIST MQ executable and source (Probe for Windows application directory)

MQ.EXE	' NIST Monte Carlo software executable
EDGES.TSR	' NIST Monte Carlo line table
MCARLO.BAT	' NIST Monte Carlo batch file for input
MCARLO.INP	' NIST Monte Carlo sample input file
CDONLY.DAT	' NIST Monte Carlo data file for MCARLO.BAT
MQ.FOR	' NIST Monte Carlo FORTRAN SOURCE
BLOCK1.CMN	' NIST Monte Carlo FORTRAN SOURCE
BLOCK2.CMN	' NIST Monte Carlo FORTRAN SOURCE
BLOCK3.CMN	' NIST Monte Carlo FORTRAN SOURCE
BLOCK4.CMN	' NIST Monte Carlo FORTRAN SOURCE
BLOCK5.CMN	' NIST Monte Carlo FORTRAN SOURCE
BLOCK6.CMN	' NIST Monte Carlo FORTRAN SOURCE
BLOCK7.CMN	' NIST Monte Carlo FORTRAN SOURCE
BLOCK8.CMN	' NIST Monte Carlo FORTRAN SOURCE
ATWT.CMN	' NIST Monte Carlo FORTRAN SOURCE
ELNAM.CMN	' NIST Monte Carlo FORTRAN SOURCE

Files that are installed to the Probe for Windows application directory but also copied to the Userdata directory (usually C:\Program Files\Probe for Windows\UserData) :

DHZ.DAT	' standard compositions from DHZ (1st Ed.)
ORE.DAT	' standard compositions from Dana's
SRM.DAT	' standard compositions from NIST SRMs
CALCZAF.DAT	' ZAF/Phi-Rho-Z sample data
GRIDMIN.DAT	' sample boundary data for demonstration
JEOLOX.MDB	' Probe for Windows sample data (oxide)
JEOLEL.MDB	' Probe for Windows sample data (elemental)
AUAGCU2.DAT	' CalcZAF binary intensity data file
NISTBIN.DAT	' CalcZAF binary intensity data file
POUCHOU.DAT	' CalcZAF binary intensity data file
XYSCAN.BAS	' sample Surfer script (run from GSScripter) (Surfer 6)
XYSCAN.BLN	' sample Surfer boundary file (Surfer 6)
XYSCAN.DAT	' sample Surfer data file (Surfer 6)
XYSCAN2.BAS	' sample Surfer script (run from GSScripter) (Surfer 7)
XYSCAN2.BLN	' sample Surfer boundary file (Surfer 7)
XYSCAN2.DAT	' sample Surfer data file (Surfer 7)
NISTBINA20.DAT	' Sample binary calculation input for CALCZAF
NISTBINZ10.DAT	' Sample binary calculation input for CALCZAF
POUCHOUA20.DAT	' Sample binary calculation input for CALCZAF
POUCHOUZ10.DAT	' Sample binary calculation input for CALCZAF
CALCBIN.DAT	' Sample calculation input for CALCZAF

Installed by IOMapper Setup (usually installed to the System or System32 folder) :

DCC20Z.DLL	' counter-timer interface DLL

PHA_32.DLL	' PHA interface DLL
SX50_32.DLL	' SX50 interface DLL
SX-NULL.DLL	' SX50 null test DLL
SX-PC11.DLL	' SX50 PC11 board DLL
SX-DC13.DLL	' SX50 DC13 board DLL
SXMON.EXE	' SX50 communication monitor program
IOMAPPER.DLL	' I/O port interface for Windows NT
IOMAPPER.SYS	' I/O port interface driver for Windows NT

Files that are created by Probe for Windows:

POSITION.MDB	' position database
SETUP.MDB	' element setup database
USER.MDB	' user database
STANDARD.DAT	' standard composition import/export ASCII file
SETUP.DAT	' element setup import/export ASCII file
USER.DAT	' user database import or export ASCII file
*.POS	' position import or export ASCII files
*.OUT	' Log window ASCII files

Running Probe for Windows for the First Time

For a first time installation and before modifying the microprobe configuration files, it is **required** that the program be run first in it's default "demonstration" mode. When Probe for Windows is run the first time, the program checks for the presence of the USERDATA sub-directory and creates if it is not present. The program then copies necessary configuration files into this directory.

Simply double-click the Probe for Windows icon to allow the program to configure these and any other necessary directories and files that it will need for analytical calculations and make sure that all of the analytical files are installed. Several sample data files are supplied to test the default installation.

Once this step is completed, the configuration of the hardware specific configuration files (PROBEWIN.INI, MOTORS.DAT and SCALERS.DAT) may be performed at this time using a text editor such as NotePad or TextPad. See below, under the Configuration Files section.

Note however, that to use the JOYWIN.EXE joystick control program, the joystick driver file must be installed into the Drivers section of the Windows Control Panel if it is not already (except for SX50 installations).

In addition, the PMC DCX motor controller driver disk for Windows must also be installed according to it's installation directions for servo motor motion, faraday cup control and crystal flipping (except for SX50, Tracor and Sesame installations). Contact Advanced MicroBeam for help with these modifications if necessary.

If the Advanced Microbeam interface is installed, the IOMAPPER driver must also be installed, according to it's instructions, for PHA and counter-timer interfacing. Contact Advanced MicroBeam for help with these modifications if necessary.

Also, if the SX50 (InterfaceType=6) interface is selected, the DRV11 interface boards and device drivers must be installed and configured. Contact Advanced MicroBeam for help with these modifications if necessary.

Removing Probe for Windows

To properly uninstall Probe for Windows NT, it is highly recommended to use the Add/Remove Programs applet in the Control Panel. This will insure that any "shared" components are properly removed from the system. Some files created by the user may need to be removed manually after the uninstall. Please contact Advanced Microbeam if necessary for more information.

Copy Protection

Probe for Windows has no copy protection for off-line processing of data or for running the program in "demonstration" mode. Please see the License Agreement section above for details on how you may copy Probe for Windows NT.

However, to run Probe for Windows under any real-time interface mode (See InterfaceType in the PROBEWIN.INI file) other than demonstration mode requires both a registration code and special hardware, only available from Advanced Microbeam.

The first time you run the software and make a connection to your microprobe the program will prompt you with a serial number and request a corresponding registration code. This registration code is available from Advanced Microbeam by supplying the displayed serial number. Once you obtain the registration code from Advanced Microbeam, simply enter it in the text field and you will never be prompted again.

If you get the error: "Error returned from DCC20AX_MasterReset (error code = 273)" then either your special hardware is not properly configured or it is not present and cannot therefore be run is any mode other than the demonstration interface (or off-line reprocessing mode).

Configuration Files

There are several configuration file used by Probe for Windows and it's companion programs. They are all editable ASCII files and may be edited (carefully!) by the user for customizing the microprobe configuration by using any text editor such as Windows NotePad. Please contact Advanced MicroBeam for help with these procedures if necessary.

The following files are used by Probe for Windows for configuration purposes and read in the order listed :

PROBEWIN.INI
CRYSTALS.DAT
ELEMENTS.DAT
MOTORS.DAT
SCALERS.DAT
DETECTORS.DAT

The PROBEWIN.INI file and other Probe for Windows configuration files ***must*** be in the C:\Program Files\Probe for Windows directory (or whatever directory Probe for Windows was installed to). These files will be automatically installed in the correct directories by the setup program.

Upgrading from 16 bit Probe for Windows

Configuration Files

If upgrading from the 16 bit Probe for Windows (version numbers less than 4.00), one may simply copy the old configuration files over to the new 32 bit Probe for Windows directory (usually C:\Program Files\Probe for Windows). Note that the 32 bit version of Probe for Windows does not use an XrayData directory to store the configuration files. All configuration files are stored in the program directory.

All 16 bit configuration files are backward compatible. Files that may be copied over are:

PROBEWIN.INI
CRYSTALS.DAT
ELEMENTS.DAT
MOTORS.DAT
SCALERS.DAT

Note that some new fields may have been added to some configuration files to provide default initialization for new features. Please carefully check the following documentation for parameters that may not be present in the previous configuration files.

Standard Database File

Standard database composition file(s) from the 16 bit version of Probe for Windows must be first exported to ASCII format using the File | Export menu item in the 16 bit Standard for Window application. The default export file is named STANDARD.DAT.

This file should be copied to the new Probe for Windows (32 bit) directory (usually C:\Program Files\Probe for Windows) and then imported using the File | Import menu item in the 32 bit Standard for Windows application.

Element Setup Database File

The element setup database file(s) from the 16 bit version of Probe for Windows must be first exported to ASCII format using the Export to ASCII File button in the Setup database dialog in the 16 bit Standard for Window application. The default export file is named SETUP.DAT.

This file should be copied to the new Probe for Windows (32 bit) directory (usually C:\Program Files\Probe for Windows) and then imported using the Import button in the Setup database dialog in the 32 bit Standard for Windows application.

Position Database File

The 16 bit position database is incompatible with the 32 bit version of the Probe for Windows software. To transfer your digitized standard position files, simply copy all ASCII *.POS files to the new 32 bit Probe for Windows directory (usually C:\Program Files\Probe for Windows) and then import as usual from the 32 bit application.

Windows Metafiles

All windows metafiles are compatible with the 32 bit version of Probe for Windows. Simply copy all *.WMF files to the new 32 bit Probe for Windows directory (usually C:\Program Files\Probe for Windows).

Userlog Database File

The Userlog database file from the 16 bit version of Probe for Windows must be first exported to ASCII format using the File | Export menu item in the 16 bit UserLog for Window application. The default export file is named USER.DAT.

This file should be copied to the new Probe for Windows (32 bit) directory (usually C:\Program Files\Probe for Windows) and then imported using the File | Import menu item in the 32 bit Userlog for Windows application.

Probe Database Files

Probe for Windows 16 bit database files are backward compatible with the 32 bit version of Probe for Windows. However, for maximum performance, rather than append data to an existing 16 bit database, it is recommended that first a new 32 bit database be created and then the run configuration be loaded using the File Setup button from the New Sample dialog.

Constants for array declarations

Some configuration file parameter allowable ranges are defined using the following constant declarations.

```
MAXCHAN% = 32              ' maximum elements per run
MAXCHAN1% = MAXCHAN% + 1   ' maximum elements plus 1
MAXSTD% = 48               ' maximum standards per run
MAXROW% = 50               ' maximum lines per sample
MAXRAY% = 6                ' maximum x-ray symbols (ka,la,ma, , , )
MAXEDG% = 9                ' maximum edge symbols
MAXELM% = 94               ' maximum elements
MAXEMP% = 20               ' maximum empirical MAC/APFs
MAXSAMPLE% = 2000          ' maximum samples per run
MAXINTF% = 4               ' maximum interferences per element
```

```
MAXAXES% = 4                ' maximum stage axes
MAXINDEX% = 1000            ' maximum standards per standard database
MAXMAN% = 16                ' maximum MAN assignments per element
MAXSET% = 30                ' maximum sets for drift correction
MAXSPEC% = 9                ' maximum spectrometers (fixed + tunable) per run
MAXSCAL% = 9                ' maximum scaler channels per run
MAXCRYS% = 6                ' maximum crystals per spectrometer
MAXCRYSTYPE% = 32           ' maximum crystal types
MAXMOT% = MAXSPEC% + 4      ' maximum motors
MAXSTDPOS% = 99             ' maximum STDPOS*.POS file names
MAXDIM% = 3                 ' maximum number of dimensions for matrix transformation
MAXCOEFF% = 3               ' maximum number of linear fit coefficients
MAXTUNABLE% = 6             ' maximum number of tunable spectrometers
MAXBITMAP% = 12             ' maximum number of stage bit map files
MAXLINE% = 32000            ' maximum number of data lines per run
MAXBIN% = 5                 ' maximum binary pairs for alpha factor calculations
MAXEMPFAC% = 100            ' maximum empirical alpha factors (from EMPFAC.DAT)
```

PROBEWIN.INI

The main configuration file used by Probe for Windows is PROBEWIN.INI. This file is located in the Probe for Windows directory (usually C:\Program Files\Probe for Windows). This is a "private" windows initialization file used only by Probe for Windows and it's companion programs. Note that Probe for Windows ***does not*** modify the Windows WIN.INI or SYSTEM.INI files. No changes are required to the AUTOEXEC.BAT or CONFIG.SYS files either. A number of "shared" components are installed to the C:\Winnt\System32 directory and they are automatically registered by the setup program. All single value parameters (e.g., the number of stage motors) used by Probe for Windows and it's companion programs are specified in this PROBEWIN.INI file.

The PROBEWIN.INI file has a standard Windows .INI file structure. That is, it consists of several distinct sections designated by a section name enclosed in square [ ] brackets. Each section then contains one or more distinct keywords to specify various single value probe configuration values. These values can be integer or string values. Strings need to be enclosed in double quotes if the string contains blanks. Real number values are read as strings and then converted to single or double precision real numbers. Booleans (true or false) values are read as integer values where 0 is false and any non-zero value is true.

The following section describes each section and the keyword definitions for each. The allowable range and the default value loaded if the keyword is not found in the file is given for each keyword.

[General]

```
KiloVolts=15
TakeOff=40
BeamCurrent=30.
BeamSize=2
Magnification=400
OxideOrElemental=2     ; 1 for oxide or 2 for elemental
PeakCenterMethod=1     ; 0 for interval, 1 for parabolic, 2 for ROM based
DebugMode=0
UserName="Probe User"
Title="Probe for Windows NT Demonstration Run for JEOL Hardware"
Description="By John Donovan and Advanced Microbeam, Inc."
FileViewer="NOTEPAD.EXE"
CustomLabel1="Department"
CustomLabel2="Account #"
CustomLabel3="Group"
CustomText1="Analytical Chemistry"
CustomText2=""
CustomText3="Microscopy"
SMTPServerAddress="advancedmicrobeam.com"
```

SMTPAddressFrom="jdonovan@advancedmicrobeam.com"
SMTPAddressTo=jdonovan@advancedmicrobeam.com
UseWavFileAfterAutomationString=""

KiloVolts=15
This keyword specifies the typical operating voltage of the microprobe. This value will be used as a default value for initialization. The default value can be changed at any time during the data acquisition process from the Acquire! window in Probe for Windows or from the Analytical | Operating Conditions menu item in Standard for Windows. The allowed kilovolt range is 1 to 100 KeV. The default value is 15 KeV.

If the OperatingVoltagePresent flag is true then the software will attempt to set the operating voltage through the software interface when the first data acquisition is performed.

TakeOff=40.0
This keyword specifies the actual x-ray take-off angle of the microprobe. This value can be changed during the data acquisition process, but is generally not modified. However for modeling various experimental effects, this value can be changed from the Analytical | Operating Conditions menu item in Standard for Windows. Typically JEOL and Cameca = 40 and ARL = 52.5. The allowed takeoff range is 1 to 90 degrees. The default value is 40 degrees.

BeamCurrent=30. ! SX-50/51/100 only
This keyword is used to determine the default beam current in nano-amps. This parameter is used simply for documentation purposes unless the hardware interface supports beam current control as specified by the BeamCurrentPresent keyword in the [Hardware] section below. The allowed beam current range is 0.1 to 1000 nA. The default value is 30 nA.

BeamSize=2 ! SX-50/51/100 only
This keyword is used to determine the default beam size in microns. This parameter is used simply for documentation purposes unless the hardware interface supports beam size control as specified by the BeamSizePresent keyword in the [Hardware] section below. The allowed beam size range is 1 to 1000 u. The default value is 2 u.

For the SX50 interface (InterfaceType=6) this parameter will force the instrument into FIX mode and set the beam size using the SIZE command. In this mode the instrument beam scanning is disabled. Therefore if beam scanning is desired, the beam size should be set to zero so that a SIZE OFF command is sent to the instrument which will enable the beam scanning.

Magnification=400 ! SX-50/51/100 only
This parameter is used to set the default magnification for use with the Digitize Image window. The default is 400 and the allowed range is 10 to 10000.

OxideOrElemental=2
This keyword is used to determine the default option for oxide calculations (oxygen calculated by stoichiometry based on the element cation ratios for each sample) for starting the first sample of a new Probe for Windows run. It can be changed at any time during the acquisition or analysis process by the user. For default elemental analysis enter 2, for default oxide analysis, enter 1. The default value is 2 for elemental analysis.

PeakCenterMethod=1
This keyword is used to determine the default peak center method used by the program. The valid values are :

- 0 for interval halving
- 1 for parabolic peak fit
- 2 for ROM based peak center **! SX-50/51/100, Sesame and TRACOR only**

The default is the parabolic peak fit.

DebugMode=0

This keyword specifies if the program is to start up in the so called "debug" mode. This option may be useful for certain trouble-shooting situations. The Probe for Windows program will always save a user data file with DebugMode turned off. Change this parameter in the PROBEWIN.INI to any non-zero value to force the program to start up in "debug" mode for trouble-shooting purposes. The default is 0 to not start in debug mode.

UserName="Probe User"
This keyword specifies the default user name for the file information table in the various Probe for Windows database files. It may be modified for a maximum character length of 64 characters. The default value is "User Name".

Title="Probe for Windows Demonstration Run"
This keyword specifies the default run title for the file information table in the various Probe for Windows database files. It may be modified for a maximum character length of 64 characters. The default value is "File Title".

Description="By John Donovan and Advanced MicroBeam, Inc."
This keyword specifies the default file description for the file information table in the various Probe for Windows database files. It may be modified for a maximum character length of 64 characters. The default value is "File Description".

FileViewer="NOTEPAD.EXE"
This keyword is used to determine the default file viewer that is to be run to view the disk log file. The default file viewer is Windows NotePad. Note, that a more robust file viewer such as TextPad or Word for Windows may be substituted instead. TextPad is a shareware Windows text editing program that is available any many FTP sites. Please contact Advanced MicroBeam for help in obtaining TextPad for Windows.

CustomLabel1="Department"
CustomLabel2="Account #"
CustomLabel3="Group"
This keyword is used to define the first custom database field label in the user database application. These fields are also visible in the File Information window for each Probe for Windows application database. The defaults are "Department", "Account #" and "Group".

CustomText1="Analytical Chemistry"
CustomText2=""
CustomText3="Microscopy"
This keyword is used to define the defaults for first, second and third custom database fields in the user database application. These fields are also visible in the File Info window for each Probe for Windows application database. The defaults are blank.

SMTPServerAddress="advancedmicrobeam.com"
SMTPAddressFrom="jdonovan@advancedmicrobeam.com"
SMTPAddressTo="jdonovan@advancedmicrobeam.com"
These keywords are to be used for e-mail notification of real-time automation errors. If these SMTP fields are valid and the option "E-mail Notification of Errors" is checked in the Automation Options dialog, then any real-time errors (for example, a blown filament message) will be trapped and forwarded to the address indicated. This option is only available for automation procedures initiated from the Automate! window.

In addition, a "progress report" message will be sent every 8 hours to the e-mail address specified in order to give positive notification of the normal functioning of the instrument during especially long runs.

UseWavFileAfterAutomationString=""
This parameter specifies a .WAV audio file to be played after automation in ProbeWin or Stage has been completed. If the .WAV file is located in the application folder or a folder in the system path then the filename only can be specified. If the file is located in another folder, the complete path must be designated.

[Software]

```
LogWindowFontName="Courier New"
LogWindowFontSize=10
AcquirePositionFontSize=9
AcquireCountFontSize=10
LogWindowInterval=0.5
RealTimeInterval=0.2
AutomateConfirmDelay=10.0
EnterPositionsRelative=1
UpdatePeakWaveScanPositions=1
MaxMenuFileArray=4
ExtendedFormat=0
MACTypeFlag=1
PositionImportExportFileType=2
DeadtimeCorrectionType=1
AutoFocusStyle=1
AutoFocusInterval=5
BiasChangeDelay=2.0
UseEmpiricalPHADefaults=0
KilovoltChangeDelay=5.0
BeamCurrentChangeDelay=5.0
BeamSizeChangeDelay=5.0
LogWindowBufferSize=29000
CommandPacingInterval=0
PeakOnAssignedStandards=0
PrintAnalyzedAndSpecifiedOnSameLine=0
NoMotorPositionLimitsChecking=0
UserDataDirectory=""
ExtendedMenu=0
AutoAnalyze=0
StatusAlwaysOnTop=0
FaradayAlwaysOnTop=0
ColumnConditionChangeDelay=5.0
SurferOutputVersionNumber=6
SelPrintStartDoc=1
UseMultiplePeakCalibrationOffset=0
```

LogWindowFontName="Courier New"
Enter the default font for use in the log window. Generally a fixed spacing (not proportional) font such as Courier or New Courier should be used for best results. The font must be already be installed in Windows. The log window font can also be changed from the Output | Log Window Font menu item in all programs. The default font name is "Courier New".

LogWindowFontSize=10
Enter the default font point size for use in the log window. If the actual point size is not available, Windows will use the nearest available font size. The log window font can also be changed from the Output | Log Window Font menu item. The allowable range is 6 to 32. The default font size is 10.

AcquirePositionFontSize=12
Enter the font size for the Acquire Window motor position display. Generally use the largest font that will allow the motor positions to fill the display area without wrapping to the next line. The allowable range is 6 to 32. The default position font size is 12.

AcquireCountFontSize=12

Enter the font size for the Acquire Window time and counter display. Generally use the largest font that will allow the timer and counter values to fill the display area without wrapping to the next line. The allowable range is 6 to 32. The default count font size is 12.

LogWindowInterval=0.5
Enter the time interval in seconds for the log window buffer to be updated. The allowable range is 0.1 to 10 seconds. The default value is 0.5 seconds.

RealTimeInterval=0.2
Enter the time interval in seconds for the microprobe position and counter displays to be refreshed. The allowable range is 0.1 to 10 seconds. The default is 0.2 seconds.

Generally, slower interfaces such as serial interfaces are not polled repeatedly and therefore use a RealTimeInterval of 0.5 seconds or so. This also applies to the Cameca Microbeam and SX50 interfaces (InterfaceType=5 and 6).

AutomateConfirmDelay=10.0
Specifies the amount of time in seconds to wait after removing the faraday cup before starting the sample x-ray count acquisition. Generally used for microprobes equipped with a beam current aperture to allow time for beam stabilization or stage x, y, z position adjustment. The allowable range is 0 to 100 seconds. The default value is 10 seconds.

EnterPositionsRelative=1
Specifies whether the Element Setup dialog uses relative or absolute off-peak position entry as a default. Specify 1 for relative off-peak position entry for the default. The default value is 0 for absolute off-peak position entry.

UpdatePeakWavescanPositions=0
Specifies whether the peak center routine updates the peakscan and wavescan limits (in addition to the off-peak positions) after a peak center. Specify 1 to have the program to update the peakscan and wavescan limits after a peak center. The default value is 0 for no update.

ExtendedFormat=0
Specifies whether the default log window output is extended format mode (more than 8 elements per row) or not. The default value is false for 8 elements per row of output. This mode is best for portrait mode printing, but uses additional vertical screen space.

MaxMenuFileArray=4
Specifies the number of previously opened Probe for Windows files that appear on the File menu. Simply click the file name in the file list and the file will be opened.

MACTypeFlag=1
Specifies the default MAC (mass absorption coefficient) file used in the matrix calculations. Note that the MCMASTER.DAT file does not contains values for emitting energies below 1 KeV.

LINEMU.DAT (Henke < 10 KeV) = 1
CITZMU.DAT (Heinrich, Armstrong) = 2.
MCMASTER.DAT McMaster (from Rivers) = 3
MAC30.DAT Heinrich (fit to Goldstein table) = 4
MACJTA.DAT (Armstrong MACCALC program) = 5

PositionImportExportFileType=1
Specifies the default position file import and export format. There are two types (type = 1 or type = 2) used by Probe for Windows and it's companion programs.

Type = 1 format is the original (default) format specified in the Import or Export Position Samples section below.

Type = 2 format is a new import/export position file format that contains two additional integer and a string field which allows the analytical sample setup number and digitized autofocus flag and file setup name to be specified. Note that the sample setup number and the file setup name should be the same for all positions in a single position sample. To convert an original format .POS file (type=1) to the new format (type=2), simply add two columns of zeros and a column of double quotes to the file using a text editor or spreadsheet program (comma, space or tab delimited).

If it is intended to use an off-line stage for digitizing of analysis positions, it is recommended that the operator use the type = 2 format for standard, unknown and wavescan digitizing with sample setup numbers or file setup names. The only valid values for this parameter are 1 or 2.

DeadtimeCorrectionType=1
Specifies the default deadtime correction type. There are two types supported, the normal expression (type = 1) and a high precision variation (type = 2) for use when the count rate exceeds 50K cps. The default expression is the normal expression. The only valid values for this parameter are 1 or 2.

Use the high precision deadtime expression when the instrument utilizes an electronically forced deadtime. In this case the deadtime is truly a constant parameter and can be experimentally determined to two or even three significant digits, allowing the precision deadtime expression to be used to significant advantage.

AutoFocusStyle=1 ! SX-50/51/100 only
Specifies the default auto focus style used during automated acquisitions. This option is ignored if the AutoFocusPresent parameter in the [Hardware] section is not set. There are several varieties for the auto focus style, as shown in the following list:

1 = auto focus on a new position sample only
2 = auto focus on every point
3 = auto focus using the digitized auto focus flags
4 = auto focus using the specified auto focus interval

AutoFocusInterval=5 ! SX-50/51/100 only
Specifies the default auto focus interval used during automated acquisitions if the auto focus feature is selected and the interval auto focus option is selected. This option is ignored if the AutoFocusPresent parameter in the [Hardware] section is not set. The default is an auto focus interval of 5 which means that the program will attempt an auto-focus every 5 positions during an automated acquisition.

BiasChangeDelay=2.0
Specifies the default time delay used when a change in detector bias is performed on a spectrometer PHA. This delay is used to allow the electronics sufficient time to settle before an acquisition is started. The default is 2.0 seconds and the value must be greater than or equal to zero and less than 100 seconds.

UseEmpiricalPHADefaults
Specifies whether to calculate PHA baseline, window, gain and bias defaults based on fit coefficients from the EMPPHA.DAT file. Only applicable when manually entering element setups. The default is to not use empirical PHA defaults. Change the UseEmpiricalPHADefaults flag to any non-zero value to use empirical PHA defaults calculated from the fit coefficients from the EMPPHA.DAT file. The format of the file is as shown here:

```
1   "TAP"   0.00    "Baseline"      0.001     1.00    0.000   0.00
1   "TAP"   0.00    "Window"        0.001    10.00    0.000   0.00
1   "TAP"   0.00    "Gain"          0.000     0.00    0.000   0.00
1   "TAP"   0.00    "Bias"        -89.430  1668.35    0.000   0.00
1   "LDEB"  0.00    "Baseline"     -0.070     0.56    0.000   0.00
1   "LDEB"  0.00    "Window"        0.001    10.00    0.000   0.00
1   "LDEB"  0.00    "Gain"          0.000     0.00    0.000   0.00
1   "LDEB"  0.00    "Bias"        -58.160  1701.24    0.000   0.00
1   "LDE1"  0.00    "Baseline"      0.040     0.95    0.000   0.00
1   "LDE1"  0.00    "Window"        0.001    10.00    0.000   0.00
1   "LDE1"  0.00    "Gain"          0.000     0.00    0.000   0.00
1   "LDE1"  0.00    "Bias"        -89.030  1656.12    0.000   0.00
1   "PET"   5.00    "Baseline"      1.200    -0.27    2.030  -2.09
1   "PET"   0.00    "Window"        0.001     5.00    0.000   0.00
1   "PET"   0.00    "Gain"          0.000     0.00    0.000   0.00
1   "PET"   0.00    "Bias"       -100.570  1670.39    0.000   0.00
```

```
2   "TAP"   0.00    "Baseline"       0.001     1.00    0.000   0.00
2   "TAP"   0.00    "Window"         0.001    10.00    0.000   0.00
2   "TAP"   0.00    "Gain"           0.000     0.00    0.000   0.00
2   "TAP"   0.00    "Bias"         -89.430  1668.35    0.000   0.00
2   "LDEC"  0.00    "Baseline"       0.060     0.72    0.000   0.00
2   "LDEC"  0.00    "Window"         0.001    10.00    0.000   0.00
2   "LDEC"  0.00    "Gain"           0.000     0.00    0.000   0.00
2   "LDEC"  0.00    "Bias"         -59.120  1688.58    0.000   0.00
3   "LiF"   8.00    "Baseline"       1.080    -0.38    0.630  -0.67
3   "LiF"   0.00    "Window"         0.001    10.00    0.000   0.00
3   "LiF"   0.00    "Gain"           0.000     0.00    0.000   0.00
3   "LiF"   0.00    "Bias"         -98.430  1841.70    0.000   0.00
3   "PET"   0.00    "Baseline"       0.180     1.06    0.000   0.00
3   "PET"   0.00    "Window"         0.001     5.00    0.000   0.00
3   "PET"   0.00    "Gain"           0.000     0.00    0.000   0.00
3   "PET"   0.00    "Bias"        -106.100  1859.76    0.000   0.00
4   "TAP"   0.00    "Baseline"       0.001     1.00    0.000   0.00
4   "TAP"   0.00    "Window"         0.001    10.00    0.000   0.00
4   "TAP"   0.00    "Gain"           0.000     0.00    0.000   0.00
4   "TAP"   0.00    "Bias"         -76.380  1749.55    0.000   0.00
4   "PET"   5.40    "Baseline"       1.470    -0.46    0.840  -0.73
4   "PET"   0.00    "Window"         0.001     5.00    0.000   0.00
4   "PET"   0.00    "Gain"           0.000     0.00    0.000   0.00
4   "PET"   0.00    "Bias"         -95.530  1764.11    0.000   0.00
5   "LIF"   8.00    "Baseline"       1.080    -0.38    0.630  -0.67
5   "LIF"   0.00    "Window"         0.001    10.00    0.000   0.00
5   "LIF"   0.00    "Gain"           0.000     0.00    0.000   0.00
5   "LIF"   0.00    "Bias"         -98.430  1841.70    0.000   0.00
5   "PET"   0.00    "Baseline"       0.180     1.06    0.000   0.00
5   "PET"   0.00    "Window"         0.001     5.00    0.000   0.00
5   "PET"   0.00    "Gain"           0.000     0.00    0.000   0.00
5   "PET"   0.00    "Bias"        -106.100  1859.76    0.000   0.00
```

The first column indicates the spectrometer number, the second column the crystal, and the third column the KeV threshold for whether to use the low or high fit coefficients, the fourth column the data type (Baseline, Window, Gain or Bias only). The next two columns are the low threshold linear fit coefficients (slope and intercept) used to calculate the PHA defaults. The last two columns are the high threshold linear fit coefficients (slope and intercept) used to calculate the PHA defaults.

The expression used to calculate the PHA parameters is shown here:

$$y_{PHA} = m \cdot x + b$$

where: m = the slope coefficient
b = the intercept coefficient
x = the natural log of the emission line energy in KeV

The program will automatically load PHA defaults values from the SCALERS.DAT file if any of the following conditions are true in the EMPPHA.DAT file:

- the spectrometer and crystal pair are not found
- the coefficient slope and intercept are both zero
- the PHA data type is not found

Also, if the KeV threshold is zero, the program will always use the low slope and intercept (assuming that they are not both zero).

KilovoltChangeDelay=5.0 ! SX-50/51/100 only

Specifies the default time delay used when a change in the operating voltage is performed. This parameter is only utilized if the OperatingVoltagePresent flag is true. The default is 5.0 seconds and the valid range is from 0.0 to 100 seconds. A change in the operating voltage will force a change in the beam current (if available).

BeamCurrentChangeDelay=5.0 ! SX-50/51/100 only

Specifies the default time delay used when a change in the beam current is performed. This parameter is only utilized if the BeamCurrentPresent flag is true. The default is 5.0 seconds and the valid range is from 0.0 to 100 seconds. A change in the beam current will force a change in the beam size (if available).

BeamSizeChangeDelay=5.0 ! SX-50/51/100 only
Specifies the default time delay used when a change in the beam size is performed. This parameter is only utilized if the BeamSizePresent flag is true. The default is 5.0 seconds and the valid range is from 0.0 to 100 seconds.

LogWindowBufferSize=29000
Specifies the default size of the Log window text buffer. The size is limited to 64K on Win95 systems and only by the available memory on Win NT systems. The default is 29000 bytes (approx. 20 pages).

CommandPacingInterval=0
Specifies the number of system yields that are executed before each "realtime" command is sent to the hardware interface. This may improve system performance especially for serial interfaces. The default is 0, the allowable values may be between 0 and 100.

PeakOnAssignedStandards=0
Specifies the default mode for whether calibration of peak positions for each element should be attempted on all standards or for the standard assigned for that element. This value will be used for a new run to load the Calibrate on Assigned Standards check box in the Automate! window. The default is zero or false to attempt peaking on all standards.

PrintAnalyzedAndSpecifiedOnSameLine=0
Specifies the default mode for whether analyzed and specified element results in the log window should be printed out on the same line. This flag may be used in conjunction with the Extended Format flag to get all elements analyzed and specified (up to 32) all printed out on one line in the log window. The default is zero or false to print the specified elements on several lines first and then print the analyzed elements on subsequent lines next.

NoMotorPositionLimitsChecking=0 ! AM interface only
Specifies whether the spectrometer motor position electrical limit software check should be implemented during the crystal flip procedure. This flag only applies to the Advanced Microbeam interface using the original JEOL crystal flipping electronics. If this flag is set to a non-zero value then the software will not check the motor position limits during a crystal flip in both the manual and automated software procedures. The default is zero or false to check the motor position limits during a crystal flip procedure.

UserDataDirectory=""
Specifies the default directory that the user data files are stored in. Normally this is "C:\Program Files\Probe for Windows\UserData" but any other valid path may be used.

ExtendedMenu=0
This flag is used to indicate if additional menus should be visible in CalcZAF. These menus add calculations for comparing atomic, mass and electron fraction first approximation calculations and are not generally utilized by most users. The default is zero to have these menu items not visible. Set the flag to a non-zero value to make these items visible.

AutoAnalyze=0
This flag is used to indicate if Probe for Windows should automatically perform a quantitative analysis after each analyzed standard or unknown data point. The default is zero to not perform a quantitative analysis after each acquisition. Set the flag to a non-zero value to have the program automatically perform a quantitative analysis after each standard or unknown acquisition.

StatusAlwaysOnTop=0
This flag will specify whether the STATUS (Automation and Analyze) forms will be "always on top" of all other forms. The default is 0 for not always on top. Set this flag to a non-zero number if it is preferred that these STATUS forms should be "always on top".

FaradayAlwaysOnTop=0
This flag will specify whether the main window of the Faraday applet (beam and faraday cup control) will be "always on top" of all other forms. The default is 0 for not always on top. Set this flag to a non-zero number if it is preferred that the Faraday program main window should be "always on top".

ColumnConditionChangeDelay=10.0
Specifies the default time delay used when a change in the column condition is performed on the microprobe. This delay is used to allow changes in the electron column configuration sufficient time to complete before an acquisition is started. The default is 10.0 seconds and the value must be greater than or equal to zero and less than 100 seconds.

SurferOutputVersionNumber=6
Specifies the default output format for the Surfer (from Golden Software) OLE scripting .BAS file. This is necessary due to the fact that there are subtle differences in the way VBA scripting is implemented in different versions of Surfer. The default is 6 for Surfer version 6.0 and the only allowed values are 6 for Surfer 6.0 or 7 for Surfer 7.0.

SelPrintStartDoc=1
Specifies whether the print option for the Log window should send nested StartDoc and EndDoc commands to the printer (the default behavior). Because some printers will lock up when they receive a nested StartDoc and EndDoc commands this option allows this default behavior to be disabled if the flag is set to zero. The default is non-zero for allowing nested StartDoc and EndDoc commands to be sent to the printer.

UseMultiplePeakCalibrationOffset=0
This parameter specifies whether the software will calculate the default spectrometer offset (from the theoretical peak position to the actual peak position) using a fixed offset or based on coefficients stored in the PROBEWIN-*X*.CAL file (where *X* is K, L or M representing each of the x-ray line families). Using this method the user can calibrate each crystal on each spectrometer so that the default peak positions are automatically updated for the proper position offsets based on the spectrometer position and the measured offsets.

The PROBEWIN-*X*.CAL file contains, for each crystal on each spectrometer, a number of elements and standards used for the peak calibration process. Up to 5 element/x-ray/standard combinations may be specified for each spectrometer/crystal calibration in the system. The software will also store the theoretical and actual (measured) peak positions for each peak center along with the fit coefficients calculated using a 2nd order polynomial fit. The fit is based on the actual peak position versus the measured offset from the theoretical peak position. For example the spectrometer position offset is calculated using the following equation:

$$P_O = P_T - P_A$$

Where P_T is the theoretical spectrometer peak position
P_A is the actual (measured) spectrometer peak position

The calculated offset is applied to the theoretical peak position as follows:

$$P_A = P_T - P_O$$

Where P_T is the theoretical spectrometer peak position
P_O is the calculated spectrometer position offset from above

Use the Peak/Scan Options dialog in program StartWin to maintain and update these spectrometer peak position offset coefficients. The default offset is zero until the user performs multiple peal calibrations. Note that if only a single x-ray line is specified for the calibration procedure on a particular spectrometer/crystal combination, the software will apply that measured offset as a constant over the entire spectrometer range. If two elements are specified, the software will apply a straight line interpolation over the spectrometer range. If three elements, then a polynomial fit is used.

[Hardware]

InterfaceType=0

```
NumberOfFixedSpecs=0
NumberOfTunableSpecs=5
NumberOfStageMotors=4

JoyStickPresent=1
JoyStickType=0
JoyStickXDeadBand=3000
JoyStickYDeadBand=3000
JoyStickZDeadBand=3000
JoystickXPolarity=0
JoystickYPolarity=1
JoystickZPolarity=0
JoystickXBuffer=1000
JoystickYBuffer=1000
JoystickZBuffer=100

RotationMotorIndexNumber=4
RotationMotorDirectionType=0

SpecBacklashFlag=0
StageBacklashFlag=0
SpecBacklashType=1
StageBacklashType=1

FilamentStandbyPresent=0
FilamentStandbyType=0

EDSInterfacePresent=1
EDSInterfaceType=0
EDS_IPAddress=""
EDS_ServicePort=""

WDS_IPAddress=""
WDS_ServicePort=""

Rlogin_IPAddress=""
Rlogin_Login=""
Rlogin_Password=""

OperatingVoltagePresent=0
OperatingVoltageType=0
OperatingVoltageTolerance=0.002

BeamCurrentPresent=0
BeamCurrentType=0
BeamCurrentTolerance=0.02

BeamSizePresent=0
BeamSizeType=0

MagnificationPresent=0
MagnificationType=1

AutoFocusPresent=0
AutoFocusType=0
```

```
ROMPeakingPresent=0
ROMPeakingType=0

CounterTimerType=0
CounterTimerDevicename="IOMapper0"
CounterTimerBeamChannel=1

ReadOnlySpecPositions=0
ReadOnlyStagePositions=0
WriteOnlyPHAHardware=1
StageMotorsAlwaysOn=0

SX50InterfaceBoardType=1
SX50SEMDeviceName="IOMapper5"
SX50WDSDeviceName="IOMapper6"
SX50PreAcquireString=""
SX50PostAcquireString=""
SX50nPacing=2
SX50UseTripleRedundancyMotor=0
SX50UseTripleRedundancyCounter=0
SX50UseDriverLocking=0

ColumnConditionPresent=0
ColumnConditionType=0
ColumnConditionMethod=0
ColumnConditionString=""

ScanRotationPresent=0
ScanRotation=0.0

DetectorSlitSizePresent=0
DetectorSlitSizeType=0
DetectorSlitPositionPresent=0
DetectorSlitPositionType=0
DetectorModePresent=0
DetectorModeType=0

EdaxStagePresent=0
EdaxStageType=0

TiltRotationPresent=0
TiltRotationType=0

MoveAllStageMotorsHardwarePresent=0
Jeol8900PreAcquireString=""
Jeol8900PostAcquireString=""
```

InterfaceType=0
Specify the hardware interface type to be used for microprobe interface control. The following values are allowed :

0 = Demonstration mode (no hardware interface)
1 = Advanced MicroBeam hardware interface
2 = Jeol 8900 interface (direct socket)
3 = Kevex Sesame hardware interface (serial)

4 = TRACOR hardware interface (serial)
5 = SX100 interface (Ethernet)
6 = SX50 (Advanced Microbeam) hardware interface (DLL)
7 = Jeol 8900 interface (rlogin)
8 = Elionix interface (DLL)

Note that specifying the wrong hardware interface could cause the computer to "lock-up" if the hardware interface defined is not actually present. The default value is 0 for demonstration mode (no hardware interface).

Special note: when using InterfaceType=6 (Cameca SX50/51 only), the spectrometer dynamic offsets MUST always be zero. Therefore, use of the SXLocal "veri spec" command is forbidden, to ensure proper functioning of the software.

Note that some video board graphics chips turn off serial port interrupts to improve screen redraw rates. This could interfere with serial communications and therefore should be disabled if any of the serial interfaces are used (Tracor and Sesame). Contact Advanced Microbeam for further information.

NumberOfFixedSpecs=0 ! ARL SEMQ only
Enter the actual number of fixed spectrometers (monochromators) on the microprobe (ARL SEMQ only). JEOL and Cameca will normally have this value set to 0. The allowable range is 0 to MAXSPEC%. The default value is 0 for no fixed spectrometers.

NumberOfTunableSpecs=5
Enter the actual number of tunable spectrometers (scanning) on the microprobe. The allowable range is 0 to MAXTUNABLE%. The default is 0 for no tunable spectrometers.

NumberOfStageMotors=3
Enter the actual number of stage motors on the microprobe. For no stage control enter 0, for X and Y motion only, enter 2, for X, Y and Z motion, enter 3, for JEOL 733 microprobes with a sample rotation motor, enter 4. The allowable range is 1 to MAXAXES%. The default value is 0 for no stage motors.

JoyStickPresent=0
A boolean value to specify whether a joystick for motor control is installed on the computer. Enter zero for no joystick or a non-zero value if a joystick device is present and to be used for motion control. A joystick connected to a game port or sound card must be installed in the Windows Control Panel to use this feature. The default is 0 for no joystick control.

The slower interfaces such as the serial (TRACOR and SESAME) and SX50 interfaces should generally disable joystick capability.

JoyStickType=0
The joystick type. This parameter species whether the joystick motion commands are sent in "position" or "velocity" mode. Previous versions of Joywin.exe used only "position" which could result in erratic motion. New versions can use a "velocity" mode of the controller which provides improved response. The preferrred mode is velocity mode. Specify 0 for position mode (default for backward compatibility) or 1 for velocity mode.

JoyStickXDeadBand=2000
The joystick X axis deadband. Applied to the X axis (side to side joystick motion) on the joystick to eliminate "jitter". Increase this value if the joystick sends stage move commands when not touched. Note that the joystick can also be re-centered by clicking the "Reset" button in the JOYWIN program interface. The allowable range is 1 to 32000. The default value is 2000.

JoyStickYDeadBand=2000
The joystick Y axis deadband. Applied to the Y axis (to and fro joystick motion) on the joystick to eliminate "jitter". Increase this value if the joystick sends stage move commands when not touched. Note that the joystick can also be re-

centered by clicking the "Reset" button in the JOYWIN program interface. The allowable range is 1 to 32000. The default value is 2000.

JoyStickZDeadBand=2000
The joystick Z axis deadband. Applied to the Z axis on the joystick to eliminate "jitter". Increase this value if the joystick sends stage move commands when not touched. Note that the joystick can also be re-centered by clicking the "Reset" button in the JOYWIN program interface. This parameter only applies to "3D" joysticks. The allowable range is 1 to 32000. The default value is 2000.

JoyStickXPolarity=0
The joystick X axis polarity. Applied to the X axis on the joystick to reverse the default direction of the motor. Change this value to "1" to reverse the polarity of the X axis joystick axis. The default value is 0.

JoyStickYPolarity=0
The joystick Y axis polarity. Applied to the Y axis on the joystick to reverse the default direction of the motor. Change this value to "1" to reverse the polarity of the Y axis joystick axis. The default value is 0.

JoyStickZPolarity=0
The joystick Z axis polarity. Applied to the Z axis on the joystick to reverse the default direction of the motor. Change this value to "1" to reverse the polarity of the Z axis joystick axis. The default value is 0. This parameter applies to 3-D joysticks only.

JoystickXBuffer=1000
JoystickYBuffer=1000
JoystickZBuffer=100
These so-called "buffer" values are used for two different but related functions depending on whether the Joywin.exe program is run in "position" or "velocity" mode (see JoyStickType parameter above).

In "position" mode (JoyStickType = 0), one may use these parameters to specify the size of the motor count "buffer" that can build up during joystick motion. If the difference between the target motor position and the actual motor position is greater than the value specified by these three parameters, then the program will not send the joystick command to the motor controller. This should have the effect of preventing the buildup of a large "backlog" of motion in the motor controller when the user "leans on" the joystick for a length of time. The result is that as soon as the user releases the joystick the motor should travel no further than the size of the "buffer".

The "buffer" values are in motor encoder counts and may be specified separately for the X, Y and Z axes. The defaults are 1000 encoder counts for the X and Y directions and 100 for the Z axis. These values are only utilized during normal joystick motion and are ignored for single axis joystick motion, for example moving the spectrometers using the X motion of the joystick.

In "velocity" mode (JoyStickType = 1), use these parameters to specify the number of milliseconds to delay before the stage motors are "turned off" after a zero velocity is issued to the controller. This occurs whenever the joystick handle is released and it enters the center "deadband" region. Use a short number of milliseconds, for example 100, for "stopping on a dime" or a larger number of milliseconds, for example 1000, for a smoother stop. These parameters only apply to the stage motors and are ignored if the "StageMotorsAlwaysOn" flag is set to a non-zero value.

RotationMotorIndexNumber=4 ! JEOL 733 only
The number of sample positions in the stage sample rotation device (JEOL 733 only). This value is only used if the NumberOfStageMotors is 4, indicating a sample rotation axis. The default is 4 index positions.

RotationMotorDirectionType=0 ! JEOL 733 only
This keyword is used to indicate the direction of rotation of the sample stage device for JEOL 733 microprobes. Enter 0 or 1.

SpecBacklashFlag=0

This parameter is used to specify the default spectrometer backlash mode for all spectrometer motion. Enter 0 for false or any non-zero value for true. The default is 0 for no spectrometer backlash.

StageBacklashFlag=0
This parameter is used to specify the default stage backlash mode for stage motion from the Move dialog.

To set stage backlash options for standards, unknowns and wavescans separately for when the automation is used, refer to the Acquisition Options button in the Acquire! dialog. Note that the StageBackLashFlag in the Move dialog overrides the backlash options in the Acquisition Options dialog.

Enter 0 for false or any non-zero value for true. The default is 0 for no stage backlash.

SpecBacklashType=1
This parameter is used to indicate the spectrometer backlash type. Specify 1 for Probe for Windows to control the spectrometer backlash, or 2 for ROM based backlash. This option is only available for certain hardware interfaces that support machine based automatic backlash or jog options (SESAME or TRACOR).

StageBacklashType=1
This parameter is used to indicate the stage backlash type. Specify 1 for Probe for Windows to control the stage backlash, or 2 for ROM based backlash. This option is only available for certain hardware interfaces that support machine based automatic backlash or jog options (SESAME or TRACOR).

FilamentStandbyPresent=0
This parameter is used to specify whether the microprobe hardware interface supports a filament standby mode for turning the filament off using the software automation. Enter 0 for false or any non-zero value for true. The default is 0 for no filament standby hardware interface.

Note that the SX50 (InterfaceType=6) interface will simply send the command "RTSK REST" to the SX ROM. This task (REST.TAS) should be created by the operator for the specified purpose using the appropriate SX commands and loaded into the SX ROM beforehand.

FilamentStandbyType=0
The filament standby type. This parameter is not yet implemented.

EDSInterfacePresent=0
This parameter is used to specify whether the microprobe hardware interface supports EDS hardware for combined WDS and EDS analysis. Enter 0 for false or any non-zero value for true. The default is 0 for no EDS hardware interface.

EDSInterfaceType=0
The EDS hardware interface type. The following high level EDS interfaces are supported:

```
EDSInterfaceType    =    0    ' Demonstration mode (concentration = 0.123456 wt. %)
EDSInterfaceType    =    1    ' Edax EDAM software interface (32 bit)
EDSInterfaceType    =    2    ' PGT software interface (32 bit)
EDSInterfaceType    =    3    ' Oxford software interface (32 bit)
EDSInterfaceType    =    4    ' Kevex software interface (32 bit)
```

EDSThinWindowPresent=0
If an ultra-thin window is used in the EDS system and combined EDS-WDS analyses are being performed, the optical light source must be turned off. This parameter is used to specify that a ultra-thin window is present that could transmit some of the optical light signal. Enter 0 for false or any non-zero value for true. The default is 0 for no EDS thin window.

EDS_IPAddress=""

For certain EDS interfaces (PGT) the IP address of the UNIX EDS acquisition server must be specified. The address must be in the correct TCP/IP "dot" format, e.g. "128.32.146.11".

EDS_ServicePort=""
For certain EDS interfaces (PGT) the service port number of the UNIX EDS acquisition server must be specified. The service port number must be an integer between 1 and 32768.

WDS_IPAddress=""
For certain WDS interfaces (JEOL 8900 and SX100) the IP address of the microprobe system controller must be specified. The address must be in the correct TCP/IP "dot" format, e.g. "128.32.146.11".

Note that for the JEOL 8900 the default IP address for the system controller is 192.6.1.11 and for the client computer it is 192.6.1.1. Note also that contrary to the JEOL documentation, only one client is supported by the system controller at a time.

WDS_ServicePort=""
For certain WDS interfaces (JEOL 8900 and SX100) the service port number of the microprobe system controller must be specified. The service port number must be an integer between 1 and 32768.

Note that for the JEOL 8900 the default service port number for the system controller is 2785.

Rlogin_IPAddress=""
The IP address for an "rlogin" interface. For the JEOL 8900 rlogin interface (InterfaceType=7) this is the IP address of the HP Unix box that normally controls the microprobe. The address must be in the correct TCP/IP "dot" format, e.g. "128.32.146.11".

Rlogin_Login=""
The login name for the "rlogin" connection. This must be a valid account with appropriate privileges to allow the software to run the xm_command binary. Note that a separate login is made for each spectrometer in the system, plus an additional one for the stage and beam control.

Rlogin_Password=""
The login password for the "rlogin" connection. This is for the above "Rlogin_Login" login name. Note that a separate login is made for each spectrometer in the system, plus an additional one for the stage and beam control.

OperatingVoltagePresent=0
This parameter is used to specify whether the microprobe hardware interface supports control of the accelerating voltage. Enter 0 for false or any non-zero value for true. The default is 0 for no operating voltage hardware interface.

OperatingVoltageType=0
The operating voltage hardware interface type. This parameter is used to determine the operating voltage interface type for Advanced Microbeam interfaces (InterfaceType = 1) when the column control option is available.

OperatingVoltageType = 0	no operating voltage column control available
OperatingVoltageType = 1	JEOL 6400 serial interface operating voltage column control
OperatingVoltageType = 2	JEOL 8600 serial interface operating voltage column control

If the JEOL serial interface for column control is available the serial port parameters in the [serial] section below must be properly set. Specifically the following parameters must be set to these values for the JEOL serial interface column control for the JSM-6400 SEM, 8600 and related electron beam instruments:

Port = 1	depends on the specific installation
HandShaking = 0	no handshaking used
Baud = 2400	also available are 1200 or 600 baud
Parity = "N"	no parity checking

DataBits = 8	8 character bits
StopBits = 1	1 stop bit

OperatingVoltageTolerance=0
The operating voltage tolerance is used to specify how close the operating voltage read from the system must be to the desired operating voltage in order to allow the program to skip re-setting the operating voltage. The parameter is in fraction units, that is 0.01 equals 1% and the default is 0.002 or 0.2%. The allowed range is 0.0001 (0.01%) to 0.1 (10%).

BeamCurrentPresent=0
This parameter is used to specify whether the microprobe hardware interface supports control of the beam current. Enter 0 for false or any non-zero value for true. The default is 0 for no beam current hardware interface.

BeamCurrentType=0
The beam current hardware interface type. This parameter is used to determine the beam current interface type for Advanced Microbeam interfaces (InterfaceType = 1) when the column control option is available.

BeamCurrentType = 0	no beam current column control available
BeamCurrentType = 1	JEOL 6400 serial interface beam current column control
BeamCurrentType = 2	JEOL 8600 serial interface beam current column control

If the JEOL serial interface for column control is available the serial port parameters in the [serial] section below must be properly set. Specifically the following parameters must be set to these values for the JEOL serial interface column control for the JSM-6400 SEM and related electron beam instruments:

Port = 1	depends on the specific installation
HandShaking = 0	no handshaking used
Baud = 2400	also available are 1200 or 600 baud
Parity = "N"	no parity checking
DataBits = 8	8 character bits
StopBits = 1	1 stop bit

BeamCurrentTolerance=0
The beam current tolerance is used to specify how close the beam current read from the system must be to the desired beam current in order to allow the program to skip re-setting the beam current. The parameter is in fraction units, that is 0.01 equals 1% and the default is 0.02 or 2%. The allowed range is 0.0001 (0.01%) to 0.1 (10%).

BeamSizePresent=0
This parameter is used to specify whether the microprobe hardware interface supports control of the beam size. Enter 0 for false or any non-zero value for true. The default is 0 for no beam size hardware interface.

BeamSizeType=0
The beam size hardware interface type. This parameter is used to determine the beam size interface type for Advanced Microbeam interfaces (InterfaceType = 1) when the column control option is available.

BeamSizeType = 0	no beam size column control available
BeamSizeType = 1	JEOL 6400 serial interface beam size column control
BeamSizeType = 2	JEOL 8600 serial interface beam size column control

If the JEOL serial interface for column control is available the serial port parameters in the [serial] section below must be properly set. Specifically the following parameters must be set to these values for the JEOL serial interface column control for the JSM-6400 SEM and related electron beam instruments:

Port = 1	depends on the specific installation
HandShaking = 0	no handshaking used
Baud = 2400	also available are 1200 or 600 baud

Parity = "N"	no parity checking
DataBits = 8	8 character bits
StopBits = 1	1 stop bit

MagnificationPresent=0
This parameter is used to specify whether the microprobe hardware interface supports control of the beam scan magnification. Enter 0 for false or any non-zero value for true. The default is 0 for no beam scan magnification hardware interface.

MagnificationType=1
The bean scan magnification hardware type. This parameter is not yet implemented.

AutoFocusPresent=0 ! SX-50/51/100 only
This parameter is used to specify whether the microprobe hardware interface supports control of the auto focus. This parameter must be set for the use of ROM based auto focus options during automated acquisitions. Only the SX50/51 microprobe supports a ROM based auto focus feature. Enter 0 for false or any non-zero value for true. The default is 0 for no auto focus hardware interface. See also, the parameter EDSThinWindowPresent above.

AutoFocusType=0
The autofocus hardware interface type. This parameter is not yet implemented.

ROMPeakingPresent=0 ! SX-50/51/100, Sesame and TRACOR only
This parameter is used to specify whether the microprobe hardware interface supports control of the ROM based peak centering hardware that is internal to the microprobe. Enter 0 for false or any non-zero value for true. The default is 0 for no ROM based peak centering hardware interface.

ROMPeakingType=0
The ROM based peak center hardware interface type. This parameter is not yet implemented.

CounterTimerType=0
This parameter is used to indicate the counter-timer hardware type and is used only by the Advanced Microbeam interface to implement a simulation mode for the counter-timer hardware. To turn on counter-timer hardware simulation, set the CounterTimerType to "1". In simulation mode the program will use the "demo" interface (InterfaceType = 0) instead of the DCC20AX board for counting purposes. The default is "0" for the DCC20AX counter-timer board interface.

CounterTimerDeviceName="IOMapper0"
Parameter to indicate the counter-timer hardware I/O device name for the Advanced MicroBeam hardware. A single counter-timer board can support asynchronous 32 bit timing and counting for 5 channels. The first channel is usually reserved for beam current counting for the Advanced Microbeam hardware (except, SX50, Sesame and Tracor installations).

Note that the counter-timer board is required for ALL non-demo interface types. Since all installations will normally utilize at least the counter-time board for x-ray imaging purposes with Micro-Image, this procedure acts as a form of copy protection.

Enter a valid device name as specified in the Win NT registry in the IOMapper key under the NT Services key using the IOMapper Control Panel applet. The default value is "IOMapper0".

Note for multiple ISA bus boards:
If more than one ISA counter timer board is required to accommodate all scaler counting channels, then the additional ISA boards must have their base address DIP switches set in 8 byte intervals to yield a consecutive I/O address space. For example, if the first counter-timer board is set to 768 (300 hex), then the second board must be addressed to 776 (308 hex). In this case a single IOMapper device can be used to address all the ISA counter-timer boards. If the ISA boards cannot be addressed to yield a consecutive I/O address space, use the method described below for PCI bus boards.

Note for multiple PCI bus boards:
If more than one PCI counter timer board is required to accommodate all scaler counting channels, then the additional PCI boards must each be addressed using a separate IOMapper device name. This is due to the fact that PCI bus addresses are specified automatically by the computer BIOS. In this case the additional IOMapper device names must be specified in the ProbeWin INI file. This is done by using comma separated strings values as seen here:

```
CounterTimerDeviceName="IOMapper0,IOMapper1,IOMapper2"
```

This example shows a three PCI board installation. Be sure that each PCI counter-timer board device is specified with a range of exactly 8 bytes in the IOMapper control panel applet.

CounterTimerBeamChannel=1
This parameter for the AM hardware interface is used to indicate if the first channel of the counter-timer board is used for the beam current measurement. Generally the first channel of the counter timer board is used for beam current measurements. If this parameter is set to zero then either counter-timer board is only used only for x-ray imaging or the beam current is measured using a separate A/D converter board. The default is 1 to indicate that the first channel of the counter-timer board is used for beam current measurements.

ReadOnlySpecPositions=0
This parameter is used to indicate if the spectrometer motor positions are "read only". That is, they cannot be set by software. This is generally only true for the SX-50 microprobe which allows reading of the spectrometer positions only. The default is zero or false for read and write of spectrometer positions.

ReadOnlyStagePositions=0
This parameter is used to indicate if the stage motor positions are "read only". That is, they cannot be set by software. This is generally only true for the SX-50 microprobe which allows reading of the stage positions only. The default is zero or false for read and write of stage positions.

WriteOnlyPHAHardware=1 ! SX-50/51/100 only
This parameter is used to indicate if the PHA hardware values are "write only". That is, they cannot be read by software. Read and write PHA capability is generally only true for the SX50 interfaces. The default is one or true for "write only" of PHA parameters.

StageMotorsAlwaysOn=0
This parameter is used to indicate if the stage motors should always stay energized after a move command. The default is to de-energize the stage motors after a move command. If stage "relaxation" after the motors are de-energized is a problem, it may be necessary to enable this parameter.

A non-zero value here will force the stage motors to always stay energized. To release the stage motors the user must therefore click the Free/Clear button in the Move window. This option affects only the AM DCX motor interface.

SX50InterfaceBoardType=1 ! SX-50/51/100 only
SX50SEMDeviceName="IOMapper5" ! SX-50/51/100 only
SX50WDSDeviceName="IOMapper6" ! SX-50/51/100 only
These parameters are used to indicate the interface board type, and device names for the SEM and WDS interface boards for the DRV11 (DR11-C) I/O PC interface boards used for interfacing to the SX50/51 CAMECA microprobe. This interface was originally a 16 bit DEC (Digital Equipment Corporation) parallel interface used with a PDP type computer.

The only valid interface board type currently supported is type = 0 for testing purposes only (SX-TEST.DLL), type = 1 for the ISA PC11 board (SX-PC11.DLL) and type = 2 for the Logical PCI board..

For the device names, enter a valid device name from the Win NT registry in the IOMapper key under the NT Services key using the IOMapper Control Panel applet. The default value is "IOMapper5" for the SX50 SEM interface board and "IOMapper6" for the SX50 WDS interface board.

Note that the Logical boards REQUIRE that the device names in the PROBEWIN.INI file be "dic0" for the SEM board and "dic1" for the WDS board (IOMapper configuration is not required for the Logical boards).

SX50PreAcquireString=""
SX50PostAcquireString=""
These parameters are used by the SX50 interface to set special conditions before and after the acquisition as specified by the user. For example, it might be desirable to give the command "MODE FIX" before an acquisition to ensure that the beam is not scanning during the counting followed by the command "MODE TV" to restore the beam scan after the acquisition is completed. The default is no command string for both parameters.

Note that the command strings are sent only to the SEM interface and therefore should be appropriate for vacuum and column commands.

SX50nPacing=2
Sets the character pacing of the SX50 parallel DRV-11 interface. The default is 2 milli-seconds, the valid range is from 1 to 50 milli-seconds. This parameter is used only for the SX50 interface (InterfaceType = 6).

SX50UseTripleRedundancyMotor=0
SX50UseTripleRedundancyCounter=0
These two flags may be used to turn on or turn off the "triple redundancy" code in the SX50 interface. Because the SX50 parallel interface is intrinsically slightly noisy, turning on one or both of these flags prohibits any possibility of data corruption during communication.

The SX50UseTripleRedundancyMotor flag affects the GetMotorPosition and GetMotorStatus functions, the SX50UseTripleRedundancyCounter flag affects the GetCounts function. When the respective flag is true (non-zero) the program will make two calls for each parameter to ensure that the data is consistent. If there is a discrepancy, a warning is sent to the log window (in Debug Mode) and the program will attempt a third time and check whether any two of the three data are consistent. If all three are inconsistent, a warning is sent to the log window (in DebugMode) and a fourth attempt is utilized.

The default is to not use the "triple redundancy" code, because the use of this feature can slightly slow down the interface. Turning off the flag (zero) will disable the "triple redundancy" code, and will provide a more responsive interface. An excellent way to check whether the "triple redundancy" code is necessary is to run the program in Debug Mode and check the log window for warnings regarding additional attempts to obtain consistent data from the SX50.

SX50UseDriverLocking=0
This flag will allow the program to use thread process locking for reading and writing from the SX50 driver. This is useful when more than one application may be communicating with the driver at the same time. The default is to not use driver locking. Set this flag to any non-zero value to use driver locking for the SX50 driver.

Note that driver locking requires v. 2.11.0000 or higher of the SX50 driver (new functions SX_Lock and SX_Unlock). To check the version of the SX50 driver, right click on the file SX50_32.DLL in the SYSTEM32 folder and click the properties menu. The version information can be seen in the Version tab.

ColumnConditionPresent=0
ColumnConditionType=0
ColumnConditionMethod=0
ColumnConditionString=""
These keywords are used to specify if a column condition interface is present and if so how it should be utilized. Normally, microprobes without column interfaces use the kilovolt, beam current and beam size parameters simply for documentation purposes.

However, on instruments with a column interface, these kilovolt, beam current and beam size parameters are actually used to also set the operating condition of the electron column. On other instruments, these conditions cannot be specified individually, and instead a "column condition string" is specified that configures the microscope for the desired operating conditions. Again, if the column condition interface is not actually present, then this field may be used simply for documentation purposes.

Some instruments, may have both the capability of setting the individual kilovolt, beam current and beam size parameters and also the capability of setting a "column condition string". This is the case with the SX50/51 instrument. In this situation either method may be used to actually set the electron column condition, however it must be noted that the even if the column condition method is utilized, the individual kilovolt (especially this parameter), beam current and beam size parameters must also be correctly specified so that the software can perform a proper quantitative matrix correction of the intensity data. This is because there is no rigorous method to determine the actual operating voltage from an arbitrary column condition string.

Therefore, to indicate the presence of a column condition interface (SX50/51 and JEOL 6400 only at the present time), set the ColumnConditionPresent keyword to a non-zero value. The default is zero for no column condition present.

To indicate the type of the column condition interface, set the ColumnConditionType. This parameter is not currently utilized. At the present time, if the InterfaceType=1 (for the Advanced Microbeam interface) then the JEOL 6400 column condition interface (serial port) is utilized. If the InterfaceType=6, then the sx50 column condition interface is utilized. This parameter may be utilized for future optional column control interfaces.

The ColumnConditionMethod is used to specify the default method to set the electron column operating conditions. If the ColumnConditionMethod is not specified or is zero, then the kilovolts, beam current and beam size parameters are individually specified for configuring the electron column. If the ColumnConditionMethod is 1, then the specified column condition string is utilized to set the electron column conditions.

The ColumnConditionString is used the specify the default column condition string. Normally this is blank but in the case of the InterfaceType=6 (SX50/51), the default is "hv15".

ScanRotationPresent=0
ScanRotation=0.0
These parameters are used to indicate that a scan rotation interface is present and what the default scan rotation should be set to upon startup. The default is 0 for no scan rotation interface present and 0.0 for zero degrees default scan rotation. Any non-zero value is used to indicate that the scan rotation interface is present and any value from 0 to 360 is valid for the default scan rotation.

DetectorSlitSizePresent=1
DetectorSlitSizeType=0
DetectorSlitPositionPresent=0
DetectorSlitPositionType=0
DetectorModePresent=1
DetectorModeType=0
These parameters are used to indicate that an interface is present for various detector parameters. Specifically for slit size, slit position and detector mode. The default is 0 for no detector interface present and 0 for the interface type (not used at this time). Any non-zero value is used to indicate that the detector interface is present.

See the DETECTORS.DAT file for specific parameter values that can be specified by the user for configuring the various detector values.

EdaxStagePresent=0
EdaxStageType=0
These parameters indicate if an Edax Stage interface is present. This can be determined by the presence of the file "StgCtl32.dll" in the C:\Edax32\sys" folder. The default is 0 for no Edax Stage interface present and 0 for the Edax Stage interface type (not used at this time). Any non-zero value is used to indicate that the Edax Stage interface is present.

TiltRotationPresent=0
TiltRotationType=0
These parameters indicate if a stage tilt and rotation interface is present. This is usually part of the stage interface (e.g. Edax Stage). The default is 0 for no Tilt/Rotation interface present and 0 for the Tilt/Rotation interface type (not used at this time). Any non-zero value is used to indicate that the Tilt/Rotation interface is present.

MoveAllStageMotorsHardwarePresent=0
This parameter (intended for the Jeol8900 and Elionix-Edax stage control interfaces only), allows probe for Windows to issue a stage move control in a single interface command. This is implemented to support interfaces that cannot move the stage motors individually. Set this value to a non-zero value to force Probe for Windows to use a single command to move all three (or two) stage motors simultaneously.

Jeol8900PreAcquireString=""
Jeol8900PostAcquireString=""
These parameters are used by the JEOL 8900 interface to set special conditions before and after the acquisition as specified by the user. For example, it might be desirable to give the command "EOS_CMD PB OFF" before an acquisition to ensure that the beam is not scanning during the x-ray counting followed by the command "EOS_CMD ON" to restore the beam scan after the acquisition is completed.

Alternatively the user might prefer to issue a change in the magnification instead, "eos_mg 100000" before the acquisition and "eos_mg 40" after the acquisition. The default is "eos_cmd pb off" for the pre-acquire command string and "eos_cmd pb on" for the post-acquire string, if the InterfaceType is 7 (Jeol 8900), otherwise the default is blank.

[Image]

```
ImageInterfacePresent=0
ImageInterfaceType=0
ImageInterfaceNameChan1="Not Assigned"
ImageInterfaceNameChan2="Not Assigned"
ImageInterfacePolarityChan1=0
ImageInterfacePolarityChan2=0
ImageInterfaceImageIxIy=1.0
ImageInterfaceCalKeV=15.0
ImageInterfaceCalMag=400.0
ImageInterfaceCalXMicrons=300.0
ImageInterfaceCalYMicrons=200.0
ImageInterfaceADRange="bipolar5"
ImageInterfaceBaseAddress=576
ImageInterfaceProgrammableGain1=1
ImageInterfaceProgrammableGain2=1
ImageInterfaceControlLogic=0
ImageInterfaceBeamXPolarity=0
ImageInterfaceBeamYPolarity=0
ImageInterfaceStageXPolarity=0
ImageInterfaceStageYPolarity=0
```

ImageInterfacePresent=0
This parameter indicates if an imaging interface is available for use by Probe for Windows in digitizing stage positions graphically. A non-zero value indicates that the imaging interface is available. The default is zero for no imaging interface.

ImageInterfaceType=0
This parameter indicates the type of imaging interface available. A value of zero is the demonstration imaging interface. The default is zero for the demonstration imaging interface. The current interfaces that are defined are:

0 = Demonstration imaging interface
1 = MicroImage-32 imaging interface
2 = SX50 ROM imaging interface
3 = WIN30 (direct) imaging interface

ImageInterfaceNameChan1="Not Assigned"
ImageInterfaceNameChan2="Not Assigned"
This keywords are used to indicate the signal types for the analog input channels. Usually either "SE" or "BSE" signals are used for image digitizing purposes.

ImageInterfacePolarityChan1=0
ImageInterfacePolarityChan2=0
These keywords specify whether the analog signal obtained from the image interface is inverted or not. A normal signal (ImageInterfacePolarityChan1=0) is inverted, that is black = 255 and white = 0. If ImageInterfacePolarityChan1 is set to a non zero number, then the analog signal is treated as non-inverted, that is black = 0 and white = 255.

ImageInterfaceImageIxIy=1.0
This parameter is used to specify the aspect ratio of X over Y, for the normal beam scan generated by the imaging interface. A value of one is the default and would specify a square beam scan. A value greater or less than one would specify a rectangular beam scan.

ImageInterfaceCalKeV=15.0
This parameter specifies the operating voltage in kilovolts used for the ImageInterfaceCalXMicrons and ImageInterfaceYMicrons calibration. The default is 15 and the minimum is 5 and maximum is 50. This parameter is not used for the MI32 image interface (ImageInterfaceType = 1).

ImageInterfaceCalMag=400.0
This parameter specifies the magnification used for the ImageInterfaceCalXMicrons and ImageInterfaceYMicrons calibration. The default is 400 and the minimum is 10 and maximum is 10000. This parameter is not used for the MI32 image interface (ImageInterfaceType = 1).

ImageInterfaceCalXMicrons=300.0
ImageInterfaceCalYMicrons=200.0
These parameters specify the actual X and Y beam scan size at the kilovolt and magnification parameters specified above. These parameters are used the calculate the stage position of the beam deflection. The default ImageInterfaceCalXmicrons is 300 and the default ImageInterfaceCalYmicrons is 200 and the minimum is 10 and maximum is 10000. These parameters are not used for the MI32 image interface (ImageInterfaceType = 1).

ImageInterfaceADRange="bipolar5"
This parameter specifies the A/D (analog to digital) input range of the WIN30 board. This parameter is only used if the ImageInterfaceType = 3 (WIN30). The default is "bipolar5" and the only other valid value is "unipolar5".

ImageInterfaceBaseAddress=576
This parameter specifies the I/O port base address for the WIN30 board. This parameter is only required for Win95 or Win98 installations. The UEIDAQ NT driver loads this parameter automatically. The default is 576 (hex 240) and the allowable range is 512 to 1024.

ImageInterfaceProgrammableGain1=1
ImageInterfaceProgrammableGain2=1
These parameters specify the analog input gain used for WIN30 board. The default is 1 (no gain) for both analog channels and the allowable values are 1, 2, 4 and 8.

ImageInterfaceControlLogic=0

The parameter specifies the external beam control logic. The default is 0 for false and any non-zero value for true. This parameter is used only for the WIN30 board.

ImageInterfaceBeamXPolarity=0
ImageInterfaceBeamYPolarity=0
ImageInterfaceStageXPolarity=0
ImageInterfaceStageYPolarity=0
These four parameters specify the display of the beam and stage related coordinate systems. That is, the image display orientation for beam polarity and the stage registration for stage polarity. The default is zero for normal (graphical) registration using lower left as minimum and upper right as maximum. Any non-zero value will re-orient the image and/or stage registration.

[Serial]

```
Port=1
HandShaking=1
Baud=9600
Parity="N"
DataBits=8
StopBits=1
```

These serial port parameters are used by some interface types (SESAME or Tracor) to allow Probe for Windows to control the microprobe hardware through the serial port. Note that the serial port base address and interrupt also need to be properly configured using the Windows Control Panel.

Port=1
The serial communication port used for hardware interfacing to the microprobe hardware. Allowable serial communication port values are 1 - 4. The default is 1 for COM1.

HandShaking=1
HandShaking defines the data flow control. The allowable values are :

0 = no handshaking
1 = XON/XOFF
2 = RTS
3 = RTS and XON/XOFF

The default is 1 for XON/XOFF handshaking data flow.

Baud=9600
The serial port baud rate. The allowable values are : 1200, 2400, 4800, 9600 and 19200. The default is 9600 for 9600 baud.

Parity="N"
The serial port parity. The allowable values are :

"N" = none
"E" = even
"O" = odd
"M" = mark
"S" = space

The default is N for no parity checking on the serial port.

DataBits=8
The serial port data bits. The allowable values are 4 or 8. The default is 8 for 8 databits.

StopBits=1
The serial port stop bits. The allowable values are 1 or 2. The default is 1 for 1 stop bit.

[Faraday]

```
FaradayCupPresent=1
FaradayCountTime=3.0
FaradayCupType=0
FaradayWaitInTime=0.2
FaradayWaitOutTime=0.2
AbsorbedCurrentPresent=0
AbsorbedCurrentType=0
DefaultBlankBeamFlag=1
MinimumFaradayCounts=1.0
FaradayCountsToCurrentFactor=1.0
FaradayBeamCurrentSafeThreshold=500.0
FaradayStagePresent=0
FaradayStagePositionsX=0.0
FaradayStagePositionsY=0.0
FaradayStagePositionsZ=0.0
FaradayStagePositionsW=0.0
FaradayStagePositionsT=0.0
FaradayStagePositionsR=0.0
```

FaradayCupPresent=0
This boolean option specifies whether a faraday cup and associated hardware is present on the microprobe. This option should be zero (false) for ARL SEMQ probes and non-zero (true) for JEOL and Cameca microprobes.

FaradayCountTime=5.0
This option specifies the default integration time used to measure the faraday cup or aperture beam current. This value is generally only used for Advanced MicroBeam hardware installations. The allowable range is 1 to 100 seconds. The default value is 5 seconds.

For other interfaces the FaradayCountTime specifies the number of beam current measurements to average. In this case, the default is 1 for one beam current average. Note that the FaradayCountTime value is also used for the Absorbed Current measurement (if indicated).

FaradayCupType=0
This option specifies the faraday cup type. It must be set to zero for automatic recording of the beam current. For manual entry of the beam current (some older SEM instruments), change this value to any non-zero value. In this case, the program will prompt the user for the beam current value. The default is zero for automatic beam current measurement.

FaradayWaitInTime=0.5
Specifies the amount of time in seconds to wait after inserting the faraday cup before starting the beam current acquisition. The allowable range is 0 to 100 seconds. The default value is 0.5 seconds.

FaradayWaitOutTime=0.5
Specifies the amount of time in seconds to wait after removing the faraday cup before starting the x-ray count acquisition. The allowable range is 0 to 100 seconds. The default value is 0.5 seconds.

AbsorbedCurrentPresent=0
This boolean option specifies whether an absorbed current interface and associated hardware is present on the microprobe. If non-zero, the program will automatically acquire the absorbed current along with the faraday cup beam current.

AbsorbedCurrentType=0
This option specifies the absorbed current type. It is not used at this time.

DefaultBlankBeamFlag=1
This parameter is used to specify the default beam blank mode. Enter 0 for false or any non-zero value for true. If set to a non-zero value, the program will automatically leave the beam blanked after a stage motion command. If 0, the program will ask the user to confirm the beam unblank operation and then unblank the beam. The default is 1 to insert the faraday cup or blank the beam as the default.

MinimumFaradayCounts=1.0
This parameter defines the threshold that the program uses to decide if the filament may have blown. For interface types <= 1 (demo and Advanced Microbeam) the default threshold is 1 count. For interfaces types > = 2 (all others) the default threshold is 0.1 nA. The range of allowable values is 0.001 to 1000.

FaradayCountsToCurrentFactor=1.0
This parameter is used to specify the beam (and absorbed) current conversion factor for converting from counts per second to current in nA for display purposes. The actual counts are always stored in the data file itself. This parameter is used only by the demo and AM interfaces, all other interfaces report beam current directly in nA.

By default the parameter is specified (starting in version 4.21) as 100.0 for the demo and AM interfaces and 1.0 for all other interfaces. The usage is as follows:

$$nA = \frac{BeamCountsPerSecond}{FaradayCountsToCurrentFactor}$$

If it is preferred to view the beam current in actual counts per second, this keyword should be added to the [Faraday] section and set to 1.0.

FaradayBeamCurrentSafeThreshold=500.0
This option sets the maximum safe threshold for the beam current so as to force the software to confirm with the user that it is safe to remove the faraday cup (or unblank) the beam. The default is 500 nA and the allowable range is 1 to 10,000 nA. Set this value lower to have the software confirm a manual faraday remove at lower currents.

FaradayStagePresent=0
This flag tells the software if a specimen (stage) mounted faraday cup is present. If this device is present a beam current reading will be made using the sample (specimen) current. Also the program will move the stage to the faraday stage position for each beam current measurement. Any non-zero value indicates that the faraday stage is present.

FaradayStagePositionsX=0.0
FaradayStagePositionsY=0.0
FaradayStagePositionsZ=0.0
FaradayStagePositionsW=0.0
FaradayStagePositionsT=0.0
FaradayStagePositionsR=0.0
These parameters allow the user to specify the default stage position (in x, y, z, (w) and tilt and rotation) for the specimen faraday measurement. These values can be updated in the program. These parameters must be in bounds for the x, y, z, (and w) positions and between 0 and 90 for tilt and between 0 and 360 for rotation.

[Counting]

```
OnPeakCountTime=10.0
OffPeakCountTime=5.0
PeakingCountTime=8.0
WavescanCountTime=6.0
QuickscanCountTime=0.5
```

OnPeakCountTime=10.0
This option specifies the default on peak counting time for both standard and unknown quantitative samples. The allowable range is 0.01 to 1000 seconds. The default value is 10 seconds.

OffPeakCountTime=5.0
This option specifies the default high and low off-peak counting time for both standard and unknown quantitative samples. The allowable range is 0.01 to 1000 seconds. The default value is 5 seconds.

PeakingCountTime=8.0
This option specifies the default spectrometer peak counting time for the Probe for Windows interval halving, parabolic or ROM based peak methods. If a spectrometer pre-scan is specified, then Probe for Windows uses a count time of 1/4 of the PeakingCountTime for the spectrometer wavelength pre-scan acquisition. The allowable range is 0.01 to 1000 seconds. The default value is 8 seconds.

WavescanCountTime=6.0
This option specifies the default spectrometer wavelength scan counting time for the Probe for Windows for normal wavescans. The allowable range is 0.01 to 1000 seconds. The default value is 6 seconds.

QuickscanCountTime=0.5
This option specifies the default spectrometer wavelength scan counting time for the Probe for Windows for "quick" spectrometer wavescans. The allowable range is 0.01 to 1000 seconds. The default value is 0.5 seconds.

[PHA]

```
PHAHardwarePresent=1
PHAHardwareType=0
PHAHardwareDeviceName="IOMapper1"
PHAHardwareNumberofBoards=1
PHAHardwareNumberofChannels=16

PHAGainBiasPresent=0
PHAGainBiasType=0
PHAGainBiasDeviceName="IOMapper2"
PHAGainBiasNumberofBoards=1
PHAGainBiasNumberofChannels=16

PHAInteDiffPresent=0
PHAInteDiffType=0
PHAInteDiffDeviceName="IOMapper3"
PHAInteDiffNumberofBoards=1
PHAInteDiffNumberofChannels=16

PHADeadTimePresent=0
PHADeadTimeType=0
PHADeadtimeDeviceName="IOMapper4"
PHADeadtimeNumberofBoards=1
PHADeadtimeNumberofChannels=16

PHACountTime=0.5
PHAIntervals=20
```

PHAHardwarePresent=0

This boolean option specifies whether PHA baseline and window voltage control hardware is implemented. Enter 0 for none or any non-zero value if present. The default is 0 for no PHA baseline and window hardware interface.

PHAHardwareType=0
Specifies the PHA baseline and window hardware type. If the PHA is a "normal" type, then the window voltage is referenced to the baseline voltage. This is the default PHA type.

For example, a baseline of 1.0 volts and a window of 2.0 volts will allow pulses between 1 and 3 volts to be counted. On the other hand, if the PHA is of the "direct" type, then the window voltage is referenced to zero. For example, a baseline of 1.0 volts and a window of 3.0 volts will allow pulses between 1 and 3 volts to be counted.

PHAHardwareType = 0	for normal PHA hardware type (window referenced to baseline) (the default)
PHAHardwareType = 1	for direct PHA hardware type (window referenced to zero)

PHAHardwareDeviceName="IOMapper1"
Indicates the device name of the hardware control DAC output board for PHA baseline and windows operation. Used only for Advanced MicroBeam hardware installations.

Note that if more than one analog output board is required to accommodate all scaler counting channels, then the additional boards must have their base address DIP switches set in 8 byte intervals to yield a consecutive I/O address space. For example, if the first analog output board is set to 768 (300 hex), then the second board must be addressed to 776 (308 hex).

Enter a valid Win NT Registry device name from the IOMapper key under the NT Services key using the IOMapper Control Panel applet. The default value is "IOMapper1".

PHAHardwareNumberofBoards=0
Indicates the number of analog output boards used to control the PHA baseline and window hardware. Note that all boards must have the same number of channels. Used only for Advanced MicroBeam hardware installations. The allowable range is 0 to 8. The default value is 0.

PHAHardwareNumberofChannels=0
Indicates the number of analog output channels on each board used to control the PHA baseline and window hardware. Note that all boards must have the same number of channels. Used only for Advanced MicroBeam hardware installations. The allowable values are 8 or 16. The default is 0.

PHAGainBiasPresent=0
This boolean option specifies whether PHA gain and bias voltage control hardware is implemented. Enter 0 for none or any non-zero value if present. Note that the Gain and Bias interface may be enabled even if the Baseline and Window interface is not. This is to allow the user to leave the PHA baseline and window parameters "wide open" and instead utilize the microprobe's gain and bias adjustments to keep the pulse height properly adjusted. This method is actually preferred for the best quantitative results.

PHAGainBiasType=0
Specifies the PHA gain and bias hardware type. Not used at this time.

PHAGainBiasDeviceName="IOMapper2"
Indicates the device name of the hardware control DAC output board for PHA gain and detector bias operation. Used only for Advanced MicroBeam hardware installations.

Note that if more than one analog output board is required to accommodate all scaler counting channels, then the additional boards must have their base address DIP switches set in 8 byte intervals to yield a consecutive I/O address space. For example, if the first analog output board is set to 768 (300 hex), then the second board must be addressed to 776 (308 hex).

Enter a valid Win NT Registry device name from the IOMapper key under the NT Services key using the IOMapper Control Panel applet. The default value is "IOMapper2".

PHAGainBiasNumberofBoards=0
Indicates the number of analog output boards used to control the PHA gain and bias hardware. Note that all boards must have the same number of channels. Used only for Advanced MicroBeam hardware installations. The allowable range is 0 to 8. The default value is 0.

PHAGainBiasNumberofChannels=0
Indicates the number of analog output channels on each board used to control the PHA gain and bias hardware. Note that all boards must have the same number of channels. Used only for Advanced MicroBeam hardware installations. The allowable values are 8 or 16. The default is 0.

PHAInteDiffPresent=0
This boolean option specifies whether PHA integral/differential control hardware is implemented. Enter 0 for none or any non-zero value if present.

PHAInteDiffType=0
Specifies the PHA integral/differential hardware type. Not used at this time.

PHAInteDiffDeviceName="IOMapper3"
Indicates the device name of the board used for PHA integral/differential control. Used only for Advanced MicroBeam hardware installations.

Note that if more than one board is required to accommodate all scaler counting channels, then the additional boards must have their base address DIP switches set in 8 byte intervals to yield a consecutive I/O address space. For example, if the first board is set to 768 (300 hex), then the second board must be addressed to 776 (308 hex).

Enter a valid Win NT Registry device name from the IOMapper key under the NT Services key using the IOMapper Control Panel applet. The default value is "IOMapper3".

PHAInteDiffNumberofBoards=0
Indicates the number of boards used to control the PHA integral/differential hardware. Note that all boards must have the same number of channels. Used only for Advanced MicroBeam hardware installations. The allowable range is 0 to 8. The default value is 0.

PHAInteDiffNumberofChannels=0
Indicates the number of output channels on each board used to control the PHA integral/differential hardware. Note that all boards must have the same number of channels. Used only for Advanced MicroBeam hardware installations. The allowable values are 8 or 16. The default is 0.

PHADeadtimePresent=0 ! SX-50/51/100 only
This boolean option specifies whether PHA deadtime control hardware is implemented. Enter 0 for none or any non-zero value if present. This flag is usually set true if the hardware interface supports an "enforced" non-extendable deadtime on the system.

PHADeadTimeType=0
Specifies the PHA deadtime hardware type. Not used at this time.

PHADeadtimeDeviceName="IOMapper4"
Indicates the device name of the hardware control output board for PHA deadtime operation. Used only for Advanced MicroBeam hardware installations.

Note that if more than one board is required to accommodate all scaler counting channels, then the additional boards must have their base address DIP switches set in 8 byte intervals to yield a consecutive I/O address space. For example, if the first board is set to 768 (300 hex), then the second board must be addressed to 776 (308 hex).

Enter a valid Win NT Registry device name from the IOMapper key under the NT Services key using the IOMapper Control Panel applet. The default value is "IOMapper4".

PHADeadtimeNumberofBoards=0
Indicates the number of analog output boards used to control the PHA deadtime hardware. Note that all boards must have the same number of channels. The allowable range is 0 to 8. The default value is 0.

PHADeadtimeNumberofChannels=0
Indicates the number of output channels on each board used to control the PHA deadtime hardware. Note that all boards must have the same number of channels. Used only for Advanced MicroBeam hardware installations. The allowable values are 8 or 16. The default is 0.

PHACountTime=.5
Specified the default count time for PHA distributions. The valid range is .1 to 1000 seconds. The default is 0.5 seconds.

PHAIntervals=5
Specifies the default number of PHA measurements (intervals) for PHA distributions. The valid range is 5 to 500 intervals. The default is 20 intervals.

[Plot]

```
MinimumKLMDisplay=0.5
GraphType=1
```

MinimumKLMDisplay=0.01
The default minimum intensity for display of KLM markers with wavescan data. To view fewer low intensity lines, increase this value. The allowable range is 0.01 to 10. The default value is 0.5.

GraphType=1
The default graph type specified in the Plot! window. The default value is 1 for a line graph (lines or lines and symbols), 0 for symbols only, 2 for Linear-Log plot and 3 for 3D plot (three axes).

[Standards]

```
IncrementXForAdditionalPoints = 4
IncrementYForReStandardizations = 6
StandardPointsToAcquire=5
StageBitMapCount=1
StageBitMapFile="JEOL733.WMF"
StageBitMapXmin = 0
StageBitMapXmax = 32
StageBitMapYmin = 0
StageBitMapYmax = 50
STDPOS1="carbide-nitride ceramics and metals"
STDPOS2="silicate minerals and oxides"
STDPOS3="semi-conductors and oxides"
STDPOS4="hydrous silicates and garnets"
STDPOS5="sulfides and elemental metals"
STDPOS6="sulfides and sulpho-salts"
STDPOS7="oxygen and metals analysis"
```

```
STDPOS8="carbonate (CO2) minerals"
STDPOS9=""
STDPOS10=""
STDPOS11=""
STDPOS12=""
STDPOS13=""
STDPOS14=""
STDPOS15=""
STDPOS16=""
```

IncrementXForAdditionalPoints=4
This option is used to specify the stage step size in microns for automatically adjusting the x-axis stage position, if the POSITION.MDB position database contains an insufficient number of digitized coordinates for a specific standard number. The allowable range is -100 to 100 microns. The default value is 0 microns.

IncrementYForReStandardizations=6
This option is used to specify the stage step size in microns for automatically adjusting the y-axis stage when the program is acquiring an additional standard set. The allowable range is -100 to 100 microns. The default value is 0 microns.

StandardPointsToAcquire=5
This option is used to specify the default number of data points to acquire on each standard sample when using the automation features. The allowable values are 1 - 50. The default is 5.

StageBitMapCount=1
This option is used to specify the number of stage bitmap files used for the display of the Stage Bit Map move window. This window, accessible from the Position Database window (see the Stage button) allows the user to indicate a stage position to move to by simply clicking a point on the stage bit map image. Up to MAXBITMAP% .WMF files may be specified on this line.

The remaining stage bitmap keywords in the INI file must contain this number of values separated by commas on each line. Up to MAXBITMAP% Windows Meta-Files may be utilized. Typically, several stage files are specified so that all typical sample mount configurations can be viewed by the user.

StageBitMapFile="JEOL733.WMF"
This line must contain exactly "StageBitMapCount" files separated by commas. The files must be Windows Meta-Files (.WMF). These files may be created using most Windows drawing programs. Up to MAXBITMAP% .WMF files may be specified on this line.

Note that if more than one stage bitmap file is entered on this line, then all files must be enclosed in double quotes. For example : StageBitMapFile="SX50_35.WMF,SX50_12.WMF,SX50_7.WMF".

StageBitMapXmin=0
StageBitMapXmax=30
StageBitMapYmin=0
StageBitMapYmax=40
This option is used to specify the x-axis and y-axis limits of the StageBitMapFile in stage coordinates. It is used to calibrate the stage image for purposes of motion and position sample plotting. The values should correspond to the corners of the image used for the StageBitMapFile. Note that these values are NOT necessarily the stage limits but instead the edges of the stage bit map file as displayed in the WMF file. If the user happens to click a region of the stage image that is outside the stage limits, the program will of course prevent the move.

The program requires that the min and max values be unequal. To achieve the correct polarity for the stage coordinates, the X or Y min and max values may be swapped in the INI file. Up to MAXBITMAP% .WMF coordinates may be specified on this line.

STDPOS1="carbide-nitride ceramics and metals"
STDPOS2="silicate minerals and oxides"
STDPOS3="semi-conductors and oxides"
STDPOS4="hydrous silicates and garnets"
STDPOS5="sulfides and elemental metals"
STDPOS6="sulfides and sulpho-salts"
STDPOS7="oxygen and metals analysis"
STDPOS8="carbonate (CO2) minerals"
STDPOS9=""
STDPOS10=""
STDPOS11=""
etc.

These strings are used to load the standard position string dialog to help the user select the correct ASCII file standard position import file. This dialog will appear when the user clicks the "Import From ASCII File" button in the Automate! window in Probe for Windows. These names are assumed to correspond to coordinate ASCII files with the following naming convention :

STDPOS1 = STDPOS1.POS
STDPOS2 = STDPOS2.POS
STDPOS3 = STDPOS3.POS
STDPOS4 = STDPOS4.POS
STDPOS5 = STDPOS5.POS
STDPOS6 = STDPOS6.POS
etc...

Up to 99 filenames of standard mounts may be specified by the user for use in importing standard position coordinates.

ELEMENTS.DAT

The data file used to define element parameters. This file is normally not edited unless the default oxide cations need to be changed permanently. The file structure consists of 94 lines (one line per element for H to Pu). The lines MUST be in atomic number order!

Each element line contains the element atomic number, lower case symbol, upper case symbol, default x-ray line, default crystal name, atomic weight, default number of oxide cations and default number of oxide oxygens.

The default x-ray lines must be either "ka", "la" or "ma". These are the only allowable x-ray lines for analysis. The CITZAF matrix correction library written by John Armstrong at the California Institute of Technology (and incorporated into Probe for Windows), only supports the use of the Kα, Lα, and Mα x-ray lines to ensure the most quantitative results. The physical parameters of the beta series lines are not known with enough accuracy for use in quantitative analysis. In practice this could be of concern when spectral interferences are present. However, Probe for Windows provides a powerful and completely quantitative spectral interference correction that can correct for interfering lines down to the trace level (Donovan, et al., 1993).

The default crystal names must correspond to a valid entry in the CRYSTALS.DAT file. The number of default oxide cations must be between 1 and 9 and the number of default oxide oxygens must be between 0 and 9.

```
  1  "h "  "H "  "ka" "WC144"    1.00800  2  1
  2  "he"  "He"  "ka" "WC144"    4.00300  1  0
  3  "li"  "Li"  "ka" "WC144"    6.93900  2  1
  4  "be"  "Be"  "ka" "WC144"    9.01200  1  1
  5  "b "  "B "  "ka" "WC144"   10.81100  2  3
  6  "c "  "C "  "ka" "NiCrBN"  12.01100  1  2
  7  "n "  "N "  "ka" "NiCrBN"  14.00700  2  5
  8  "o "  "O "  "ka" "NiCrBN"  16.00000  1  0
  9  "f "  "F "  "ka" "NiCrBN"  18.99800  1  0
 10  "ne"  "Ne"  "ka" "NiCrBN"  20.17900  1  0
 11  "na"  "Na"  "ka" "TAP"     22.99000  2  1
```

```
 12  "mg"  "Mg"  "ka" "TAP"     24.30500  1  1
 13  "al"  "Al"  "ka" "PET"     26.98200  2  3
 14  "si"  "Si"  "ka" "PET"     28.08600  1  2
 15  "p "  "P "  "ka" "PET"     30.97400  2  5
 16  "s "  "S "  "ka" "PET"     32.06400  1  3
 17  "cl"  "Cl"  "ka" "PET"     35.45300  1  0
 18  "ar"  "Ar"  "ka" "PET"     39.94800  1  0
 19  "k "  "K "  "ka" "PET"     39.10200  2  1
 20  "ca"  "Ca"  "ka" "LiF"     40.08000  1  1
 21  "sc"  "Sc"  "ka" "LiF"     44.95600  2  3
 22  "ti"  "Ti"  "ka" "LiF"     47.90000  1  2
 23  "v "  "V "  "ka" "LiF"     50.94200  2  3
 24  "cr"  "Cr"  "ka" "LiF"     51.99600  2  3
 25  "mn"  "Mn"  "ka" "LiF"     54.93800  1  1
 26  "fe"  "Fe"  "ka" "LiF"     55.84700  1  1
 27  "co"  "Co"  "ka" "LiF"     58.93300  1  1
 28  "ni"  "Ni"  "ka" "LiF"     58.71000  1  1
 29  "cu"  "Cu"  "ka" "LiF"     63.54600  2  1
 30  "zn"  "Zn"  "ka" "LiF"     65.37000  1  1
 31  "ga"  "Ga"  "ka" "LiF"     69.72000  2  3
 32  "ge"  "Ge"  "ka" "LiF"     72.59000  1  2
 33  "as"  "As"  "la" "LiF"     74.92200  2  3
 34  "se"  "Se"  "la" "LiF"     78.96000  1  2
 35  "br"  "Br"  "la" "LiF"     79.90400  2  5
 36  "kr"  "Kr"  "la" "LiF"     83.80000  1  0
 37  "rb"  "Rb"  "la" "PET"     85.47000  2  1
 38  "sr"  "Sr"  "la" "PET"     87.62000  1  1
 39  "y "  "Y "  "la" "PET"     88.90500  2  3
 40  "zr"  "Zr"  "la" "PET"     91.22000  1  2
 41  "nb"  "Nb"  "la" "PET"     92.90600  2  5
 42  "mo"  "Mo"  "la" "PET"     95.94000  1  3
 43  "tc"  "Tc"  "la" "PET"     99.00000  1  4
 44  "ru"  "Ru"  "la" "PET"    101.07000  1  2
 45  "rh"  "Rh"  "la" "PET"    102.90500  2  3
 46  "pd"  "Pd"  "la" "PET"    106.40000  1  1
 47  "ag"  "Ag"  "la" "PET"    107.86800  2  1
 48  "cd"  "Cd"  "la" "PET"    112.40000  1  1
 49  "in"  "In"  "la" "PET"    114.82000  2  1
 50  "sn"  "Sn"  "la" "LiF"    118.69000  1  2
 51  "sb"  "Sb"  "la" "LiF"    121.75000  2  3
 52  "te"  "Te"  "la" "LiF"    127.60000  1  2
 53  "i "  "I "  "la" "LiF"    126.90400  2  5
 54  "xe"  "Xe"  "la" "LiF"    131.30000  1  0
 55  "cs"  "Cs"  "la" "LiF"    132.91000  2  1
 56  "ba"  "Ba"  "la" "LiF"    137.34000  1  1
 57  "la"  "La"  "la" "LiF"    138.91000  2  3
 58  "ce"  "Ce"  "la" "LiF"    140.12000  2  3
 59  "pr"  "Pr"  "la" "LiF"    140.90700  2  3
 60  "nd"  "Nd"  "la" "LiF"    144.24000  2  3
 61  "pm"  "Pm"  "la" "LiF"    147.00000  2  3
 62  "sm"  "Sm"  "la" "LiF"    150.35000  2  3
 63  "eu"  "Eu"  "la" "LiF"    151.96000  2  3
 64  "gd"  "Gd"  "la" "LiF"    157.25000  2  3
 65  "tb"  "Tb"  "la" "LiF"    158.92400  2  3
 66  "dy"  "Dy"  "la" "LiF"    162.50000  2  3
 67  "ho"  "Ho"  "la" "LiF"    164.93000  2  3
 68  "er"  "Er"  "la" "LiF"    167.26000  2  3
 69  "tm"  "Tm"  "la" "LiF"    168.93400  2  3
 70  "yb"  "Yb"  "la" "LiF"    173.04000  2  3
 71  "lu"  "Lu"  "la" "LiF"    174.97000  2  3
 72  "hf"  "Hf"  "la" "LiF"    178.49000  1  2
 73  "ta"  "Ta"  "la" "LiF"    180.94800  2  5
 74  "w "  "W "  "la" "LiF"    183.85000  1  3
 75  "re"  "Re"  "la" "LiF"    186.20000  1  2
 76  "os"  "Os"  "la" "LiF"    190.20000  1  2
 77  "ir"  "Ir"  "la" "LiF"    192.20000  1  2
 78  "pt"  "Pt"  "la" "LiF"    195.09000  1  1
 79  "au"  "Au"  "la" "PET"    196.97000  2  3
 80  "hg"  "Hg"  "la" "PET"    200.59000  1  1
 81  "tl"  "Tl"  "ma" "PET"    204.37000  2  3
 82  "pb"  "Pb"  "ma" "PET"    207.19000  1  1
 83  "bi"  "Bi"  "ma" "PET"    208.98000  2  3
 84  "po"  "Po"  "la" "LiF220" 210.00000  1  1
 85  "at"  "At"  "la" "LiF220" 210.00000  1  1
 86  "rn"  "Rn"  "la" "LiF220" 222.00000  1  0
 87  "fr"  "Fr"  "la" "LiF220" 223.00000  2  1
```

```
 88  "ra"  "Ra"  "la" "LiF220" 226.00000  1  1
 89  "ac"  "Ac"  "la" "LiF220" 227.00000  2  3
 90  "th"  "Th"  "ma" "PET"    232.03800  1  2
 91  "pa"  "Pa"  "ma" "PET"    231.00000  2  5
 92  "u "  "U "  "ma" "PET"    238.03000  1  2
 93  "np"  "Np"  "la" "LiF220" 237.00000  2  5
 94  "pu"  "Pu"  "la" "LiF220" 244.00000  1  2
```

CHARGES.DAT

The data file used to define elemental atomic charge parameters. This file is normally not edited unless the default atomic charge defaults need to be changed permanently. The file structure consists of 94 lines (one line per element for H to Pu). The lines MUST be in atomic number order!

Each element line contains the element atomic number, upper case symbol, and the atomic charge. The atomic charge must be between –10 and +10.

CRYSTALS.DAT

The data file used to define crystal parameters. The file structure consists of one line per crystal. Generally this file is edited to specify special analyzing crystals such as layered dispersive elements (LDEs). The maximum number of defined crystals is determined by the MaxCrysType parameter in Probe for Windows (currently set to 32). Each line contains the crystal name, the 2d spacing, the crystal K reflection parameter, followed by the default peaking element and x-ray line (not used at this time).

The crystals can be defined in any order, although ordered by 2d spacing is most convenient for editing purposes.

The spectrometer position of a specific wavelength is calculated according to the following formula. Note that Probe for Windows always assumes a 1st order line for the purposes of the calculation :

$$\sin \phi = \frac{N\lambda}{\left(2d*\left(1-\frac{K}{N^2}\right)\right)}$$

Where : N is the order
d is the d spacing of the analyzing crystal
λ is the wavelength
K is the crystal reflection factor

```
"LiF420"    1.802         0.           "fe"    "ka"
"LiF220"    2.848         0.           "fe"    "ka"
"LIF"       4.0267        0.000058     "fe"    "ka"
"NaCl"      5.641         0.           "fe"    "ka"
"Si"        6.2709        0.           "ca"    "ka"
"Ge"        6.532         0.           "ca"    "ka"
"QTZ011"    6.686         0.           "ca"    "ka"
"QTZ010"    8.50          0.           "ca"    "ka"
"PET"       8.750         0.000144     "ca"    "ka"
"EDT"       8.808         0.           "ca"    "ka"
"ADP"       10.648        0.000476     "ca"    "ka"
"GYPSUM"    15.19         0.           "si"    "ka"
"MICA"      19.8          0.           "si"    "ka"
"KAP"       26.63         0.           "si"    "ka"
"RAP"       26.12         0.           "si"    "ka"
"TAP"       25.745        0.00218      "si"    "ka"
"WSi55"     55.0          0.           "o"     "ka"
"WSi60"     60.0          0.01483      "o"     "ka"
"WSi63"     63.0          0.           "o"     "ka"
"NiCrBN"    65.0          0.           "o"     "ka"
"WC144"     144.0         0.           "c"     "ka"
"PbSTEA"    100.6         0.           "c"     "ka"
"OV 95"     96.8          0.           "c"     "ka"
"OV 200"    197.4         0.           "b"     "ka"
""          0.            0.           ""      ""
""          0.            0.           ""      ""
""          0.            0.           ""      ""
""          0.            0.           ""      ""
""          0.            0.           ""      ""
""          0.            0.           ""      ""
```

```
 ""           0.               0.           ""       ""
 ""           0.               0.           ""       ""
```

MOTORS.DAT

The data file used to define motor parameters. The motor array size (the number of data values on each line) is defined as the sum of the "NumberOfTunableSpecs" and the "NumberOfStageMotors" as specified in the PROBEWIN.INI file. **Note that a comment string enclosed in double quotes, MUST be present at the end of each line.** Several unused parameter lines are at the end of the file for future expansion. Note that the tunable spectrometer motors must be listed first on each line, followed by the stage motors.

```
     "1"       "2"       "3"       "4"       "X"       "Y"       "Z"   "motor labels"
   1.050     1.050     1.050     1.050      5.00     10.00      5.00   "low limit spect/stage motors"
   3.785     3.785     3.785     3.785      610.      595.      320.   "high limit spect/stage motors hilim"
148571.4  148571.4  148571.4  148571.4 3333.3333 3333.3333 3333.3333   "unit to steps conversion"
     1.0       1.0       1.0       1.0     100.0     100.0     100.0   "unit to LiF200 angstrom or micron conversion"
      10        10        10        10        20        20        20   "completion step tolerance for motors"
    400.      400.      400.      400.      400.      400.      400.   "backlash size, (hilimit - lolimit)/backlash size"
     200       200       200       200       200       200       200   "derivative gain"
     200       200       200       200       200       200       200   "integral gain"
     100       100       100       100       100       100       100   "integration limit"
    2000      2000      2000      2000      2000      2000      2000   "following error"
      80        80        80        80       200       200       200   "acceleration"
  240000    240000    240000    240000    400000    400000    400000   "velocity"
     600       600       600       600       600       600       600   "gain"
      11        11        11        11        11        11        11   "hard limit mode (low xor high xor smooth)"
     1.0       1.0       1.0       1.0       1.0       1.0       1.0   "joystick sensitivity"
     1.5       1.5       1.5       1.5       1.5       1.5       1.5   "joystick acceleration"
    .002      .002      .002      .002      .002      .002      .002   "backlash tolerance"
   3.785     3.785     3.785     3.785      320.      270.       20.   "park positions"
       0         0         0         0         0         0         0   "unused"
       0         0         0         0         0         0         0   "unused"
       0         0         0         0         0         0         0   "unused"
       0         0         0         0         0         0         0   "unused"
       0         0         0         0         0         0         0   "unused"
```

Line 1 (spectrometer and stage motor labels)

```
     "1"       "2"       "3"       "4"       "X"       "Y"       "Z"   "motor labels"
```

The motor labels are used to load several dialog controls to allow the user to select a specific motor axis for a specified operation. The motor labels must be enclosed in double quotes.

Line 2 (motor low limits)

```
   1.050     1.050     1.050     1.050      5.00     10.00      5.00   "low limit spect/stage motors"
```

The motor low limit positions are used to define a software low limit before the electrical limit switch position has been reached. This is to prevent the user from accidentally moving to a position outside the normal range of motion. Note that the JOYWIN program ignores these software limits to allow the user to manually drive to the limit switch position for the purposes of manually flipping a spectrometer crystal. The motor low limits are defined in spectrometer and stage units.

Line 3 (motor high limits)

```
   3.785     3.785     3.785     3.785      610.      595.      320.   "high limit spect/stage motors hilim"
```

The motor high limit positions are used to define a software high limit before the electrical limit switch position has been reached. This is to prevent the user from accidentally moving to a position outside the normal range of motion. Note that the JOYWIN program ignores these software limits to allow the user to manually drive to the limit switch position for the purposes of manually flipping a spectrometer crystal. The motor high limits are defined in spectrometer and stage units. The motor high limits must be greater than the motor low limits.

Line 4 (spectrometer and stage units to motor step conversion factors)

```
148571.4  148571.4  148571.4  148571.4 3333.3333 3333.3333 3333.3333   "unit to steps conversion"
```

The step conversion factors indicate the motor "step conversion" factors for each motor in the system for converting from spectrometer or stage units to motor controller steps. Spectrometer motors first followed by stage motors. The conversion factors must be not equal to zero. If it is necessary to reverse the directionality of the motor (other than the stage rotation motor) simply change the sign of the factor or reverse the wires on the servo motor. The motor conversion factors are defined below :

"step conversion" = "motor steps" per "probe motor unit distance"

For example, if the microprobe X axis stage is calibrated in millimeters and it requires 10,000 motor steps to move 0.1 millimeter, then the motor conversion factor for that motor will be 100000.

Note that is the TRACOR interface type is specified (InterfaceType = 4), then this parameter must be set to 200 for each spectrometer motor for the ROM peaking option to operate correctly.

Line 5 (spectrometer and stage units to angstrom or micron conversion factors)

```
     1.0       1.0       1.0       1.0     100.0     100.0     100.0   "unit to LiF200 angstrom or micron conversion"
```

These parameters define the conversion factor for converting from spectrometer units to angstroms (assuming an LiF crystal present) or stage units to microns. The conversion factors must not be equal to zero.

Note that although almost any spectrometer units may be used (sin theta, l-units, Bragg angle, etc.) , the spectrometer units chosen by the user MUST be directly proportional to angstrom units. For this reason, the program cannot utilize energy units (for example KeV) since these units are inversely proportional to angstrom units.

Microprobe	Spectrometer	Stage
ARL SEMQ	1.0	100.0
JEOL	0.014381	1000.0
Cameca	4.0267 E-5	1.0

The spectrometer conversion factors are used to convert spectrometer position to angstroms for wavescan plotting. The stage motor conversion factors are used to calculate stage coordinates in microns. This allows the program to calculate output of traverse data in relative microns. For example if the stage is read in mm units, the stage units to micron conversion factor will be 1000.

Note that some interfaces (Elionix) where the Rowland circle are variable, the conversion factor is calculated on the fly and this parameter is therefore only "nominal".

Some other helpful expressions :

ARL SEMQ Crystal Conversion factors (2d/LiF2d)

Angstroms = 2d/LiF2d * Spectrometer Reading (LiF2d = 4.0267)

JEOL L-Unit Crystal Conversion factors (d/R, where R = 140.0)

Angstroms = d/R * Spectrometer Reading

Cameca Sinϕ Crystal Conversion factors (2d)

Angstroms = 2d * Spectrometer Reading

Elionix Crystal Conversion factor (where R is the Rowland circle and depends on the crystal type)

Angstroms = 2d/2R * Spectrometer Reading

```
Rowland LIF = 200.00 mm       (2d = 4.0267 A)
```

```
Rowland PET = 200.00 mm       (2d = 8.742 A)
Rowland TAP = 200.00 mm       (2d = 25.757 A)
Rowland LSM = 185.00 mm       (2d = 80.000 A)
```

Line 6 **(motor step tolerance)**

```
     10        10        10        10        20        20        20   "completion step tolerance for motors"
```

These parameters define the motor step tolerances for a motion completion. This parameter is only used by the Advanced MicroBeam hardware servo motor interface. The values must be greater than zero.

Normally the values entered should be the largest values that can be tolerated to reduce the time required to "home" the motor. Although a properly adjusted servo loop can usually converge within one encoder step, the resulting positional precision may not actually be necessary.

Line 7 **(backlash size parameter)**

```
   400.      400.      400.      400.      400.      400.      400.   "backlash size, (hilimit - lolimit)/backlash size"
```

These parameters define the size of the backlash motions used for the spectrometer and stage motors. The absolute value of each backlash size parameter must be greater than 10.0. Note that the larger the value, the smaller the resulting backlash motion will be. The calculation for the backlash size is :

$$B_{Size} = \frac{ABS(P_{HiLim} - P_{LoLim})}{B_{factor}}$$

Where :	B_{Size}	is the size of calculated backlash motion in motor units
	P_{HiLim}	is the motor high limit
	P_{LoLim}	is the motor low limit
	B_{factor}	is the backlash size parameter for the motor

Note that if the backlash size parameter is negative, all wavelength scans will scan from the low end to the high end and, if the backlash size parameter is positive, all wavelength scans will scan from the high end to the low end. In addition, each motor (spectrometer or stage) can have either polarity. That is, one spectrometer can have a negative backlash, while the others have positive backlash values.

Normally the backlash factor for each motor should be selected to produce the smallest backlash size that can be tolerated to reduce the time required for the backlash correction.

Note that the program will attempt to determine if a backlash is required based on the last direction of motion for each motor. If the backlash factor is positive and the last motion was from a higher to a lower position and the size of the motion was larger than the calculated backlash size, then a backlash will not be performed on the next occurrence of the backlash adjustment for that motor.

Line 8-15 **(DCX servo loop tuning parameters)**

```
    200       200       200       200       200       200       200   "derivative gain"
    200       200       200       200       200       200       200   "integral gain"
    100       100       100       100       100       100       100   "integration limit"
   2000      2000      2000      2000      2000      2000      2000   "following error"
     80        80        80        80       200       200       200   "acceleration"
 240000    240000    240000    240000    400000    400000    400000   "velocity"
    600       600       600       600       600       600       600   "gain"
     11        11        11        11        11        11        11   "hard limit mode (low xor high xor smooth)"
```

These motor parameters are used only by the Advanced MicroBeam hardware and allow the user to "tune" the servo loop parameters to modify the motor response. The last parameter, the hard limit mode, should not be modified. The values must be greater than zero.

The motor parameters should not normally be modified. For details, see the PMC DCX documentation or contact Advanced MicroBeam.

If the motor stalls due to a mechanical problem, a following error is generated. The following error is the maximum allowable difference in motor steps between the actual position and the calculated trajectory position. If the total magnitude of the move is smaller than the following error tolerance, and the motor stalls, the program will instead generate a motor time out error message.

The hard limit mode flag is used to determine the method used to stop a spectrometer or stage motor in case an electrical limit switch is reached. The value must be either 7 (abrupt stop) or 11 (smooth stop).

Line 16-17 (joystick sensitivity and acceleration)

```
     1.0       1.0       1.0       1.0       1.0       1.0       1.0   "joystick sensitivity"
     1.5       1.5       1.5       1.5       1.5       1.5       1.5   "joystick acceleration"
```

These motor parameters are used to allow the user to "tune" the joystick for sensitivity and acceleration for each motor axis. The values must be greater than zero. The expression used to calculate the joystick response is shown here :

$$J_m = J_m \cdot (J_s)^{J_a}$$

where : J_m is the joystick move size
J_s is the joystick sensitivity
J_a is the joystick acceleration

Line 18 (backlash tolerance)

```
    .002      .002      .002      .002      .002      .002      .002   "backlash tolerance"
```

These motor parameters are used to allow the user to "tune" the backlash tolerance which the program uses to decide whether to apply a "smart" backlash to the current move. The default is 0.002 (0.2%) and the values must be between 0.1 and 0.000001. Use a smaller value to have the program apply the backlash more often.

Line 19 (park positions)

```
   3.785     3.785     3.785     3.785      320.      270.       20.   "park positions"
```

These motor parameters are used to set the "park" positions for the spectrometer and stage motors. The park spectrometer and park stage buttons are available from the JoyWin application. If no position is entered (read zero), then the motor hilimit is used for the spectrometer park positions and the midway point between the hilimit and lolimit is used for the stage park positions. The "wmotor", if available, is ignored for the park position procedure.

The stage park position can be specified so as to be used for the instrument sample exchange position if desired.

Line 20-25 (not used at this time)

```
       0         0         0         0         0         0         0   "unused"
       0         0         0         0         0         0         0   "unused"
       0         0         0         0         0         0         0   "unused"
       0         0         0         0         0         0         0   "unused"
       0         0         0         0         0         0         0   "unused"
```

SCALERS.DAT

The data file used to define scaler parameters. The scaler array size (the number of data values on each line) is defined as the sum of the "NumberofFixedSpecs" and the "NumberOfTunableSpecs" in the PROBEWIN.INI file. The fixed spectrometer scalers (if any) must precede the tunable spectrometer scaler on each line. **Note that a comment string enclosed in double quotes MUST be present at the end of each line.** Several unused parameter lines are at the end of the file for future expansion.

```
  "Si"     "Fe"     "Ca"     "Al"     "1"      "2"      "3"      "4"       "scaler labels"
  "si"     "fe"     "ca"     "al"     ""       ""       ""       ""        "fixed scaler elements"
  "ka"     "ka"     "ka"     "ka"     ""       ""       ""       ""        "fixed scaler x-rays"
  0        0        0        0        0        0        0        0         "crystal flipping flag"
  0.       0.       0.       0.       0.       0.       0.       0.        "crystal flipping position"
  1        1        1        1        2        2        2        2         "number of crystals"
  "ADP"    "LiF"    "LiF"    "PET"    "ADP"    "LiF"    "TAP"    "LiF220"  "crystal types1"
  ""       ""       ""       ""       "NiCrBN" "ADP"    "LiF"    "PET"     "crystal types2"
  ""       ""       ""       ""       ""       ""       ""       ""        "crystal types3"
  ""       ""       ""       ""       ""       ""       ""       ""        "crystal types4"
  ""       ""       ""       ""       ""       ""       ""       ""        "crystal types5"
  ""       ""       ""       ""       ""       ""       ""       ""        "crystal types6"
```

```
 1.0      1.0      1.0      1.0      1.0      1.0      1.0      1.0       "deadtime in microseconds"
 0.       0.       0.       0.       90.      90.      90.      90.       "off-peak size, (hilimit - lolimit)/off-peak size"
 0.       0.       0.       0.       60.      60.      60.      60.       "wavescan size, (hilimit - lolimit)/wavescan size"
 0.       0.       0.       0.       80.      80.      80.      80.       "peakscan size, (hilimit - lolimit)/peakscan size"
 0.       0.       0.       0.       100      100      100      100       "wavescan steps"
 0.       0.       0.       0.       40       40       40       40        "peakscan steps"
 0.       0.       0.       0.       0.001    0.001    0.001    0.001     "LiF peaking start size"
 0.       0.       0.       0.       0.0002   0.0002   0.0002   0.0002    "LiF peaking stop size"
 0        0        0        0        30       30       30       30        "maximum peaking cycles"
 0.       0.       0.       0.       5.       5.       5.       5.        "minimum peak to background"
 0.       0.       0.       0.       10.      10.      10.      10.       "minimum peak intensity"
 1.       1.       1.       1.       1.       1.       1.       1.        "default PHA baseline voltages"
 10.      10.      10.      10.      10.      10.      10.      10.       "default PHA window voltages"
32.      64.      64.      32.      16.      16.      64.      32.         "default PHA gain"
 1.       1.       1.       1.       1.       1.       1.       1.        "default detector bias"
 1.       1.       1.       1.       1.       1.       1.       1.        "PHA baseline full scale factors"
 1.       1.       1.       1.       1.       1.       1.       1.        "PHA window full scale factors"
 1.       1.       1.       1.       1.       1.       1.       1.        "PHA gain scale factors"
 1.       1.       1.       1.       1.       1.       1.       1.        "Detector bias scale factors"
 127.     127.     127.     127.     127.     127.     127.     127.      "Roland Circle (mm)"
 0        0        0        0        0        0        0        0         "Crystal flip delays"
 0        0        0        0        400.     400.     400.     400.      "Spec offset warning factors"
 0        0        0        0        0        0        0        0         "Cameca integer deadtimes"
 0        0        0        0        0        0        0        0         "unused"
 0        0        0        0        0        0        0        0         "unused"
 0        0        0        0        0        0        0        0         "unused"
 0        0        0        0        0        0        0        0         "unused"
 0        0        0        0        0        0        0        0         "unused"
```

Line 1 (scaler labels)

```
 "Si"     "Fe"     "Ca"     "Al"     "1"      "2"      "3"      "4"       "scaler labels"
```

These scaler labels are used to load several dialog controls to specify the counting channel labels. The scaler labels must be enclosed in double quotes.

Line 2 (fixed spectrometer element labels)

```
 "si"     "fe"     "ca"     "al"     ""       ""       ""       ""        "fixed scaler elements"
```

The fixed spectrometer elements define the element that each fixed spectrometer (if any are present) is tuned to. The element symbol must be a valid element symbol for each fixed spectrometer as defined by the "NumberofFixedSpecs" parameter in the C:\Program Files\Probe for Windows\PROBEWIN.INI file.

Line 3 (fixed spectrometer x-ray labels)

```
 "ka"     "ka"     "ka"     "ka"     ""       ""       ""       ""        "fixed scaler x-rays"
```

The fixed spectrometer x-rays define the x-ray that each fixed spectrometer (if any are present) is tuned to. The x-ray symbol must be a valid element symbol ("ka", "la" or "ma") for each fixed spectrometer as defined by the "NumberofFixedSpecs" parameter in the C:\Program Files\Probe for Windows\PROBEWIN.INI file.

Line 4 (tunable spectrometer crystal flip flags)

```
 0        0        0        0        0        0        0        0         "crystal flipping flag"
```

Indicate the type of automated crystal flipping supported by the hardware. Be aware that flipping crystals within a quantitative run may reduce the accuracy of the analysis. The allowable range of values is 0 to 4.

0	**no automated crystal flipping**
1	**automated crystal flipping in any position**
2	**automated crystal flipping at "crystal flipping position" (Cameca and JEOL 733)**
3	**automated crystal flipping at any position greater than "crystal flipping position"**
4	**automated crystal flipping at any position less than "crystal flipping position"**

Line 5 (tunable spectrometer crystal flip positions)

```
 0.       0.       0.       0.       0.       0.       0.       0.        "crystal flipping position"
```

This line is used to indicate the crystal flipping position used when flipping the crystal (if required). See also "crystal flipping flag" above. The values must be greater than zero and within the mechanical range of the spectrometer. Note that the software will disable the software limit checking when performing a crystal flip since some spectrometer mechanisms require that the crystal flip occur at the motor limit position.

Line 6 (number of crystals on each fixed or tunable spectrometer)

```
 1        1        1        1        2        2        2        2         "number of crystals"
```

This line is used to indicate the actual number of crystals on each fixed or tunable spectrometer. The range of allowable values is 1 to 6. Fixed spectrometers must have only one crystal.

Line 7-12 (crystal types on each fixed or tunable spectrometer)

```
 "ADP"    "LiF"    "LiF"    "PET"    "ADP"    "LiF"    "TAP"    "LiF220"  "crystal types1"
```

```
  ""       ""       ""       ""       "NiCrBN" "ADP"    "LiF"    "PET"     "crystal types2"
  ""       ""       ""       ""       ""       ""       ""       ""        "crystal types3"
  ""       ""       ""       ""       ""       ""       ""       ""        "crystal types4"
  ""       ""       ""       ""       ""       ""       ""       ""        "crystal types5"
  ""       ""       ""       ""       ""       ""       ""       ""        "crystal types6"
```

Use these lines to specify the crystal types on each fixed or tunable spectrometer. The crystal types must be enclosed in double quotes and must match one of the crystal types as defined in the C:\Program Files\Probe for Windows\CRYSTALS.DAT file.

Line 13 (detector deadtime)

```
  1.0      1.0      1.0      1.0      1.0      1.0      1.0      1.0       "deadtime in microseconds"
```

Use these lines to specify the default detector deadtime in micro-seconds. These values are used by the *software* to calculate the deadtime for quantitative analyses. The range of allowable values is from 0.1 to 10 microseconds.

The detector deadtime value specified here is NOT used for setting the non-extendable or "enforced" (DTIM) deadtime for those microprobe hardware that support it (Cameca SX-50/51 only). The non-extendable or "enforced" value deadtime value is instead specified on line 35 below. Note that in this case, because the SX50/51 interface only supports an integer deadtime value, the actual deadtime of the system may vary slightly from the integer values specified. However careful deadtime calibration measurements should be performed (using program StartWin or the optional Excel deadtime acquisiton and calculation spreadsheets provided with the Remote OLE interface option) at the "nominal" integer setting to determine the precise actual deadtime coefficient of the SX50/51 pulse processor, and these should be specified here for the analysis correction routine for the most rigorous deadtime correction possible.

See also the option for the "precision" deadtime correction in the PROBEWIN.INI file under the [Software] section and in the ANALYSIS window options for high count rates.

Line 14-16 (off-peak/wavescan/peakscan offset size parameters)

```
  0.       0.       0.       0.       90.      90.      90.      90.       "off-peak size, (hilimit - lolimit)/off-peak size"
  0.       0.       0.       0.       60.      60.      60.      60.       "wavescan size, (hilimit - lolimit)/wavescan size"
  0.       0.       0.       0.       80.      80.      80.      80.       "peakscan size, (hilimit - lolimit)/peakscan size"
```

Use these lines to specify the default off-peak, wavescan and peakscan offset sizes respectively. These factors are the fraction of the total spectrometer range used for the off-peak position and wavelength scan ranges for x-ray lines that use an LiF (200) analyzing crystal and are at the high theta spectrometer limit.

X-ray lines analyzed using other crystals and at lower thetas will have their off-peak and wavelength scan ranges adjusted for the 2d spacing of the crystal and the spectrometer position. The actual expression used is shown below for (in this example) calculating the default off-peak positions. Of course all off-peak and wavelength scan ranges may be changed at anytime by the user within the run.

$$P_{OffPeak} = P_{OnPeak} \pm \frac{ABS(P_{HiLim} - P_{LoLim})}{O_{factor}} \sqrt[3]{\frac{2d_{OnPeak}}{2d_{LiF\,200}}} (*2.0) \sqrt{\frac{P_{LoLim} + ABS(P_{HiLim} - P_{LoLim})}{P_{OnPeak}}}$$

Where : P_{OffPeak}	is the calculated default off-peak position
P_{OnPeak}	is the spectrometer on-peak position
P_{HiLim}	is the spectrometer high limit
P_{LoLim}	is the spectrometer low limit
$2d_{OnPeak}$	is the 2d spacing of the analyzing crystal
$2\ \mathrm{d}_{\mathrm{LiF\,200}}$	is the 2d spacing for LiF (4.0267)
O_{factor}	is the off-peak/wavescan/peakscan factor

Note the (*2.0) factor, which is applied if the 2d spacing of the crystal is 30 angstroms or larger in order to account for the poorer resolution of pseudo-crystals such as stearate or layered dispersive element (LDE) analyzers.

Line 17-18 (number of wavescan or peakscan steps)

```
  0.       0.       0.       0.       100      100      100      100       "wavescan steps"
  0.       0.       0.       0.       40       40       40       40        "peakscan steps"
```

These parameters are used to define the default wavescan and peakscan (pre-scan before peak centering) number of steps. The number of steps must be greater than 5 and the maximum number of peakscan steps must be less than or equal to MaxRow%.

Line 19-20 (spectrometer peak center start and stop sizes)

```
  0.      0.      0.      0.      0.001   0.001   0.001   0.001     "LiF peaking start size"
  0.      0.      0.      0.      0.0002  0.0002  0.0002  0.0002    "LiF peaking stop size"
```

These parameters are used to define the default start and stop step sizes which is used for all three types of spectrometer peaking (interval, parabolic and ROM based).

For interval halving spectrometer peak centering, the peaking start size is the spectrometer step size used to begin the interval halving peak center assuming an LiF (200) crystal and a theta at the highest spectrometer setting. For example, K kα on an LiF crystal (SEMQ probe) or Ti kα on a LiF crystal (Cameca probe). Each time the interval halving peaking procedure reverses direction, the interval size is halved. When the interval size is less than the peaking stop size the spectrometer is considered to be properly peaked.

For parabolic fit peak centering, the peaking start size determines the offset used to measure the high and low side peak intensities. The peaking start size should be adjusted to yield an intensity approximately 1/2 the peak intensity for both the high and low side of the peak, to obtain a good fit to the peak shape. If the parabolic fit spectrometer positions need adjustment, the program shifts the middle, high and low positions by 1/2 the peaking start size and tries again.

For the ROM based peak centering (if available), the peaking start size is again utilized for best results, depending on the interface type.

JEOL 8900 Direct (InterfaceType=2)- The "Peaking Start Size" is not used. Instead a peak parameter of 1 (fine scan) is always used.

Sesame (InterfaceType=3)- The "Peaking Start Size" is not used by the Sesame interface. The function simply sends the "PEAK" command for the specified motor, at the current position.

Tracor (InterfaceType=4)- The Tracor peak center procedure uses a two step peak center (an initial peak center based on the "Peaking Start Size" and a final peak center based on the "Peaking Stop Size"). The total number of steps is hard-coded since the PAC cannot update this system parameter while another spectrometer is being peaked.. The number of TRACOR motor steps per peak scan point is calculated from the number of motor steps per l-unit conversion factor (from the MOTORS.DAT file), the "Peaking Start Size", (or) the "Peaking Stop Size" and the number of peak scan steps (hard-coded to 40). The following expression is used to calculate this value, and assumes a TRACOR peak scan width equal to ten times the Peaking Size :

$$sp = M * P * 10 / N$$

where sp is the Tracor ROM peak center scan motor steps per point
M is the motor steps to units conversion factor (from MOTORS.DAT)
P is the Peaking Start Size (initial) and Peaking Stop Size (final)
N is the number of peak scan steps (hard-coded to 40)

The Tracor step count time is calculated from the Peaking Count Time (divided by four). Note that the PAC Crystal Factor variable must be set to 1 on all spectrometer-crystals for this procedure to perform correctly.

After the initial Tracor peak center has completed, the procedure moves the motor position to the calculated centroid returned from the PAC, and a final Tracor peak center is performed using the "Peaking Stop Size" parameter to calculate the motor steps/point, before the new peak intensity is measured.

SX100 (InterfaceType=5)- The "Peaking Start Size" is modified to produce a number between 0 and 4 by utilizing the following expression :

$$pw = (M_{high} - M_{Low}) * P / 3000000$$

where	pw	is the SX100 peak center width
	M_{high}	is the motor high limit
	M_{low}	is the motor low limit
	P	is the Peaking Start Size

SX50 (InterfaceType=6)- The "Peaking Start Size" is modified to produce a number between 0 and 4 by utilizing the following expression.

$$pw = (M_{high} - M_{Low}) * P / 3000000$$

where	pw	is the SX50 peak center width
	M_{high}	is the motor high limit
	M_{low}	is the motor low limit
	P	is the Peaking Start Size

If the SX50 peak center width is less than or equal to 2, the SX50 driver sends the "PE" command for the specified motor. Otherwise, if the SX50 peak center width is greater or equal to 3, the SX50 driver sends the "PE WIDE" command instead.

JEOL 8900 Direct (InterfaceType=7)- The "Peaking Start Size" is not used. Instead a peak parameter of 1 (fine scan) is always used.

The program uses a larger start and stop size for lower thetas and larger crystal 2d spacings. A smaller stop peaking step size will force the program to more carefully adjust the spectrometer position, however this will require more time to achieve. Be sure that the start peaking size is large enough for the program to measure a significant count change. The start values must be larger than 1/6000th of the total spectrometer range. The stop peaking size must be less than the start peaking size. The actual calculation for a specific peak position for a specific crystal is shown here :

$$S_{actualstart} = S_{start} \bullet \sqrt[3]{\frac{2d_{OnPeak}}{2d_{LiF\,200}}} (*2.0) \sqrt{\frac{P_{LoLim} + ABS\,(P_{HiLim} - P_{LoLim})}{P_{OnPeak}}}$$

Where :	$S_{actualstart}$	is the actual calculated step size
	S_{start}	is the spectrometer default start or stop size
	P_{HiLim}	is the spectrometer high limit
	P_{LoLim}	is the spectrometer low limit
	P_{OnPeak}	is the spectrometer on-peak position
	$2\,d_{OnPeak}$	is the 2d spacing of the analyzing crystal
	$2\,d_{LiF\,200}$	is the 2d spacing for LiF (4.0267)

Some suggested start and stop peaking sizes for various instruments for efficient peaking:

Instrument	Spectrometer Units	Start Size	Stop Size
ARL SEMQ	angstroms (LiF)	.00085	.00015
JEOL 733	L-units	.04	.004
JEOL 8600	L-units	.05	.005

Cameca SX-50/51	Sinϕ * 10^5	20	2
Cameca MBX	Sinϕ	.0002	.00002

Line 21-23 **(peaking cycles, P/B and minimum intensities)**

```
  0        0        0        0        30       30       30       30        "maximum peaking cycles"
  0.       0.       0.       0.       5.       5.       5.       5.        "minimum peak to background"
  0.       0.       0.       0.       10.      10.      10.      10.       "minimum peak intensity"
```

These parameters are used to define additional peak centering default parameters for the interval halving and parabolic peak center. The maximum peaking cycles is used to specify the maximum number of interval halving attempts the program will make to find the peak center. If the number of peaking cycles is small or the peak start size is small or the peak is too far from the starting position, the peak center may fail. The allowable range is 5 to 50 maximum peak cycles.

The minimum peak to background is the minimum peak to background ratio that the program will attempt a peak center on. The values must be greater than 1.5.

The minimum peak intensity is the minimum peak intensity in counts per second that the program will attempt a peak center on. The values must be greater than 10 counts per second.

Line 24-25 **(PHA baseline, window default voltages)**

```
  1.       1.       1.       1.       1.       1.       1.       1.        "default PHA baseline voltages"
  10.      10.      10.      10.      10.      10.      10.      10.       "default PHA window voltages"
```

The default baseline and window PHA values are specified on this line. The values must be greater than zero.

Line 26-27 **(PHA gain and bias defaults)**

```
 32.      64.      64.      32.      16.      16.      64.      32.        "default PHA gain"
  1.       1.       1.       1.       1.       1.       1.       1.        "default detector bias"
```

The default gain and bias values are specified on this line. The values must be greater than zero.

Line 28-31 **(PHA baseline, window, gain and bias scale factors)**

```
  1.       1.       1.       1.       1.       1.       1.       1.        "PHA baseline full scale factors"
  1.       1.       1.       1.       1.       1.       1.       1.        "PHA window full scale factors"
  1.       1.       1.       1.       1.       1.       1.       1.        "PHA gain scale factors"
  1.       1.       1.       1.       1.       1.       1.       1.        "Detector bias scale factors"
```

The PHA scale factors are used for PHA hardware that does not accept 0 to 10 volt control voltages. If the PHA hardware does give a 10 volt output for a 10 volt input control voltage then the scale factors will be 1. If the PHA hardware outputs full scale (10 volts) using a lower input control voltage, then the scale factors should be adjusted. For example, if the PHA hardware wants a .33 volt input to output 10 volts, then set the appropriate PHA scale factor to .33 for that channel. The values must be greater than 0.0.

Line 32 **(spectrometer Roland circle radius in mm)**

```
  127.     127.     127.     127.     127.     127.     127.     127.      "Roland Circle (mm)"
```

The values on this line are used to specify the spectrometer Roland circle. These values are not used at this time. The values must be greater than 0.0.

Line 33 **(crystal flip delays in seconds)**

```
  0        0        0        0        0        0        0        0         "Crystal flip delays"
```

The values on this line are used to define the type and duration of the crystal flip delay used to wait for a crystal flip to complete. If the value is -1, then the program will use a hardware "busy" signal from the DCX board (AM hardware only). If the value is greater than zero then the program will simply delay for the duration specified before allowing the program to continue. If the SX50 hardware interface (InterfaceType=6) is used, then the values on this line are ignored.

Line 34 **(spectrometer offset warning factors)**

```
  0        0        0        0        400.     400.     400.     400.      "Spec offset warning factors"
```

The values on this line are used to define the size of the maximum offset for a spectrometer from it's theoretical position for the purposes of printing a warning message to the log window. If the actual spectrometer position offset is less than the spectrometer range (as defined in the MOTORS.DAT file) divided by this spectrometer offset warning factor then the warning will not be printed. The default value is 400.

Line 35 **(Cameca integer deadtimes)**

```
  0        0        0        0        0        0        0        0         "Cameca integer deadtimes"
```

This line is used to explicitly specify the integer deadtime constants used to set the Cameca PHA hardware (non-extendible or "enforced" deadtime. It is the equivalent of the SX50/51 "DTIM" parameter that is set using the "SACQ" command. Therefore this data is used only by the Cameca SX50/51 hardware interface and the values are not accessible from within the program. These integer deadtime values are distinct from the single precision deadtimes specified on line 13 above which are used to perform the actual deadtime correction in the analysis routines.

The typical procedure is to set the deadtimes on the Cameca PHA hardware all to zero and then to measure the "intrinsic" deadtime of the system using a range of beam currents from approximately 10 to 200 nA on a pure metal x-ray line such as Si Ka (PET and TAP) or Ti Ka (PET and LIF). A sufficient counting time should be used to obtain .2% precision or better. The best method is to find the "worst case" deadtime for each spectrometer since the deadtimes may vary somewhat as a function of detector bias and x-ray line energy.

Assume that the "intrinsic" deadtimes measured (when all spectrometers are set to "DTIM"=0) are 2.23, 3.14, 3.45, 3.78 and 2.15. The next step would be to set the DTIM deadtime parameter for each spectrometer to a value large enough to completely "mask" this intrinsic" deadtime, that is values of 3, 4, 4, 4 and 3. Now since these integer deadtime are not accurately set by the Cameca hardware, the operator must now re-run the deadtime calibration measurement using these new values and note the actual deadtimes. In this case depending on the instrument, the measured deadtimes will be somewhat larger, say, 3.75, 4.65, 4.12, 4.89 and 3.32.

In the example just described, the "DTIM" values of 3, 4, 4, 4 and 3 should be entered on line 35 for setting the Cameca hardware and the measured values that correspond to them, that is, 3.75, 4.65, 4.12, 4.89 and 3.32 should be entered on line 13 above to use in the software correction.

The integer "DTIM" deadtime values must be between zero and ten. If a value of zero is read, then the program will load the single precision deadtime from line 13 and truncate to integer each value for setting the Cameca PHA hardware to an approximate value.

Line 36-39   (not used at this time)

```
  0       0       0       0       0       0       0       0        "unused"
  0       0       0       0       0       0       0       0        "unused"
  0       0       0       0       0       0       0       0        "unused"
  0       0       0       0       0       0       0       0        "unused"
```

DETECTORS.DAT

The data file used to define detector parameters. The scaler array size (the number of data values on each line) is defined as the sum of the "NumberofFixedSpecs" and the "NumberOfTunableSpecs" in the PROBEWIN.INI file. The fixed spectrometer scalers (if any) must precede the tunable spectrometer scaler on each line. **Note that a comment string enclosed in double quotes MUST be present at the end of each line.** Several unused parameter lines are throughout the file for future expansion.

```
     "1"         "Scaler Labels"
       3         "Number of Slit Sizes"
 "2.0mm"         "Slit Size Strings[1]"
 "0.5mm"         "Slit Size Strings[2]"
 "0.3mm"         "Slit Size Strings[3]"
      ""         "Slit Size Strings[4]"
      ""         "Slit Size Strings[5]"
      ""         "Slit Size Strings[6]"
      ""         "Slit Size Strings[7]"
      ""         "Slit Size Strings[8]"
      ""         "Slit Size Strings[9]"
      ""         "Slit Size Strings[10]"
      ""         "Slit Size Strings[11]"
      ""         "Slit Size Strings[12]"
       0         "Number of Slit Positions"
      ""         "Slit Position Strings[1]"
      ""         "Slit Position Strings[2]"
      ""         "Slit Position Strings[3]"
      ""         "Slit Position Strings[4]"
      ""         "Slit Position Strings[5]"
      ""         "Slit Position Strings[6]"
      ""         "Slit Position Strings[7]"
      ""         "Slit Position Strings[8]"
      ""         "Slit Position Strings[9]"
      ""         "Slit Position Strings[10]"
      ""         "Slit Position Strings[11]"
      ""         "Slit Position Strings[12]"
       4         "Number of Detector Modes"
"SPC- FPC-"      "Detector Mode Strings[1]"
```

```
"SPC- FPC+"      "Detector Mode Strings[2]"
"SPC+ FPC-"      "Detector Mode Strings[3]"
"SPC+ FPC+"      "Detector Mode Strings[4]"
      ""         "Detector Mode Strings[5]"
      ""         "Detector Mode Strings[6]"
      ""         "Detector Mode Strings[7]"
      ""         "Detector Mode Strings[8]"
      ""         "Detector Mode Strings[9]"
      ""         "Detector Mode Strings[10]"
      ""         "Detector Mode Strings[11]"
      ""         "Detector Mode Strings[12]"
       0         "Number of Unused"
      ""         "Unused Strings[1]"
      ""         "Unused Strings[2]"
      ""         "Unused Strings[3]"
      ""         "Unused Strings[4]"
      ""         "Unused Strings[5]"
      ""         "Unused Strings[6]"
      ""         "Unused Strings[7]"
      ""         "Unused Strings[8]"
      ""         "Unused Strings[9]"
      ""         "Unused Strings[10]"
      ""         "Unused Strings[11]"
      ""         "Unused Strings[12]"
       2         "Slit Size Exchange Flags [0=none, 1=any position, 2=at position]"
       0         "Slit Position Exchange Flags [0=none, 1=any position, 2=at position]"
       1         "Detector Mode Exchange Flags [0=none, 1=any position, 2=at position]"
       0         "Unused Exchange Flags [0=none, 1=any position, 2=at position]"
   320.0         "Slit Size Exchange Positions"
     0.0         "Slit Position Exchange Positions"
     0.0         "Detector Mode Exchange Positions"
     0.0         "Unused Exchange Positions"
   185.0         "Slit Size Exchange Rowlands"
     0.0         "Slit Position Exchange Rowlands"
     0.0         "Detector Mode Exchange Rowlands"
     0.0         "Unused Exchange Rowlands"
       2         "Default Slit Size Indexes [1 to Number of Slit Sizes]"
       1         "Default Slit Position Indexes [1 to Number of Slit Positions]"
       4         "Default Detector Mode Indexes [1 to Number of Detector Modes]"
       1         "Default Unused Indexes [1 to Number of Unused]"
```

Line 1 (scaler labels)

```
     "1"         "Scaler Labels"
```

These scaler labels are used to load several dialog controls to specify the counting channel labels. The scaler labels must be enclosed in double quotes and must be identical to the values in the SCALERS.DAT file.

Line 2 (number of slit sizes)

```
       3         "Number of Slit Sizes"
```

Lines 3-14 (slit size strings)

```
 "2.0mm"         "Slit Size Strings[1]"
 "0.5mm"         "Slit Size Strings[2]"
 "0.3mm"         "Slit Size Strings[3]"
      ""         "Slit Size Strings[4]"
      ""         "Slit Size Strings[5]"
      ""         "Slit Size Strings[6]"
      ""         "Slit Size Strings[7]"
      ""         "Slit Size Strings[8]"
      ""         "Slit Size Strings[9]"
      ""         "Slit Size Strings[10]"
      ""         "Slit Size Strings[11]"
      ""         "Slit Size Strings[12]"
```

Line two indicates the number of valid slit size strings that will follow each scaler label. Do not enclose the number in quotes. The next 12 lines contain the actual slit size strings that will appear in the dialog for the user to choose from. These must be enclosed in double quotes. The actual value that is set by the program is depends on the detector hardware interface. For example, the Elionix interface will send slit size flags corresponding to 2.0mm, 0.5mm and 0.3 mm, while the MicroSpec interface (AM interface) will send values between 0 to 32 for the slit sizes.

Line 15 (number of slit positions)

```
       0         "Number of Slit Positions"
```

Lines 16-27 (slit position strings)

```
      ""         "Slit Position Strings[1]"
      ""         "Slit Position Strings[2]"
      ""         "Slit Position Strings[3]"
      ""         "Slit Position Strings[4]"
      ""         "Slit Position Strings[5]"
      ""         "Slit Position Strings[6]"
      ""         "Slit Position Strings[7]"
      ""         "Slit Position Strings[8]"
      ""         "Slit Position Strings[9]"
      ""         "Slit Position Strings[10]"
      ""         "Slit Position Strings[11]"
      ""         "Slit Position Strings[12]"
```

Line fifteen indicates the number of valid slit position strings that will follow each scaler label. Do not enclose the number in quotes. The next 12 lines contain the actual slit position strings that will appear in the dialog for the user to choose from. These must be enclosed in double quotes. The actual value that is set by the program is depends on the hardware interface. For example the Microspec detector interface (AM interface) will send values between 0 to 256 to specify the slit position.

Line 28 (number of detector modes)

```
      4         "Number of Detector Modes"
```

Lines 29-40 (detector mode strings)

```
"SPC- FPC-"     "Detector Mode Strings[1]"
"SPC- FPC+"     "Detector Mode Strings[2]"
"SPC+ FPC-"     "Detector Mode Strings[3]"
"SPC+ FPC+"     "Detector Mode Strings[4]"
     ""         "Detector Mode Strings[5]"
     ""         "Detector Mode Strings[6]"
     ""         "Detector Mode Strings[7]"
     ""         "Detector Mode Strings[8]"
     ""         "Detector Mode Strings[9]"
     ""         "Detector Mode Strings[10]"
     ""         "Detector Mode Strings[11]"
     ""         "Detector Mode Strings[12]"
```

Line twenty-eight indicates the number of valid detector mode strings that will follow each scaler label. Do not enclose the number in quotes. The next 12 lines contain the actual detector mode strings that will appear in the dialog for the user to choose from. These must be enclosed in double quotes. The actual value that is set by the program is depends on the hardware interface. For example, the Elionix detector interface will send values from 0 to 3 which correspond to SPC off and FPC off, SPC off and FPC on, SPC on and FPC off, and SPC on and FPC on, respectively.

Line 41 (number of unused)

```
      0         "Number of unused"
```

Lines 42-53 (unused strings)

```
     ""         "Unused String[1]"
     ""         "Unused String[2]"
     ""         "Unused String[3]"
     ""         "Unused String[4]"
     ""         "Unused String[5]"
     ""         "Unused String[6]"
     ""         "Unused String[7]"
     ""         "Unused String[8]"
     ""         "Unused String[9]"
     ""         "Unused String[10]"
     ""         "Unused String[11]"
     ""         "Unused String[12]"
```

Lines forty-one to fifty-three are unused for the time being and are reserved for future expansion of the detector parameter interface.

Line 54 (slit size exchange flag)
Line 55 (slit position exchange flag)
Line 56 (detector mode exchange flag)
Line 57 (unused exchange flag)

```
      2         "Slit Size Exchange Flag"
      0         "Slit Position Exchange Flag"
      1         "Detector Mode Exchange Flag"
      0         "Unused Exchange Flag"
```

Lines fifty four to fifty seven are flags that indicate the type of the detector exchange mechanism. The allowable range of values is 0 to 2.

The example lines shown above indicate that the slit size must be exchanged at the specified spectrometer position (and Rowland circle) given below, while the slit position mechanism is not automated, and finally while the detector mode exchange can be automated in any spectrometer position.

0	no automated exchange
1	automated exchange in any position (or Rowland circle)
2	automated exchange only at "exchange position (and Rowland circle)" (Elionix)

Line 58 (slit size exchange position)
Line 59 (slit position exchange position)
Line 60 (detector mode exchange position)
Line 61 (unused exchange position)

```
  320.0         "Slit Size Exchange Position"
    0.0         "Slit Position Exchange Position"
    0.0         "Detector Mode Exchange Position"
    0.0         "Unused Exchange Position"
```

Line 62 (slit size exchange Rowland circle)

Line 63 (slit position exchange Rowland circle)
Line 64 (detector mode exchange Rowland circle)
Line 65 (unused exchange Rowland circle)

```
  185.0         "Slit Size Exchange Rowland"
    0.0         "Slit Position Exchange Rowland"
    0.0         "Detector Mode Exchange Rowland"
    0.0         "Unused Exchange Rowland"
```

Lines fifty eight to sixty five specify the spectrometer position (and Rowland circle) for the automated exchange mechanism. If the value is zero, it will be ignored and assumed to be unnecessary for the exchange. This field is required if the corresponding exchange flag in lines fifty four to fifty six is set to a value of two.

The allowable values must be within the spectrometer range specified in the SCALERS.DAT file. The Rowland circle values may be any non-zero number if it is required to be specified.

Line 66 (Default slit size index)
Line 67 (Default slit position index)
Line 68 (Default detector mode index)
Line 69 (Default unused index)

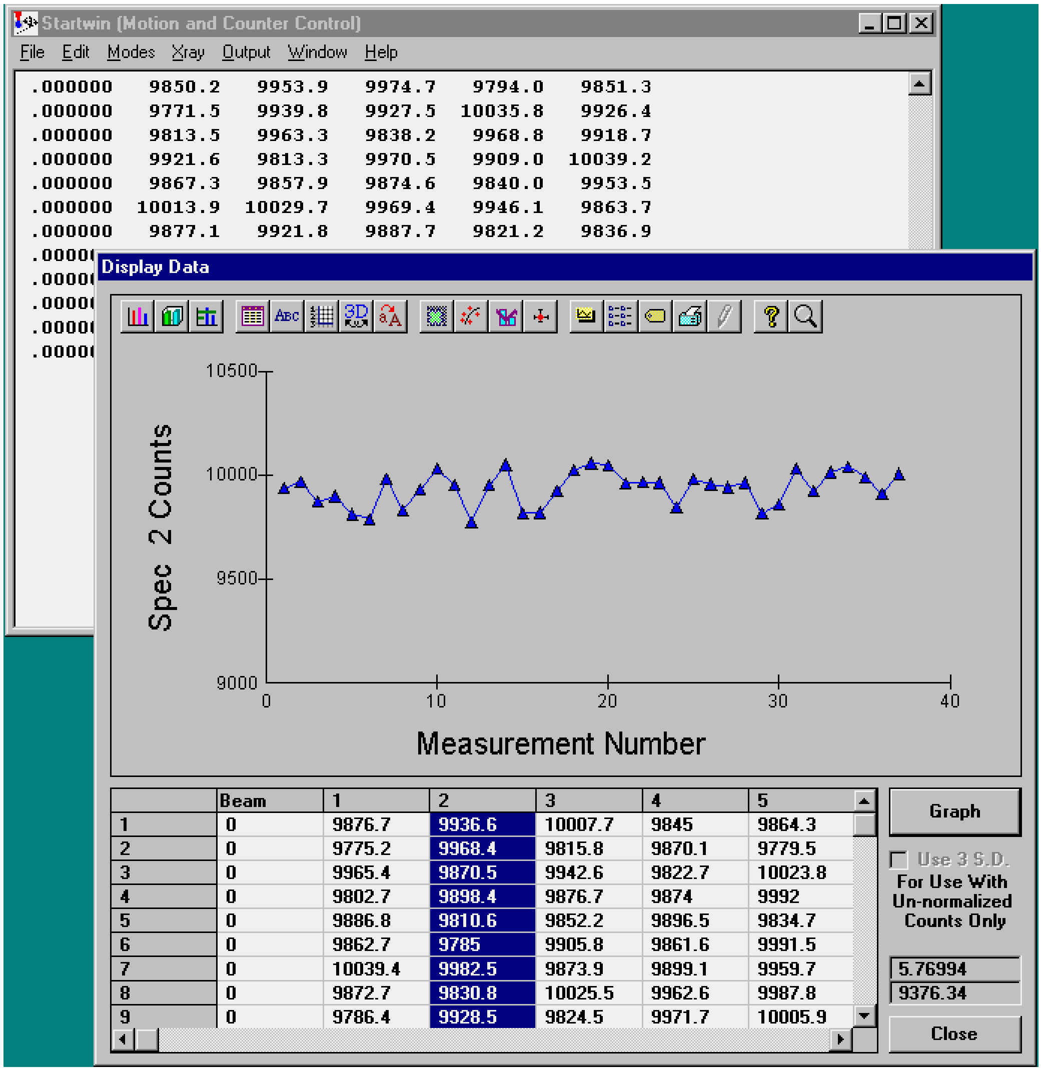
```
      2         "Default Slit Size Index [1 to Number of Slit Sizes]"
      1         "Default Slit Position Index [1 to Number of Slit Positions]"
      4         "Default Detector Mode Index [1 to Number of Detector Modes]"
      1         "Default Unused Index [1 to Number of Unused]"
```

Lines sixty six to sixty nine specify the default detector settings for the given spectrometer scaler number. These are index numbers and must correspond with the slit size, slit position and detector mode strings in the previous sections. Values must be between 1 and the maximum number specified in the "number of slit sizes", "number of slit positions" and "number of detector modes" values.

StartWin

Overview

Program STARTWIN is used to provide a simple interface to the microprobe motion and counting hardware functions. From STARTWIN, the user can move the spectrometer and stage motors, flip spectrometer crystals, blank or unblank the beam and measure x-ray intensities.



```
.000000    9850.2    9953.9    9974.7    9794.0    9851.3
.000000    9771.5    9939.8    9927.5   10035.8    9926.4
.000000    9813.5    9963.3    9838.2    9968.8    9918.7
.000000    9921.6    9813.3    9970.5    9909.0   10039.2
.000000    9867.3    9857.9    9874.6    9840.0    9953.5
.000000   10013.9   10029.7    9969.4    9946.1    9863.7
.000000    9877.1    9921.8    9887.7    9821.2    9836.9
.0000
.0000
.0000
.0000
.0000
```

	Beam	1	2	3	4	5
1	0	9876.7	9936.6	10007.7	9845	9864.3
2	0	9775.2	9968.4	9815.8	9870.1	9779.5
3	0	9965.4	9870.5	9942.6	9822.7	10023.8
4	0	9802.7	9898.4	9876.7	9874	9992
5	0	9886.8	9810.6	9852.2	9896.5	9834.7
6	0	9862.7	9785	9905.8	9861.6	9991.5
7	0	10039.4	9982.5	9873.9	9899.1	9959.7
8	0	9872.7	9830.8	10025.5	9962.6	9987.8
9	0	9786.4	9928.5	9824.5	9971.7	10005.9

5.76994
9376.34

Description

STARTWIN (Start for Windows) is a utility application for general purpose control of the microprobe hardware. It supports stage and spectrometer motion, crystal flipping and scaler counting. It also includes features for peak centering, beam and detector stability, multiple spectrometer wavescans and PHA distribution tests and spectrometer reproducibility routines.

The main window of STARTWIN is a scrollable text window which will record all output and data acquisition. Most of the functionality in STARTWIN is included in the larger Probe for Windows application and help for these functions can be found in the Probe for Windows documentation and help sections. The following specific functions are unique to STARTWIN :

Beam and Detector Stability

To test the beam and/or detector stability select from the Modes menu the data acquisition options desired for the test. Normally to test beam and/or detector stability, one should make sure that the Measure Faraday menu item is checked (even for aperture microprobes) and from the Count Times button in the Count Acquisition window, confirm that the Use Beam Drift Correction check box is unchecked in order to disable the beam drift correction.

It may also be desirable to check the Cycle menu item to continuously acquire data for an extended period of time. Finally, when ready, simply click the Start Count button. After a suitable number of acquisitions have been acquired, click the Cancel button in the Automation Status window to stop the acquisition.

Next, the data may be graphically plotted by using the mouse to select the data in the log window to plot. When the desired data is selected, simply use the Output | Plot Count Data menu to graph the results. By default all data columns are selected, however to plot a single data column simply click the column label of the desired data and click the Graph button. The Use 3 S.D. check box will display +/- 3 standard deviation lines for evaluation purposes if checked.

Spectrometer Reproducibility

To test spectrometer reproducibility, setup the count acquisition as before in the Counter and Detector Stability section, peak the spectrometer or spectrometers to test using the Peak/Scan Options and Start Peak center buttons, and then select the Move to Off Peaks menu item in the Modes menu to enable spectrometer motion.

Again, acquire a sufficient number of data acquisitions, select the data by dragging the mouse and use the Output | Plot Count Data menu to graph the results.

Deadtime Calibration

To calibrate the deadtime constants for each spectrometer, StartWin may be utilized. StartWin is capable of producing a data set that is easily copied and pasted into Excel for use with Paul Carpenter's deadtime spreadsheet template.

First, set a sufficient count time (for example 60 seconds) for a precise measurement of intensities. If your interface supports sample (absorbed) current measurements, select this option also. Turn off the beam drift correction to acquire "raw" intensities. Then tune all spectrometers to the same element, for example Ti Ka on LiF and PET or Si Ka on PET and TAP. Use pure metal standards for high count rates and acquire 5 points at 10 different currents from about 10 nA to 200 nA.

Copy and paste the data set from the StartWin log window into a blank Excel spreadsheet and duplicate the beam current column if the absorbed current column is missing. See DEADTIME.DOC for more information on the data format required by DEADTIME.XLS. Finally create a column of the count time in the proper position (typically 1 second for data acquired by StartWin.

When the data is properly formatted, simply copy and paste the data set into the DEADTIME.XLS spreadsheet. The calculations on the new data set will be automatically updated and plotted. Use these new figures to update the values in the SCALERS.DAT file. Note that the actual calibrated deadtimes are entered on line 13, while the constant integer deadtime constants used to set the PHA interface, are specified on line 35 for the Cameca SX50/51 interface only.

Be aware however that deadtime is actually not a constant and can vary with the line energy of the x-ray being measured. For this reason, it is best if a pulse "stretcher" circuit can be placed in front of the counter-timer board to ensure that a "forced" deadtime is used to mask the actual deadtime of the spectrometer (as is done with the Cameca SX50/51 PHA hardware). If the pulse stretcher is adjusted to produce pulse widths greater than the worst deadtime seen on the spectrometer than the deadtime of the pulse stretcher (which is constant) can be used for the software deadtime correction. This will result in a much more accurate deadtime correction even at very high count rates.

Peak/Scan Option

The Peak/Scan Options dialog allows the user to specify conditions for wavelength scans and peak calibrations (peak centering). To initiate a wavelength scan first indicate the start and stop positions in the Peak/Scan dialog and click OK. Then click the Start Wavescan button to start the spectrometers scanning. Data is saved to the log window and may also be plotted using the Output | Plot Count Data menu or exported to an ASCII file.

Peak centering is started by indicating the on-peak positions in the Peak/Scan dialog and clicking OK and then clicking the Start Peak Center button. Individual spectrometers may be disabled or enabled using the "Yes" checkboxes in the Peak/Scan dialog. Two additional peaking options are provided and described in the next section.

Alignment Peak Center

From the Peak/Scan Option dialog the user may select "Alignment" peak centering. This option causes the program to perform a series of peak center procedures over a range of the Z stage axis. This test is useful in order to determine if the focal circle of the spectrometers are properly aligned. If the maximum intensity is obtained at the z-axis optical focus then the Rowland circle is properly adjusted.

Use the Alignment Parameters button to specify the z-axis range and number of peak calibrations to be performed for each spectrometer. To start the Alignment Peak Center check the Alignment Peak Center checkbox, click OK and click the Start Peak Center button.

Calibrate Peak Center

The Calibrate Peak Center option allows the user to specify a number of elements and corresponding standards for the program to perform multiple peak calibrations. After the peak centering has been performed the program will save the theoretical spectrometer positions along with the actual measured peak positions and a least squares fit to the data for the purpose of calculated an variable offset function for calculating the actual peak position of any element on that spectrometer/crystal combination.

The default constant "offset" from actual spectrometer position is calculated as follows:

$$O_C = P_T - P_A$$

where: P_T = the theoretical spectrometer peak position

P_A = the actual or measured spectrometer peak position

and is always applied unless the user performs the procedures described in this section. The default constant offset is usually sufficient if the mis-calibration of the spectrometer mechanism is constant over the range of motion. However, if the offset varies as a function of spectrometer position, then the multiple peak calibration procedure will allow the use of variable offsets for each spectrometer-crystal combination for highest accuracy.

The multiple peak calibrations for each x-ray family (K, L and M) are stored in separate files which are named PROBEWIN-*X*.CAL where *X* is K, L or M depending on the line type. This due to the fact that slight differences in partially resolved alpha-1,2 peaks behave differently for each line family. There the calibrations for each family are stored separately. This difference for K and L fits can be seen the figure at the end of this section.

Note that each spectrometer/crystal combination should be calibrated using multiple peaks. If less than three elements are specified the program will fit the data to a straight line. If less than two elements are specified the program will use a constant offset for the spectrometer correction which will have a similar accuracy to the default constant offset calculation.

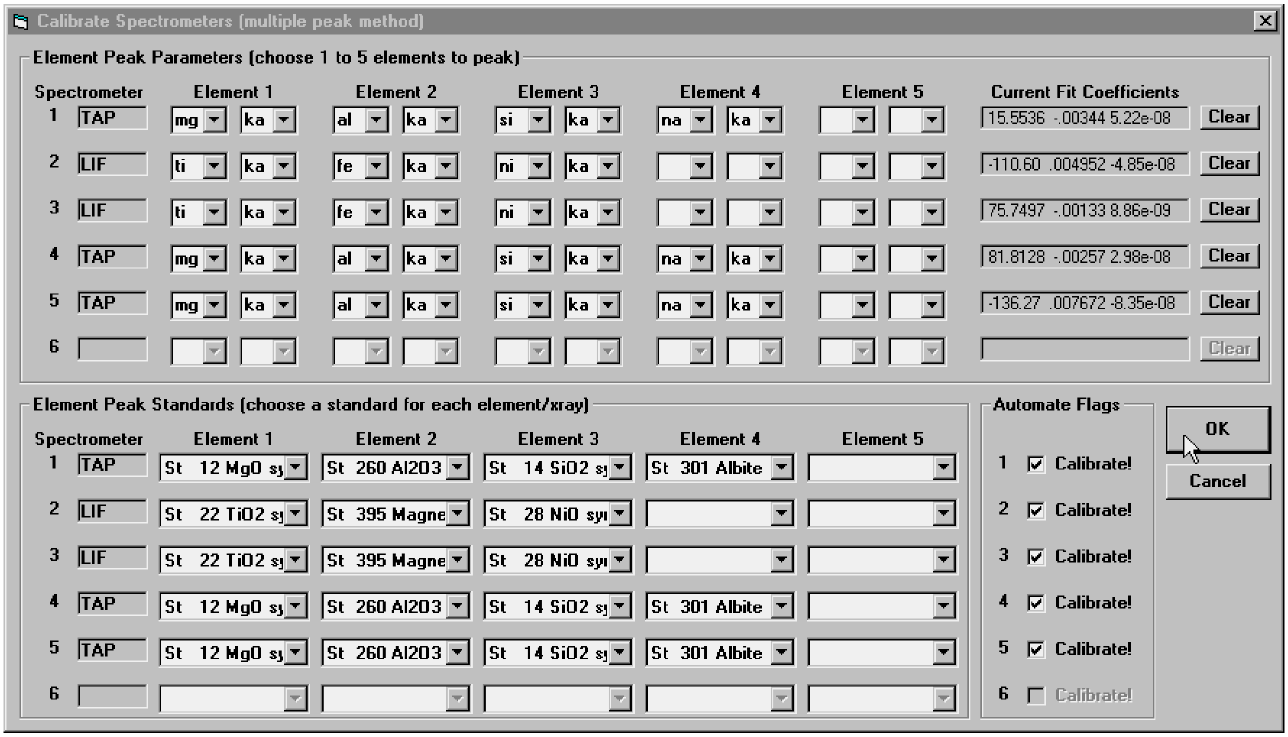
Note also that a two crystal spectrometer will require two separate peak center procedures, one for each spectrometer/crystal combination although all tunable spectrometers can be calibrated automatically in a single run.

See the keyword "UseMultiplePeakCalibrationOffset" in the [software] section of the PROBEWIN.INI file for further instructions. This parameter must be set to a non-zero value to use this feature. Note that the calibration data is saved to an ASCII file called PROBEWIN-*X*.CAL and can be viewed using a text editor, but should not be edited by hand.

When performing multiple peak calibrations be sure to run graphical PHA distribution scans (from the PHA dialog which is accessed using the PHA button) using the lowest energy line specified for each spectrometer/crystal combination to make sure that the detector bias and gain are properly set. The baseline and window settings should be wide enough to allow for a range of energies since the entire spectrometer range will normally be utilized.

In addition, it is recommended that the count time in the Peak/Scan dialog be set to a sufficiently long time to provide proper statistics for the calibration, at least 20-40 seconds. Also each standard must be properly digitized and the position confirmed before the multiple peak calibration procedure is performed (it is important that the optical focus be precisely adjusted for each standard used in the procedure).

Use the Calibrate Parameters button to set the elements and standards for each spectrometer/crystal combination. To start a multiple peak center, check the Calibrate Peak Center checkbox, click OK and click the Start Peak Center button. To perform multiple peak centers on the other crystals, use the Move dialog to flip crystals and use the Calibrate Parameter dialog for each additional crystal.



Calibrate Spectrometers (multiple peak method)

Element Peak Parameters (choose 1 to 5 elements to peak)

Spectrometer		Element 1	Element 2	Element 3	Element 4	Element 5	Current Fit Coefficients	
1	TAP	mg ka	al ka	si ka	na ka		15.5536 -.00344 5.22e-08	Clear
2	LIF	ti ka	fe ka	ni ka			-110.60 .004952 -4.85e-08	Clear
3	LIF	ti ka	fe ka	ni ka			75.7497 -.00133 8.86e-09	Clear
4	TAP	mg ka	al ka	si ka	na ka		81.8128 -.00257 2.98e-08	Clear
5	TAP	mg ka	al ka	si ka	na ka		-136.27 .007672 -8.35e-08	Clear
6								Clear

Element Peak Standards (choose a standard for each element/xray)

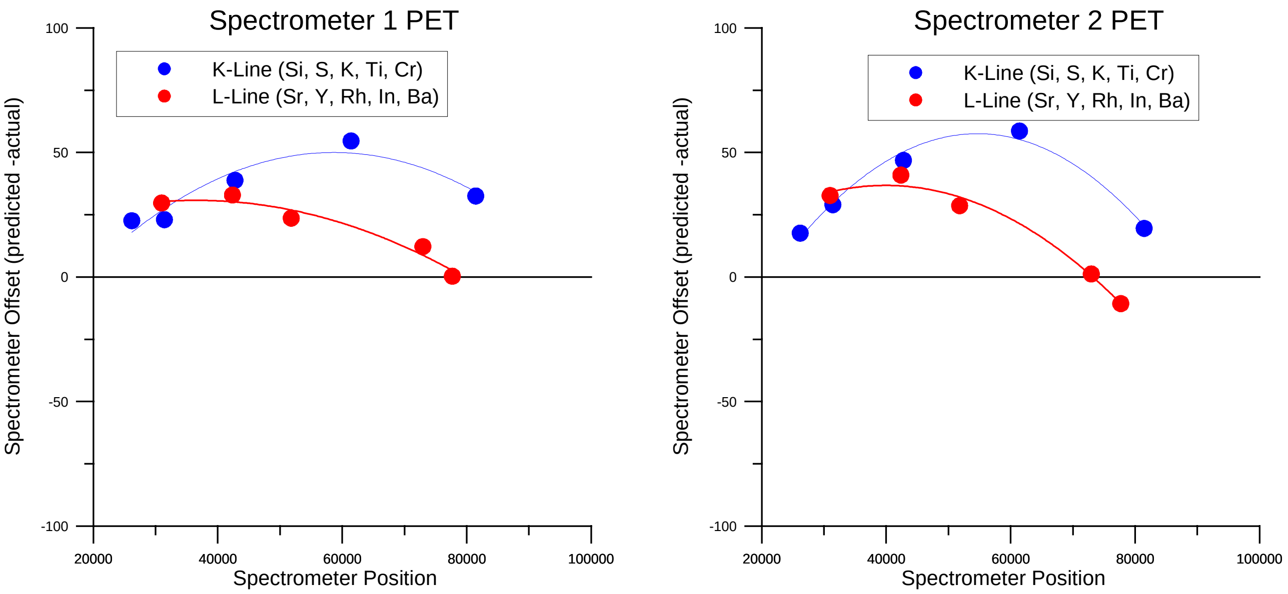
Spectrometer		Element 1	Element 2	Element 3	Element 4	Element 5
1	TAP	St 12 MgO s	St 260 Al2O3	St 14 SiO2 s	St 301 Albite	
2	LIF	St 22 TiO2 s	St 395 Magne	St 28 NiO sy		
3	LIF	St 22 TiO2 s	St 395 Magne	St 28 NiO sy		
4	TAP	St 12 MgO s	St 260 Al2O3	St 14 SiO2 s	St 301 Albite	
5	TAP	St 12 MgO s	St 260 Al2O3	St 14 SiO2 s	St 301 Albite	
6						

Automate Flags

1 ☑ Calibrate!
2 ☑ Calibrate!
3 ☑ Calibrate!
4 ☑ Calibrate!
5 ☑ Calibrate!
6 ☐ Calibrate!

When a spectrometer/crystal combination has been properly calibrated, the program will automatically load the "corrected" peak position for an element based on the fit to the measured spectrometer positions. This may be tested using the element/x-ray target positions in the Move dialog. The "corrected" spectrometer positions will also be utilized in the Elements/Cations dialog of Probe for Windows for new elements not loaded from the element Setup database or another file or sample setup.

Spectrometer Calibration Offsets for K and L lines on PET Crystals



A new modification allows the user to specify the starting peak position for the peak center procedure for each element on each spectrometer. This is especially useful in cases where the theoretical peak position is completely off the peak due to severe misalignment of the spectrometer. The starting peak center position loaded is by default based on the theoretical peak position or the previously stored actual peak position from the .CAL file (if available). Which default starting peak position is loaded depends on whether the Load Peak Center Start From Previous Peak Positions checkbox in the Calibrate Peak dialog box is checked (default=checked).

Multiple Peak Calibration for Spectrometer to Angstroms Calculations

When Probe for Windows calculates the calibrated angstrom of a given spectrometer position, by default the program bases the conversion on the constant offset calculated at the element peak position for each analytical channel using the following standard expression:

$$angstroms = \frac{(P_S + P_O)*K_S*2d*(1-K)}{2d_{LIF}}$$

where: P_S = the position for the spectrometer
P_O = the measured or calibrated spectrometer offset from the theoretical (constant or variable)
K_S = the conversion factor for spectrometer units to LIF angstroms (SCALERS.DAT)
$2d$ = the 2d spacing of the spectrometer crystal
K = the crystal constant (CRYSTALS.DAT)
$2d_{LIF}$ = the 2d for LIF (4.0267)

To calculated a variable spectrometer offset the program utilizes a series of peak center calibration performed in the StartWin application as described above. The multiple peak calibration data is stored in the .CAL files described above and the following is a small part of such a file produced by running a series of peak calibrations on an SX-51 microprobe where the spectrometer positions range roughly from 22000 to 86000.

```
 2                              "PET"            5
"si"             "ka"                81454.48         81426              14
"s"              "ka"                61415.59         61354              327
"k"              "ka"                42776.79         42730              374
"ti"             "ka"                31430.02         31403              22
"cr"             "ka"                26186.62         26175              24
 2               -107.9718           5.904327E-03                  -5.182411E-08
```

The above calibrations are for the PET crystal on spectrometer two using K line intensities. The calibration is based on 5 peak centers using Si, S, K Ti and Cr. These elements are designed to cover the full range of the spectrometer crystal whenever practical. The first column of numbers are the theoretical calculated peak positions and the second column of numbers are the actual or measured peak positions. The offset between these two sets of spectrometer positions is fit to a 2nd order polynomial and the coefficients are stored on the last line of the dataset as can be seen above. The actual application of the variable offset for the purposes of spectrometer position to angstrom conversion is as follows:

First, the program calculates the expected peak position for the calibration element used for the wavescan based on the variable offset obtained from the calibration coefficients measured above using the polynomial expression:

$$P_E = P_{On-Peak} + (K_1 + K_2 * P_{On-Peak} + K_3 * P_{On-Peak}^2)$$

where: K_1, K_2, K_3 = the calibration coefficients from the multiple peak calibration polynomial fit
$P_{On-Peak}$ = the theoretical on peak position of the calibration element

This expected on-peak position from the multiple peak calibration coefficients is used to calculate the difference from the actual measured on-peak position as follows:

$$O_V = P_A - P_E$$

where: P_A = the actual or measured on-peak spectrometer position

P_E = the expected spectrometer position based on the previous calculation

This spectrometer position offset is used to temporarily modify the variable offset calibration intercept coefficient (first term) obtained from the .CAL file as follows:

$$K_1^{'} = K_1 - O_V$$

where: K_1 = the intercept coefficient from the multiple peak calibration polynomial fit

O_V = the difference between the actual on-peak and the expected on-peak from the previous equation

In this way, the modified expected position calculation will obtain a modified variable offset equal to the constant offset when the spectrometer position is at the actual on-peak position, but the calculated offset will still vary based on the slope and curvature terms as the spectrometer position departs from the on-peak position as follows:

$$P_E^{'} = P + (K_1^{'} + K_2 * P + K_3 * P^2)$$

where: $K_1^{'}$ = the modified intercept term of the multiple peak calibration polynomial fit

K_2, K_3 = the slope and curvature coefficients of the multiple peak calibration polynomial fit

P = the spectrometer position

By doing this, the angstrom calculation is based on the multiple peak calibration for high accuracy over the entire spectrometer range, but can still be re-calibrated simply by a single re-peaking of the spectrometer peak position in the future.

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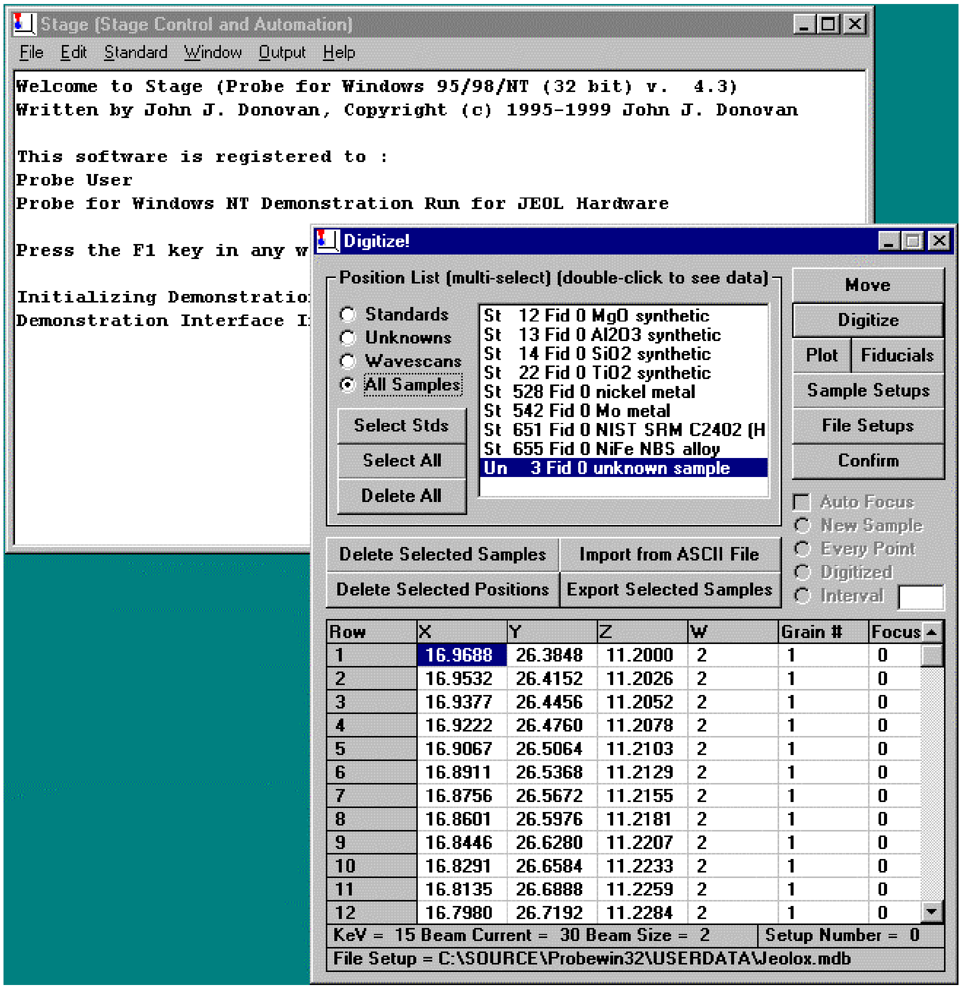
Stage

Overview

Program STAGE.EXE is used to provide a simple interface for digitizing standard, unknown and wavescan position samples and ASCII import position files. Normally, the standard mounts are digitized using Stage for Windows and saved as ASCII .POS files so that they may be imported into the Position database file from the Automate! window of Probe for Windows at any time later. Stage for Windows can also be used for coordinate transformation and confirmation of sample positions.



```
Welcome to Stage (Probe for Windows 95/98/NT (32 bit) v.  4.3)
Written by John J. Donovan, Copyright (c) 1995-1999 John J. Donovan

This software is registered to :
Probe User
Probe for Windows NT Demonstration Run for JEOL Hardware

Press the F1 key in any w

Initializing Demonstratio
Demonstration Interface I
```

```
St   12 Fid 0 MgO synthetic
St   13 Fid 0 Al2O3 synthetic
St   14 Fid 0 SiO2 synthetic
St   22 Fid 0 TiO2 synthetic
St  528 Fid 0 nickel metal
St  542 Fid 0 Mo metal
St  651 Fid 0 NIST SRM C2402 (H
St  655 Fid 0 NiFe NBS alloy
Un    3 Fid 0 unknown sample
```

Row	X	Y	Z	W	Grain #	Focus
1	16.9688	26.3848	11.2000	2	1	0
2	16.9532	26.4152	11.2026	2	1	0
3	16.9377	26.4456	11.2052	2	1	0
4	16.9222	26.4760	11.2078	2	1	0
5	16.9067	26.5064	11.2103	2	1	0
6	16.8911	26.5368	11.2129	2	1	0
7	16.8756	26.5672	11.2155	2	1	0
8	16.8601	26.5976	11.2181	2	1	0
9	16.8446	26.6280	11.2207	2	1	0
10	16.8291	26.6584	11.2233	2	1	0
11	16.8135	26.6888	11.2259	2	1	0
12	16.7980	26.7192	11.2284	2	1	0

KeV = 15 Beam Current = 30 Beam Size = 2   Setup Number = 0

File Setup = C:\SOURCE\Probewin32\USERDATA\Jeolox.mdb

Description

The STAGE application is a simple utility application for digitizing and confirmation of standard, unknown or wavescan coordinates. It is intended specifically for the digitization of pre-digitized standard mounts in the microprobe, but can also be used off-line on a suitably equipped light microscope for digitizing unknown samples referenced to fiducial markings for precise relocation of small features in the electron microprobe.

Most of the functionality in STAGE is included in the larger Probe for Windows application and help for these functions can be found in the Probe for Windows documentation and help sections. The following specific functions are unique to STAGE.

Digitize Positions

The Digitize Positions menu item is found under the Window menu and is a subset of the functions found the Automate! window of Probe for Windows. Although STAGE contains the full Digitize button feature set of Probe for Windows, the only automation action that can be initiated is through the Confirm button. This button will cause the program to automatically proceed to each selected position sample for confirmation of the digitized positions.

Although the Stage application only allows confirmation of position coordinates, it contains the full set of coordinate fiducial and transformation routines and can therefore be utilized on an off-line light microscope for digitizing of standard or unknown samples.

A data field is provided (see PositionImportExportFileType in the PROBEWIN.INI file section) to export and import the sample setup numbers to the .POS ASCII position files in addition to the sample name and coordinates. If this option is utilized, care must be taken to ensure that the setup numbers match the appropriate setups in the Probe for Windows database file that they are intended to be acquired with.

Another data field is provided (see PositionImportExportFileType in the PROBEWIN.INI file section) to export and import the file setup names to the .POS ASCII position files in addition to the sample name and coordinates. If this option is utilized, care must be taken to ensure that the Probe file setups specified in the position file, actually exist as specified by the directory path.

Digitizing Standard Mounts with More than 48 Standards

When creating digitized standard files for the standard mounts, it should be noted that the program has a limit of MAXSTD% standards (normally equals 48). However, this limitation is easily overcome for standard mounts that contain more than 48 standards. The following instructions will explain how to bypass the 48 standard limit in the STAGE digitize feature:

1. First clear the current position database (POSITION.MDB) by clicking the Standards option and then clicking Delete All from the Digitize! window.

2. Next from the Standard | Add Standards To Run menu (from the main STAGE window) select the first 48 standards to be digitized.

3. Now digitize these 48 standards using the Digitize Sample Positions dialog. Typically only a single random point need be digitized, but more will be utilized by the software if they are digitized explicitly.

4. Next, exit the STAGE program and re-start it. Open the Digitize! window. The first 48 standard sample positions will be listed.

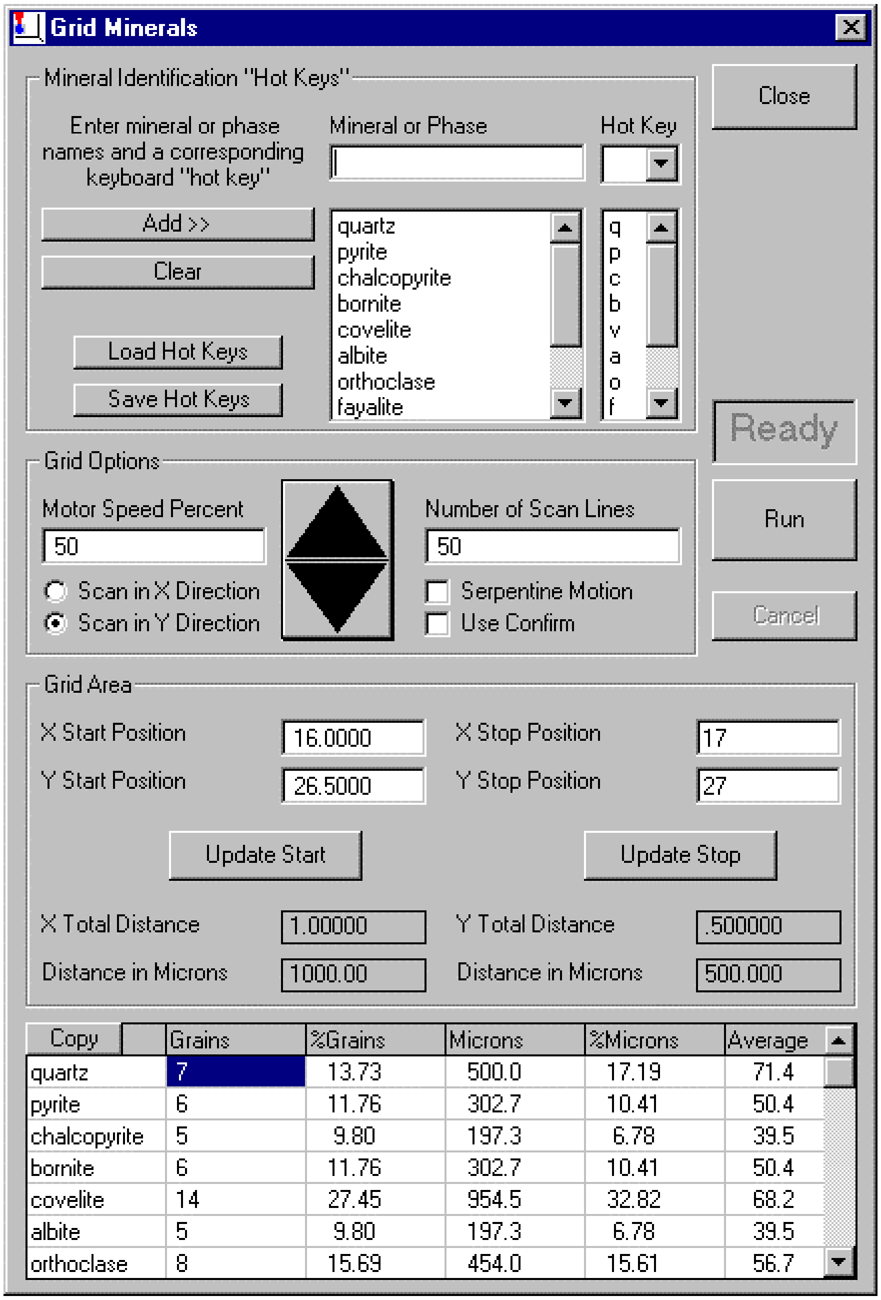
5. Now, go back to the Standard | Add Standards To Run menu and add an additional 48 standards for the next phase of digitizing.

6. Digitize these next 48 standards, again using the Digitize Sample Positions dialog.

7. Repeat steps 4, 5 and 6 if necessary.

Grid Minerals

The Grid Minerals menu item is found under the Window menu and is intended for phase mapping of unknown samples. The feature will cause the stage to move in a raster or serpentine motion over a specified area of the sample to allow mapping of phase boundaries for modal analysis based upon user definitions of various keyboard characters.



Mineral Identification "Hot Keys"

Enter mineral or phase names and a corresponding keyboard "hot key"

Mineral or Phase	Hot Key
quartz	q
pyrite	p
chalcopyrite	c
bornite	b
covelite	v
albite	a
orthoclase	o
fayalite	f

Grid Options

Motor Speed Percent: 50

○ Scan in X Direction

● Scan in Y Direction

Number of Scan Lines: 50

☐ Serpentine Motion

☐ Use Confirm

Grid Area

X Start Position: 16.0000

Y Start Position: 26.5000

X Stop Position: 17

Y Stop Position: 27

X Total Distance: 1.00000

Distance in Microns: 1000.00

Y Total Distance: .500000

Distance in Microns: 500.000

	Grains	%Grains	Microns	%Microns	Average
quartz	7	13.73	500.0	17.19	71.4
pyrite	6	11.76	302.7	10.41	50.4
chalcopyrite	5	9.80	197.3	6.78	39.5
bornite	6	11.76	302.7	10.41	50.4
covelite	14	27.45	954.5	32.82	68.2
albite	5	9.80	197.3	6.78	39.5
orthoclase	8	15.69	454.0	15.61	56.7

The following output is created by the grid minerals procedure and saved automatically to an output file (.OUT) file for subsequent processing of the phase coordinate data :

```
 30.0000 20.0000 29.5867 20.0000 "a" "albite"
 29.5867 20.0000 29.1733 20.0000 "q" "quartz"
 29.1733 20.0000 28.9667 20.0000 "q" "quartz"
 28.9667 20.0000 28.5533 20.0000 "a" "albite"
 28.5533 20.0000 28.2433 20.0000 "a" "albite"
 28.2433 20.0000 28.1400 20.0000 "a" "albite"
 28.1400 20.0000 27.5200 20.0000 "o" "orthoclase"

[some data deleted to save room]

 28.8233 25.0000 28.9266 25.0000 "o" "orthoclase"
 28.9266 25.0000 29.4433 25.0000 "a" "albite"
 29.4433 25.0000 29.5466 25.0000 "o" "orthoclase"
 29.5466 25.0000 29.5466 25.0000 "a" "albite"
 29.5466 25.0000 30.0000 25.0000 "q" "quartz"

Grid Minerals Results:

Mineral        Grains         %Grains        Microns        %Microns       Average
quartz          8                8.42         1733.4           7.67          216.7
pyrite          6                6.32         1033.3           4.57          172.2
chalcopyrite    16              16.84         1446.7           6.40           90.4
bornite         5                5.26         1550.0           6.86          310.0
covelite        10              10.53         5440.0          24.06          544.0
albite          17              17.89         3720.0          16.45          218.8
orthoclase      14              14.74         2893.3          12.80          206.7
fayalite        9                9.47         2273.3          10.05          252.6
hematite        2                2.11          556.7           2.46          278.4
geothite        3                3.16          620.0           2.74          206.7
magnesite       5                5.26         1343.3           5.94          268.7
```

To perform a grid minerals procedure, specify the grid area in stage coordinates and click the Run button. The program will drive to the X and Y start coordinates and wait for keyboard events to define the grain boundaries. Note that the stage will only move while a key is pressed, although any keyboard character will suffice not only the keyboard characters defined in the "hot key" list.

To quit the grid mineral procedure before it has completed, click the Cancel button just below the Run button. Note that keyboard character events will only be recognized by the grid minerals procedure if the Grid Minerals dialog is the active window.

The grid minerals feature supports the following options:

Mineral Identification "Hot Keys"

Add >>- add the mineral or phase and hot key character defined to the list

Clear- clear the mineral/phase hot key list

Load or Save Hot Key File- define and save or load a specific set of "hot keys". That is minerals or phases that are associated with a alphabetic keyboard character. The following is an example hot key file :

```
"quartz"         "q"
"pyrite"         "p"
"chalcopyrite"   "c"
"bornite"        "b"
"covelite"       "v"
"albite"         "a"
"orthoclase"     "o"
"fayalite"       "f"
"hematite"       "h"
"geothite"       "g"
"magnesite"      "m"
```

Note that file is a simple <tab> delimited ASCII file and that both the mineral/phase and the hot key character fields are enclosed in double quotes.

Grid Options

Motor Speed Percent- This text field specifies the initial stage motor speed when the grid minerals procedure is run. It represents the percent of the full motor speed. The default value is 50 %. To modify the value, either before or during the grid minerals procedure, click the spin button up arrow to increase the speed or the down arrow to decrease the speed. The value will increment or decrement by 10% of the current value with each click.

Number of Scan Lines- the number of scan lines the procedure will perform. The density of scan lines should reflect the average grain size in order to avoid aliasing effects.

Scan in X Direction/Scan in Y Direction- these option button specify the direction in which the grain boundaries are scanned during each raster or serpentine stage move.

Serpentine- performs a serpentine scan as opposed to a simple raster scan. Depending on the backlash characteristics of the digitizing stage and the grain size this may or may not be a appropriate choice.

Use Confirm- if this item is checked, the program will allow the user a time interval at the start of each scan line in order to modify the stage focus or other make other adjustments.

Grid Area

X/Y Start/Stop Position- enter the start and stop positions of the area to have the grid minerals procedure performed on. The coordinate must be in stage coordinates, although the total X and Y extents are reported in both stage units and microns. Use the joystick and the Update buttons to define the area to specific sample areas.

Grid Results

Copy- The results of the grid minerals procedure is reported "on the fly" and at the conclusion of the procedure. Use the copy button to copy the grid results at any time to the clipboard. The fields are:

Grains- The number of grains digitized for each mineral or phase. A single grain being defined by a key down and a key up event.

% Grains- the percent abundance of the number of grains for each mineral or phase (does not include undefined grains).

Microns- the total distance (width) of the grains in microns for each mineral or phase (does not include undefined grains).

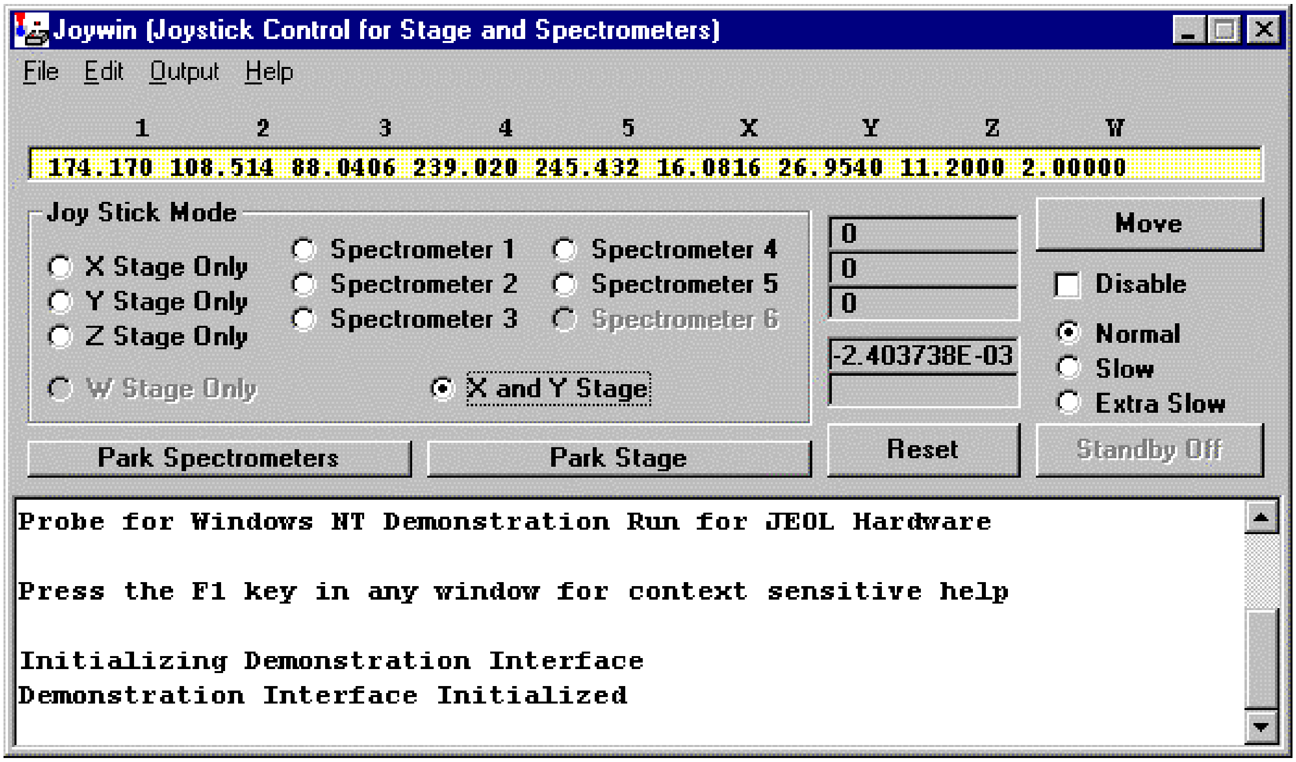
% Microns- the percent microns for each mineral or phase (does not include undefined grains).

Average- the average grain distance (width) for each mineral or phase in microns (does not include undefined grains).

JoyWin

Overview

Program JOYWIN.EXE is a small applet for joystick control of the stage and spectrometers. The default is X and Y stage control, although any single motor (including spectrometers) may be controlled also.



1	2	3	4	5	X	Y	Z	W
174.170	108.514	88.0406	239.020	245.432	16.0816	26.9540	11.2000	2.00000

Joy Stick Mode:
○ X Stage Only
○ Y Stage Only
○ Z Stage Only
○ W Stage Only
○ Spectrometer 1
○ Spectrometer 2
○ Spectrometer 3
○ Spectrometer 4
○ Spectrometer 5
○ Spectrometer 6
● X and Y Stage

0
0
0
-2.403738E-03

☐ Disable
● Normal
○ Slow
○ Extra Slow

```
Probe for Windows NT Demonstration Run for JEOL Hardware

Press the F1 key in any window for context sensitive help

Initializing Demonstration Interface
Demonstration Interface Initialized
```

Description

The JOYWIN application is a small utility application for control of the stage (or individual spectrometers) using a standard joystick. It is intended to be automatically started from the Windows Startup group in order to initialize the microprobe hardware (Advanced MicroBeam hardware only) and run minimized in the background for full time access to the joystick for stage x-y control.

It is critical that JOYWIN be the first Probe for Windows program run, for proper initialization of the motor controller when using the Advanced Microbeam servo motor interface. This restriction also includes the Micro-Image digital imaging software since it requires the stage motors to be properly initialized before it can utilize them. For similar reasons it is also important that JOYWIN be the last program in the Probe for Window program group to be terminated. It is for these reasons that the JOYWIN application should be started automatically from the StartUp group and left running in the background (perhaps minimized) while other Probe for Windows applications are run. If it is necessary to manually re-start JOYWIN, then all other Probe for Windows applications must be terminated before running JOYWIN again.

Most of the motion functionality in JOYWIN is included in the Probe for Windows application and help for these functions can be found in the Probe for Windows documentation and help sections.

Feature Details

- to control the z stage axis (when X and Y Stage option is selected), simply move the joystick in a side to side direction while holding joystick button number 1 down.

- to jog the stage (x, y and z) at the current position, click joystick button number 2.

- to control a specific spectrometer select the corresponding option button for the spectrometer from the main JOYWIN window

- to enable more precise control, check the Slow Speed check box

- to disable the joystick (to prevent spurious motion during long automated acquisitions for example), check the Disable check box

- to re-center the joystick, click the Reset button. This is especially useful if the joystick is causing the stage to "drift" one the program is running.

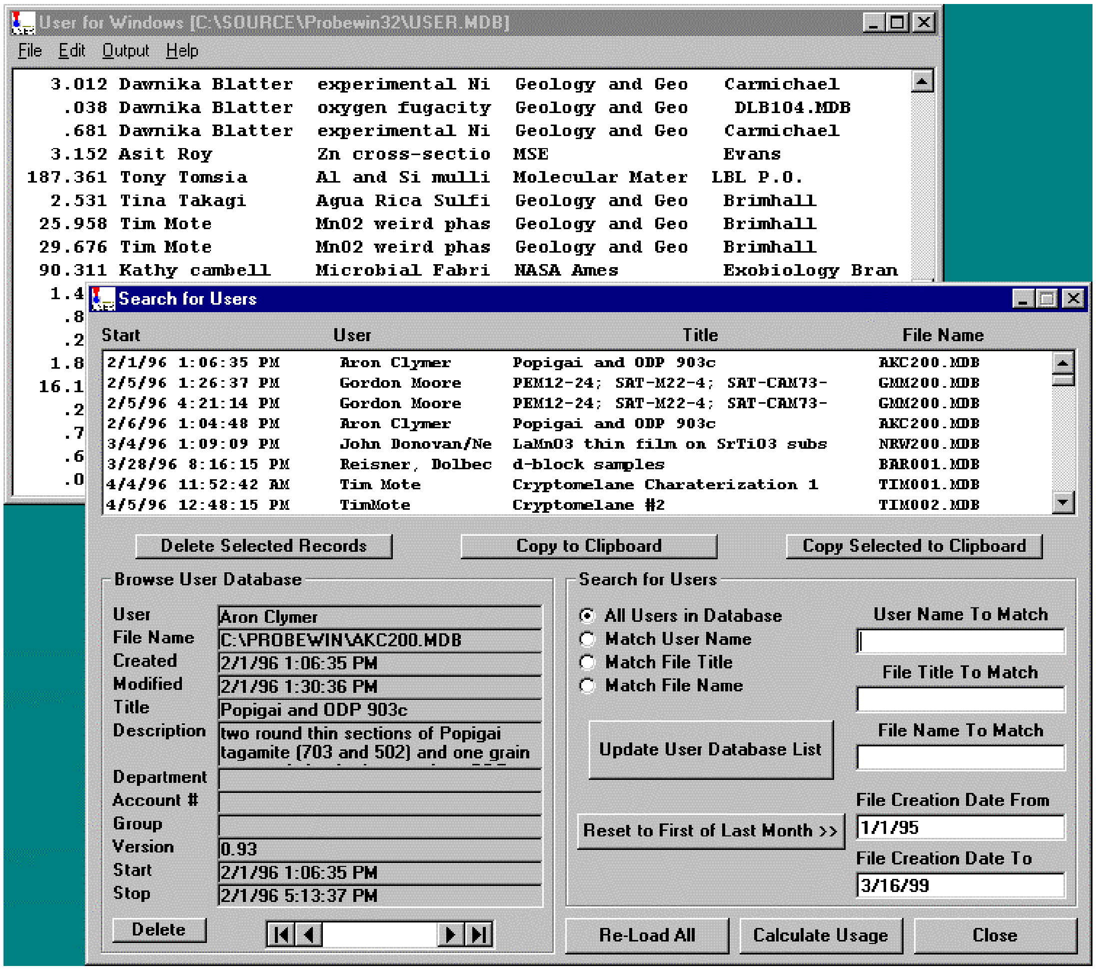
Be sure that all other Probe for Windows applications are closed before starting or re-starting the JOYWIN application. Once the application has started, it is usually most convenient to click the minimize button in the upper right of the JOYWIN main window to keep the application running in the background while using a minimum of screen space.

UserWin

Overview

Program USERWIN.EXE is another small applet for accessing the probe user database that is automatically maintained by Probe for Windows whenever the program is opened in "real-time" mode (that is, interfaced to the microprobe hardware).

It contains extensive search and list capabilities and can calculate the hourly usage of the microprobe in a specified date range for the specified user records.



```
  3.012 Dawnika Blatter   experimental Ni  Geology and Geo   Carmichael
   .038 Dawnika Blatter   oxygen fugacity  Geology and Geo    DLB104.MDB
   .681 Dawnika Blatter   experimental Ni  Geology and Geo   Carmichael
  3.152 Asit Roy          Zn cross-sectio  MSE               Evans
187.361 Tony Tomsia       Al and Si mulli  Molecular Mater  LBL P.O.
  2.531 Tina Takagi       Agua Rica Sulfi  Geology and Geo   Brimhall
 25.958 Tim Mote          MnO2 weird phas  Geology and Geo   Brimhall
 29.676 Tim Mote          MnO2 weird phas  Geology and Geo   Brimhall
 90.311 Kathy cambell     Microbial Fabri  NASA Ames         Exobiology Bran
  1.4
   .8
   .2
  1.8
 16.1
   .2
   .7
   .6
   .0
```

```
Start                 User             Title                              File Name
2/1/96 1:06:35 PM     Aron Clymer      Popigai and ODP 903c               AKC200.MDB
2/5/96 1:26:37 PM     Gordon Moore     PEM12-24; SAT-M22-4; SAT-CAM73-    GMM200.MDB
2/5/96 4:21:14 PM     Gordon Moore     PEM12-24; SAT-M22-4; SAT-CAM73-    GMM200.MDB
2/6/96 1:04:48 PM     Aron Clymer      Popigai and ODP 903c               AKC200.MDB
3/4/96 1:09:09 PM     John Donovan/Ne  LaMnO3 thin film on SrTiO3 subs    NRW200.MDB
3/28/96 8:16:15 PM    Reisner, Dolbec  d-block samples                    BAR001.MDB
4/4/96 11:52:42 AM    Tim Mote         Cryptomelane Charaterization 1     TIM001.MDB
4/5/96 12:48:15 PM    TimMote          Cryptomelane #2                    TIM002.MDB
```

Browse User Database

User Aron Clymer
File Name C:\PROBEWIN\AKC200.MDB
Created 2/1/96 1:06:35 PM
Modified 2/1/96 1:30:36 PM
Title Popigai and ODP 903c
Description two round thin sections of Popigai tagamite (703 and 502) and one grain
Department
Account #
Group
Version 0.93
Start 2/1/96 1:06:35 PM
Stop 2/1/96 5:13:37 PM

Search for Users

● All Users in Database
○ Match User Name
○ Match File Title
○ Match File Name

User Name To Match
File Title To Match
File Name To Match
File Creation Date From 1/1/95
File Creation Date To 3/16/99

Description

The USERWIN application is a small utility for access to the real-time user database. All user activity (when interfaced to the microprobe hardware) is automatically recorded and can be accessed for statistical and recharge purposes.

Hourly usage can be calculated for the displayed records and the selection of records can be based on a number of criteria including name, title of run, filename and date range. The calculated hours can be printed out for laboratory documentation purposes and is also saved automatically to a tab delimited file.

Creating a New User Database

The operator may find that it is necessary to create a new user database. This might be to take advantage of new database fields that may have been added to the program. For example, beginning in version 2.05, three additional user customizable fields were added for improved documentation purposes. If the current user database was created using a version prior to version 2.05, then a new user database will have to be created to access these new fields. To create a new user database and retain the information in the current user database, simply follow the steps below:

1. Backup your current user database. The user database is in the Probe for Windows directory and is named USER.MDB. In fact, backup all files in the Probe for Windows directory to be safe.

2. Run the User for Windows application. The default user database is opened automatically. Move the Search for Users window to one side so that the main window is visible (do not click the Close button).

3. Click File | Export and confirm the USER.DAT export file. All user records will be exported. This takes only a second or two.

4. Click File | Close to close the current user database.

5. Click File | Import and select the import file. Click OK to select the previously created default USER.DAT. Next, select a new user database. Click OK to select the default USER.MDB and confirm the overwrite operation.

6. Modify the File Info fields as desired. The records will be imported in a few seconds and the Search for Users window will display the new user database.

Calculating Hourly Usage

To calculate the hourly usage of all user records, simply click the Calculate Usage button in the Search for Users window. To calculate the hourly usage of the most recent records, click the Reset to First of Last Month button and then click the Update User Database List. Then click the Calculate Usage button again. In a similar fashion, any selected subset of records may be obtained using the various Search For Users options and the hourly usage calculated.

Each time the hourly usage is calculated, output is directed to both the log window and an ASCII tab delimited text file for importing into Excel or other spreadsheet software. The log window output or selection may also be copied to the clipboard or printed.

Using the Data Cursor

In the lower left of the Search for Users window a number of database fields and a data cursor are visible. Using the data cursor allows one to browse through all or selected records. The outside arrows on the data cursor move to the beginning or end of the database and the inside arrows move one record at a time forward or backward.

Modifying the Custom Data Fields

Several user database fields may be defined by the user for special documentation or accounting purposes. They are all text fields, up to 64 characters in length. To change the field labels and/or the default text, see the instructions under the [general] section of the PROBEWIN.INI configuration file documentation above.

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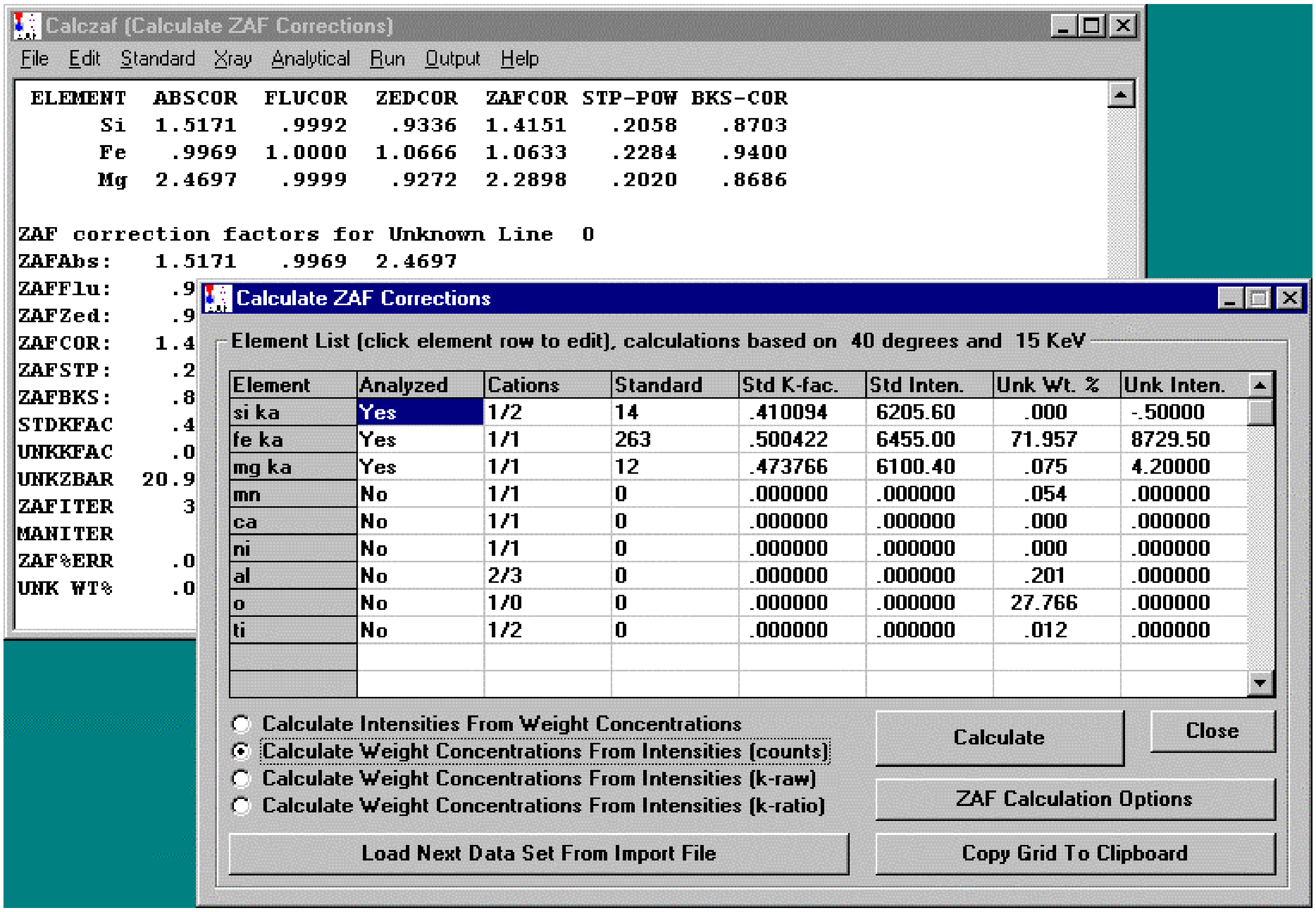
CalcZAF

Overview

Program CALCZAF.EXE is another small applet for "stand-alone" calculation of ZAF and Phi-Rho-Z matrix correction parameters. CalcZAF can accept composition to calculate intensities (k-ratios) or calculate composition from intensities using normalized k-ratios, raw k-ratios or unknown and standard counts. K-ratios or counts measured on compound standards may be utilized, the software will load standard compositions from the Standard composition database.



```
 ELEMENT  ABSCOR  FLUCOR  ZEDCOR  ZAFCOR STP-POW BKS-COR
      Si  1.5171   .9992   .9336  1.4151   .2058   .8703
      Fe   .9969  1.0000  1.0666  1.0633   .2284   .9400
      Mg  2.4697   .9999   .9272  2.2898   .2020   .8686

ZAF correction factors for Unknown Line  0
ZAFAbs:   1.5171   .9969  2.4697
ZAFFlu:     .9
ZAFZed:     .9
ZAFCOR:    1.4
ZAFSTP:     .2
ZAFBKS:     .8
STDKFAC     .4
UNKKFAC     .0
UNKZBAR   20.9
ZAFITER      3
MANITER
ZAF%ERR     .0
UNK WT%     .0
```

Element List (click element row to edit), calculations based on 40 degrees and 15 KeV

Element	Analyzed	Cations	Standard	Std K-fac.	Std Inten.	Unk Wt. %	Unk Inten.
si ka	Yes	1/2	14	.410094	6205.60	.000	-.50000
fe ka	Yes	1/1	263	.500422	6455.00	71.957	8729.50
mg ka	Yes	1/1	12	.473766	6100.40	.075	4.20000
mn	No	1/1	0	.000000	.000000	.054	.000000
ca	No	1/1	0	.000000	.000000	.000	.000000
ni	No	1/1	0	.000000	.000000	.000	.000000
al	No	2/3	0	.000000	.000000	.201	.000000
o	No	1/0	0	.000000	.000000	27.766	.000000
ti	No	1/2	0	.000000	.000000	.012	.000000

○ Calculate Intensities From Weight Concentrations
● Calculate Weight Concentrations From Intensities (counts)
○ Calculate Weight Concentrations From Intensities (k-raw)
○ Calculate Weight Concentrations From Intensities (k-ratio)

CalcZAF can also calculate large amounts of binary composition data and create data suitable for plotting error histograms.

Description

Program CalcZAF is a small utility program for stand-alone calculations using the ZAF and Phi-Rho-Z algorithms contained in the Probe for Windows software

CalcZAF allows the user to perform various types of calculations for concentrations to intensities or intensities to concentrations:

> 0. Concentrations to intensities- this calculation calculates the elemental k-factors for the specified concentrations. This is similar to the normal standard k-factor calculation performed for the primary standards by Probe for Windows. Note that although Probe for Windows only performs an "elemental" standard k-factor calculation, CalcZAF can also perform an "oxide" standard k-factor calculation. Requires elemental concentrations.
>
> 1. Count intensities to concentrations- this calculation calculates elemental concentrations using both unknown and standard intensities and an assigned standard to calculate the standard k-factor for each "analyzed" element. Intensities for the standard and unknown must be in the same units.
>
> The program will automatically calculate the standard k-factors for each element, however the correct standard must first be assigned from the list of standards in the run. See the Standards | Add Standards to Run menu item to add standards to the run that can be assigned to the "analyzed" elements.
>
> 2. Raw k-ratio intensities to concentrations- this calculation is similar to the above, except that the standard intensity is not required to be entered as it is already "included" in the raw unknown k-ratio. Note that a standard must still be assigned to each "analyzed" element.
>
> 3. K-ratio intensities to concentrations- this calculation is based on an already "normalized" elemental k-ratio (to 1.000). Therefore, only the element, x-ray and unknown "intensity" must be entered for the calculation to be performed. No standard assignments are required, however the program will assign a "dummy" standard assignment of 32767 to each analyzed element.

Importing Concentration or Intensity Data From a Disk File

CalcZAF allows the user to create ASCII files for importing concentration or intensity data from a disk file. An example file is supplied which is called CALCZAF.DAT. See the File | Open and File | Close menu for loading input data from file. Click the Calculate button to perform the calculations and click the Load Next Dataset from Input File button to load the next data set.

Each dataset in the input file consists of 3 or more lines. The first line contains the calculation mode (defined above), number of analyzed and specified elements, operating voltage and takeoff angle. The second line contains the oxide or elemental calculation flag (1=oxide,2=elemental), the element by difference, element by stoichiometry to stoichiometric oxygen, the stoichiometric ratio, the element by stoichiometry relative to another element, the element to which the stoichiometry is calculated relative to and the relative ratio. The third (and subsequent lines) are the element data for each element including the element symbol, the x-ray symbol ("ka", "la" or "ma" or "" for a specified concentration), the number of cation atoms, the number of oxygen atoms, the standard number assigned as the primary standard (from the Probe for Windows STANDARD.MDB default database), the concentrations (for intensity calculations), the unknown intensity and the standard intensity (if required). This structure is repeated as often as desired for each dataset. The program will automatically detect if the last dataset has been reached.

The structure is shown below in pseudo-code:

```
' Read calculation mode (0, 1, 2, or 3), number of elements, kilovolts and takeoff
Input #3, CalcMode%, LastChan%, Kilovolts!, Takeoff!

' Read oxide/elemental mode, difference, stoichiometry, relative
Input #3, OxideOrElemental%, DifferenceElement$, StoichiometryElement$, StoichiometryRatio!, RelativeElement$, RelativeToElement$, RelativeRatio!

' Loop on each element
For i% = 1 To LastChan%
    Input #3, Elsyms$(i%), Xrsyms$(i%), NumCat%(i%), NumOxd%(i%), StdAssigns%(i%), ElmPercents!(i%), UnkCounts!(i%), StdCounts!(i%)
next i%
```

Note: all strings (element symbols, etc.) must be in double quotes, elements not analyzed (specified concentrations or calculated) are indicated by a blank (empty double quotes) x-ray line string. If the element is a specified concentration, be sure to give the concentration in elemental weight percent for the "ElmPercents!(I%)" parameter and leave the count intensity fields zero.

An example of a CalcZAF input file demonstrating each calculation mode is shown here:

```
0,2,15,40.                     ' 1st dataset (calculates MgO intensities)
2,"","",0.0,"","",0.0
"mg","ka",1,1,0,60.0,0.0,0.0
"o","ka",1,0,0,40.0,0.0,0.0
1,3,15,40.                     ' 2nd dataset (calculates concentrations from unk and std intensities)
2,"","",0.0,"","",0.0
"fe","ka",1,1,895,0.0,7568.1,10265.7
"si","ka",1,2,14,0.0,1329.4,5268.2
"o","ka",1,0,895,0.0,2519.6,2498.1
2,3,15,40.          ' 3rd dataset (calculates concentrations from "raw" k-ratios)
2,"","",0.0,"","",0.0
"fe","ka",1,1,895,0.0,.96283,0.
"si","ka",1,2,914,0.0,.00003,0.
"o","ka",1,0,895,0.0,1.09972,0.
3,2,20,40.          ' 4th dataset (calculates concentrations from normalized k-ratios)
2,"","",0.0,"","",0.0
"mg","ka",1,1,0,0.,.418853,0.0
"o","ka",1,0,0,0.,.190763,0.0
```

Calculating Error Histograms from Binary Composition Input Files

CalcZAF can also calculate error histograms from appropriate input files containing binary compositions and experimentally measured k-ratios. See the Analytical | Calculate Binary Intensities menu.

The user may select an input file of binary compositions for the calculation of error histograms. The calculations are based on the current ZAF and MAC selections. The output is filtered using the options in the Binary Calculation Options dialog.

Note that all input files for binary calculations have a .DAT extension and must conform to the format specified below. Do not confuse these files with CalcZAF input files or other files with the extension .DAT. Use a text editor to examine the input file if necessary.

Some sample input files of binary compositions are supplied, e.g., AUAGCU2.DAT, NISTBIN.DAT and POUCHOU.DAT. An example of the file format is seen here:

```
79   29   2   5   15.   52.5   .8015   .1983   .7400   .0
79   29   2   5   15.   52.5   .6036   .3964   .5110   .0
79   29   2   5   15.   52.5   .4010   .5992   .3120   .0
79   29   2   5   15.   52.5   .2012   .7985   .1450   .0
```

The input data file format assumes one line for each binary. The first two columns are the atomic numbers of the two binary components to be calculated. The second two columns are the x-ray lines to use (1 = Ka, 2 = La, 3 = Ma, 4 = by difference, 5 = specified, that is, do not calculate the intensity). The next two columns are the operating voltage and take-off angle. The next two columns are the weight fractions of the binary components. The last two columns contains the k-exp values for calculation of k-calc/k-exp. If the second element x-ray line is "specified", then no experimental k-ratio value is required (use zero).

Log Window

The main window of CalcZAF is a scrollable text window which will record all output and calculations. This log window is a fully cut and paste enabled text window which can be used to transfer data or analysis results to any other Windows application such as a word processor or spreadsheet.

Simply click and drag the mouse to select text and use the <ctrl> c, <ctrl> x or <ctrl> v key combinations to copy, cut or paste text to or from the Windows Clipboard. Or use the Edit menu for these and other Clipboard functions.

The size of the log window buffer can be specified in the PROBEWIN.INI file and is limited only by the amount of memory available. If the Save Log to Disk File option is selected from the Output menu, then all log window output is mirrored to a text file, including any text manually entered by the user using the keyboard. The log window font and font size is specified in the PROBEWIN.INI file and may be changed during a run from the Output menu. To view a copy of the log file during a run, select the View Disk Log option from the Output menu.

CalcZAF Window Details

ZAF Calculation Window Details

The ZAF Calculation window provides an interface for viewing and entering x-ray compositions and intensities.

Calculate

This button will start the current intensity or composition calculation. The results will be displayed in both the log window and the grid control. Use the Copy Grid to Clipboard button to copy the grid contents for pasting into another document.

Close

This will close the currently open CalcZAF input data file. This is used in cases where it is desired to open another CalcZAF input data file.

ZAF Calculation Options

Calculate With Stoichiometric Oxygen

CalcZAF allows the user to display the results of an analysis in oxide weight percents based on the cation ratios defined for each element. In addition the user may select to calculate oxygen by stoichiometry if oxygen is not an analyzed element. If oxygen is measured or calculated by stoichiometry, the program will automatically calculate the actual excess or deficit oxygen in the analysis if the Display As Oxide option is selected.

If the element to be calculated by formula basis, difference or stoichiometry is not already in the sample setup, it must be added as a "not analyzed" element using the Elements/Cations button first, by simply clicking any empty row in the element list and typing in an element symbol with a blank x-ray line.

Note that if the sample being analyzed is a standard and oxygen is not measured, then the program will automatically specify any oxygen from the standard database. However, if the sample is an unknown, then the program will add in any specified oxygen as "excess" oxygen into the matrix correction, if the user has specified any element concentrations.

Calculate As Elemental

This option calculates all elements as pure elements without the addition of stoichiometric oxygen in the matrix correction calculation.

Element by Difference

An element by difference can be included in this way also to account for their affect on the x-ray intensities. The element by difference must be one of the specified elements in the sample setup. Note that the this option should be utilized carefully, because the calculation will always result in a 100% total which could mask the results of a bad acquisition.

Stoichiometry to Oxygen

Another useful feature for the analysis of carbonate or borate samples in an oxide run is the use of the "element by stoichiometry to the stoichiometric element (oxygen)" option. With this feature the analyst can analyze just the cations (Fe, Mg, Ca, etc.) in a sample and have the oxygen calculated by stoichiometry and another specified element (usually C for carbonates or B for borates) calculated relative to oxygen. In the case of carbonates, for example $CaCO3$, carbon is always in the ratio 1 to 3 to oxygen.

Therefore by simply specifying C by stoichiometry relative to the stoichiometric element (oxygen) at 0.333 (1 divided by 3) the correct amount of both oxygen and carbon will be incorporated into the ZAF matrix correction and totals without analyzing for either. This calculation should only be used with compounds where the ratio to oxygen is both known and unchanging.

Note that oxygen must be an analyzed or specified element before this calculation can be applied. One more point about element by stoichiometry to oxygen. Consider the example of a trace element analysis of several metals in an alumina ($Al2O3$) matrix. If Al and O are not to be analyzed, yet the user desires to have $Al2O3$ added to the matrix correction, how can this be accomplished?

There are two ways this can be achieved. One way would be to simply specify Al by difference and calculate oxygen by stoichiometry. The program will then correctly add in the proper amount of stoichiometric $Al2O3$ to the matrix correction for each analysis line. The other way is to use the element by stoichiometry to oxygen calculation as discussed below.

Adjust the cation ratios of the metals to elemental stoichiometry (one cation and zero anions). Next, select "element by stoichiometry to oxygen" and (in this example) select "al" as the element by stoichiometry. To achieve a 2 to 3 ratio, next enter "0.666" Al atoms per O atom. Run the calculation and note that $Al2O3$ was not added to the matrix calculation! What happened? In this example, the user had selected a cation ratio for the analyzed elements of all elemental atoms, and since there was zero oxygen to begin the iteration, the program never got to add the Al which then never added the stoichiometric oxygen! How can this be avoided? Simply specify some small concentration of an oxide element (for instance $SiO2$) in the specified element concentration, say 0.05 %. This will give the iteration a chance to get started, and allow it to converge on a very close approximation of the $Al2O3$ by difference!

Stoichiometry to Another Element

Yet another option for recalculation is the element relative to another element (by stoichiometry). This calculation is similar to the "element by stoichiometry to oxygen" as described below, however the user may select any other analyzed or specified element as the stoichiometric basis element.

Load Next Dataset From File

This button will load the next sample from the current CalcZAF input datafile.

Copy Grid To Clipboard

This button will copy the contents of the gird to the Windows clipboard. The contents can then be pasted into another windows document or spreadsheet.

CalcZAF Menu Details

File

The File menu can be used to open an existing data file, close the current run, print the current log window contents or log window selection or exit the program.

Open

The Open menu allows the user to select an existing CalcZAF datafile to process previously entered data. All CalcZAF input data files have an extension .DAT and must conform to the specific format described below. Do not confuse .DAT files for other applications with CalcZAF data files. If necessary inspect the input file with a text editor to determine if it actually conforms to the CalcZAF input format.

Some sample CalcZAF input files are supplied, e.g., CALCZAF.DAT and CALCBIN.DAT.

Close

The Close menu allows the user to close the currently open CalcZAF database file. This can be used to close the current file before opening a different CalcZAF database file without re-starting the program.

Print Log

This menu allows the user to print the entire contents of the log window or selected portions of the log window. Be sure to click the Selection field if only a text selection is to be printed, otherwise the program will print the entire log window text.

Print Setup

This menu allows the user to change the default printer or printer setup options for the currently selected printer.

Exit

This menu exits the program.

Edit

Cut

This menu cuts the selected text from the log window to the Windows Clipboard.

Copy

This menu copies the selected text from the log window to the Windows Clipboard.

Paste

This menu pastes text from the Windows Clipboard to the log window.

Select All

This menu selects all text in the log window.

Clear All

This menu clears all text from the log window.

Standard

Standard Database

This menu invokes a shell process to launch the Standard for Windows database application (STANDARD.EXE) as an asynchronous process. The standard database is used to maintain the database of standard compositions and also includes a number of features for exploring analytical conditions based on various standard compositions.

Since the shell process runs independently, it can be accessed during an automation or analysis procedure.

Add Standards To Run

This menu allows the user to specify additional standards to be used in the currently open CalcZAF run.

Note that if the run is closed before any data is acquired for these newly added standards, then they will have to be added again, if the run is re-opened later to acquire data for them. This is because CalcZAF updates the list of standards in the run each time a database file is opened based on the standard samples that have been actually created.

All standards that need to be digitized, acquired or assigned, must be first added to the run using this window dialog.

X-Ray

X-Ray Database

This menu allows the user the view specified ranges of the NIST x-ray wavelength database. This database covers the wavelength range from approximately 0.5 to 100.0 angstroms and includes higher order reflections as well. Higher order reflections are reduced in intensity by 25% for each subsequent order to simulate the effect of PHA analysis.

The x-ray list range (Start Angstroms and Stop Angstroms) and minimum search intensity (Minimum Intensity) of the x-ray list can be specified by the user. Note that since the display is limited to about 1000 items, the program will automatically increase the minimum intensity until the returned list of x-ray lines is less than 1000. To view x-ray lines of lesser intensity, simply reduce the x-ray range and click the Re-Load button.

Description of the X-ray Database (adapted from NIST documentation by C. Fiori)

The NIST x-ray database is based on 4985 (1st order) entries and includes all the measurable x-ray lines, satellites and absorption edges from under 100 eV to over 120 keV. Additionally, most of the x-ray lines and satellites are assigned a relative intensity (relative to the alpha-1 line in each family). The data base was assembled primarily from four sources:

1.) B.L. Doyle, W.F. Chambers, T.M. Christensen, J.M. Hall and G.H. Pepper "SINE THETA SETTINGS FOR X-RAY SPECTROMETERS", Atomic Data and Nuclear Data Tables Vol. 24, No 5, 1979.

2.) E.W. White, G.V. Gibbs, G.G. Johnson Jr. and G.R. Zechman "X-RAY WAVELENGTHS AND CRYSTAL INTERCHANGE SETTINGS FOR WAVELENGTH GEARED CURVED CRYSTAL SPECTROMETERS" Report of the Pennsylvania State Univ., 1964.

3.) J.A. Bearden "X-RAY WAVELENGTHS AND X-RAY ATOMIC ENERGY LEVELS" Rev. Mod. Phys., Vol. 39, No. 78, 1967.

4.) J.A Bearden and A.F. Burr,"REEVALUATION OF X-RAY ATOMIC ENERGY LEVELS", Rev. Mod. Phys., Vol. 31, No. 1, 1967.

Each x-ray line or edge series as a function of atomic number was fit to a fourth degree polynomial. The fit was subtracted from the appropriate data and the residuals plotted and examined. In this way rogue entries could be identified and corrected. The resulting data base is considered to be sufficiently accurate for any application involving the Si (Li) x-ray detector and single crystal wavelength spectrometers.

Note that the last entry in the x-ray database window gives a code for the source of the entry. If the column is blank the source is reference 2. If the column contains the letter "C" the source is reference 1. If the letters "BB" appear, the source is reference 4. The letters "W,F" mean that reference 2 was used but the relative transition probability has been adjusted by Fiori. Reference 3 was used as a check since it is the source of many of the entries of reference 1.

In column 3 the notation KA1,2 means the entry is the weighted sum of the KA1 and KA2 in the ratio 2 to 1. For low atomic number the entries are not self consistent since the data is from different sources. If the column begins with the capital letter S then the entry is a satellite line due to doubly ionized atoms. The relative transition values for these entries are only valid for electron excited specimens, and are, at best, estimates.

Th following are Siegbahn to shell-transition notation conversions:

```
KA    = KA1+KA2+KA3
KA1,2 = (2*KA1+KA2)/3
KA1  = K-L3
KA2  = K-L2
KA3  = K-L1
KB   = SUM(KBn)
KBX  = Metal
KB1  = K-M3
KB1' = KB1+KB3+KB5
KB2  = (K-N3)+(K-N2)
KB2' = K-N3
KB2'' = K-N2
KB3  = K-M2
KB4  = K-N4)+(K-N5)
KB5  = K-M4)+(K-M5)
KB5' = K-M5
KB5'' = K-M4
```

```
Kd1  = K-O3
Kd2  = K-O2
LA   = LA1+LA2
LA1  = L3-M5
LA2  = L3-M4
LB1  = L2-M4
LB10 = L1-M4
LB15 = L3-N4
LB17 = L2-M3
LB2  = L3-N5
LB3  = L1-M3
LB4  = L1-M2
LB5  = L3-O4)+(L3-O5)
LB6  = L3-N1
LB7  = L3-O1
LB9  = L1-M5
LG1  = L2-N4
LG11 = L1-N5
LG2  = L1-N2
LG3  = L1-N3
LG4  = L1-O3
LG4' = L1-O2
LG6  = L2-O4
LG8  = L2-O1
Ll   = L3-M1
Ln   = L2-M1
Ls   = L3-M3
Lt   = L3-M2
Lu   = L3-N6)+(L3-N7)
Lv   = L2-N6
MA1  = M5-N7
MA2  = M5-N6
MB   = M4-N6
MG   = M3-N5
MG2  = M3-N4
MZ1  = M5-N3
MZ2  = M4-N2
Md   = M2-N4
Me   = M3-O5
```

Emission Table

This menu lists a table of emission energies (or angstroms) for all elements and their major analytical x-ray lines.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries use the CalcZAF program.

Edge Table

This menu lists a table of absorption edge energies (or angstroms) for all elements and their major analytical x-ray lines.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries use the CalcZAF program.

Fluorescent Yield Table

This menu lists a table of fluorescent yield fractions for all elements and their major analytical x-ray lines.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries use the CalcZAF program.

MAC Table

This menu lists a table of MACs (mass absorption coefficients) for the specified element and it's major analytical x-ray lines.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries use the CalcZAF program.

The program will automatically load the values based on the selected default MAC table. See the Analytical | ZAF Selection section for a description of the MAC table choices.

Edit X-Ray Table

This menu items opens a small dialog for the purposes of editing single values in the default x-ray emission table. All values are entered in electron volt units (eV). Be aware that changes to this table will affect the calculation of x-ray intensities and compositions in the software.

Edit X-Edge Table

This menu items opens a small dialog for the purposes of editing single values in the default x-ray edge table. All values are entered in electron volt units (eV). Be aware that changes to this table will affect the calculation of x-ray intensities and compositions in the software.

Edit X-Flur Table

This menu items opens a small dialog for the purposes of editing single values in the default x-ray fluorescence yield table. Be aware that changes to this table will affect the calculation of x-ray intensities and compositions in the software.

Edit MAC Table

This menu items opens a small dialog for the purposes of editing single values in the default mass absorption coefficient table. Be aware that changes to this table will affect the calculation of x-ray intensities and compositions in the software.

Convert ELEMINFO.DAT

This menu will initiate a conversion routine to convert the original CITZAF element emission and edge energy file to the binary format used by CalcZAF.

Convert MACMAT*.DAT

This menu will initiate a conversion routine to convert the original CITZAF mass absorption coefficient file to the binary format used by CalcZAF.

Create New Xray Database

This menu will initiate a conversion routine to convert the binary x-ray datafile XRAY.ALL (created by program MakeXray.exe) to the relational database format used by CalcZAF.

Create New McMaster MAC Table

This menu item will use the McMaster mass absorption coefficient fit polynomials to calculate a new mass absorption coefficient table. This table may be selected from the ZAF Selections menu by clicking the MACs button.

Create New MAC30 MAC Table

This menu item will use the MAC30 (Heinrich fit to the Goldstein data) mass absorption coefficient fit polynomials to calculate a new mass absorption coefficient table. This table may be selected from the ZAF Selections menu by clicking the MACs button.

Create New MACJTA MAC Table

This menu item will use the Armstrong (simple Heinrich fit) mass absorption coefficient fit polynomials to calculate a new mass absorption coefficient table. This calculation is from Armstrong's MACCALC program. This table may be selected from the ZAF Selections menu by clicking the MACs button.

Analytical

Display Elements

This menu item will make the ZAF Calculations window visible if it has been minimized or closed.

Calculate Elemental-To-Oxide Factors

This menu will calculate elemental to oxide conversion factors based on the current cation and oxide ratios (as defined by defaults in the ELEMENTS.DAT file).

Calculate Oxide-to-Elemental Factors

This menu will calculate oxide to elemental conversion factors based on the current cation and oxide ratios (as defined by defaults in the ELEMENTS.DAT file).

Empirical MACs

For complete details on this menu, see the help section under the heading: Probe Menu Details | Analytical | Empirical MACs.

ZAF Selections

For complete details on this menu, see the help section under the heading: Probe Menu Details | Analytical | ZAF Selections.

Operating Conditions

This menu allows the user to change the default Takeoff angle and KeV for modeling different microanalytical conditions for the current composition or intensity data.

Binary Calculation Options

This menu allows the user to specify minimum and maximum absorption, fluorescence and atomic number corrections for the purposes of filtering the output to the data file.

Binary Histogram Options

This menu allows the user to specify histogram limits (high and low) and the number of histogram bins for plotting error histograms from binary intensity compositional calculations.

Calculate Binary Intensities (Output Calculated Intensity)

This menu items will allow the user to select an input file of binary compositions for the calculation of error histograms. The calculations are based on the current ZAF and MAC selections. The output is filtered using the options in the Binary Calculation Options dialog.

Note that all input files for binary calculations have a .DAT extension and must conform to the format specified below. Do not confuse these files with CalcZAF input files or other files with the extension .DAT.

Some sample input files of binary compositions are supplied, e.g.,

AUAGCU2.DAT	Au-Ag-Cu alloy k-ratios from NIST (measured at 52.5 takeoff)
NISTBIN.DAT	NIST k-ratio database (compiled by Heinrich)
NISTBINA20.DAT	NIST k-ratio database (compiled by Heinrich), Absorption correction > than 20%
NISTBINZ10.DAT	NIST k-ratio database (compiled by Heinrich), Atomic Number correction > than 10%
POUCHOU.DAT.	Pouchou k-ratio database
POUCHOUA20.DAT.	Pouchou k-ratio database (compiled by Heinrich), Absorption correction > than 20%
POUCHOUZ10.DAT.	Pouchou k-ratio database (compiled by Heinrich), Atomic Number correction > than 10%

An example of the file format is seen here:

```
79   29   2   5   15.   52.5   .8015   .1983   .7400   .0
79   29   2   5   15.   52.5   .6036   .3964   .5110   .0
79   29   2   5   15.   52.5   .4010   .5992   .3120   .0
79   29   2   5   15.   52.5   .2012   .7985   .1450   .0
```

The data file format assumes one line for each binary. The first two columns are the atomic numbers of the two binary components to be calculated. The second two columns are the x-ray lines to use (1 = Ka, 2 = La, 3 = Ma, 4 = by difference, 5 = specified, that is, do not calculate the intensity). The next two columns are the operating voltage and take-off angle. The next two columns are the weight fractions of the binary components. The last two columns contains the k-exp values for calculation of k-calc/k-exp. If the second element x-ray line is "specified" then no experimental k-ratio value is required.

Calculate Binary Intensities (Output Atomic First Approximation)

This menu is only visible if the ExtendedMenu flag is set in the PROBEWIN.INI file. It is used to calculate atomic fraction first approximations, but only those binaries that meet the minimum or maximum correction factor output filter criteria are actually written to the file.

Calculate Binary Intensities (Output Mass First Approximation)

This menu is only visible if the ExtendedMenu flag is set in the PROBEWIN.INI file. It is used to calculate mass fraction first approximations, but only those binaries that meet the minimum or maximum correction factor output filter criteria are actually written to the file.

Calculate Binary Intensities (Output Electron First Approximation)

This menu is only visible if the ExtendedMenu flag is set in the PROBEWIN.INI file. It is used to calculate electron fraction first approximations, but only those binaries that meet the minimum or maximum correction factor output filter criteria are actually written to the file.

Calculate First Approximations Only (Atomic Fraction)

This menu is only visible if the ExtendedMenu flag is set in the PROBEWIN.INI file. It is used to calculate atomic fraction first approximations, but without any correction factor output filter options.

Calculate First Approximations Only (Mass Fraction)

This menu is only visible if the ExtendedMenu flag is set in the PROBEWIN.INI file. It is used to calculate mass fraction first approximations, but without any correction factor output filter options.

Calculate First Approximations Only (Electron Fraction)

This menu is only visible if the ExtendedMenu flag is set in the PROBEWIN.INI file. It is used to calculate electron fraction first approximations, but without any correction factor output filter options.

Run

This menu will output a variety of run information in summary format to the log window. This information can then be cut and pasted to another application or simply selected and printed using the File | Print Log menu.

List Standard Compositions

This menu item will list the compositions of all standards in the current run based on the current values in the standard composition database.

List Current MACs

This menu item will list the currently loaded MACs (mass absorption coefficients) from the last quantitative analysis. Note that any empirical MACs listed from this menu will be used to overwrite table based MACs automatically during calculations.

List Analysis Parameters

This menu item will list the current analysis conditions.

Model Detection Limits

This menu item will allow the user the estimate the 99% confidence (3 sigma) values for the minimum concentration detected based on a specified the unknown count time or the count time required to detect a specified concentration. This will allow the user to design the required analytical setup for trace element determinations based on a given set of intensities from the standard on-peak and unknown background measurements.

The expression for minimum detection concentration is:

$$X_U = \frac{3\sqrt{I_U^B \cdot t_U^B \cdot B_U} \cdot C_S}{t_U^B \cdot B_U \cdot I_S^P}$$

The expression for counting time required for the detection of the given concentration is:

$$t_U^B = \frac{9 \cdot I_U^B \cdot (C_S)^2}{(X_U)^2 \cdot (I_S^P)^2 \cdot B_U}$$

where: I_U^B is the intensity of the background measurement in cps/nA (divide the unknown counts by beam current)

t_U^B is the count time used for the unknown background or peak measurement (must be equal)

B_U is the beam current used for the unknown measurement in nA

C_S is the concentration of the element in the standard in weight percent (wt. %)

I_S^P is the intensity of the standard in cps/nA (divide the standard intensity by standard beam current)

X_U is the unknown concentration in weight percent (wt. %)

Note that all calculations are performed at 3 sigma (standard deviations) and assume that the background and peak measurement count times on the unknown are equal. There is no correction for unknown ZAF correction, but it could easily be applied by simply multiplying the ZAF factor times the resulting concentration or count time.

Remember that weight percent can be converted to ppm (parts per million) using the following expression:

$$ppm = wt\% \cdot 10{,}000$$

Therefore, 0.010 wt% is equal to 100 ppm or 0.001 wt. % is equal to 10 ppm.

Note the calculation assumes that the counting time used for the unknown peak and background measurements are equal. This is usually the case for trace elements since at zero concentration both the peak and off-peak measurements have equal statistical weight. Note also that the unknown background time is the sum of both the high off-peak and low off-peak count times so that:

$$t_U^P = t_U^H + t_U^L = t_U^B$$

where: t_U^P is the count time for the on-peak measurement

t_U^H is the count time for the high off-peak measurement

t_U^L is the count time for the low off-peak measurement

t_U^B is the count time for the combined high and low off-peak measurements (used in the above calculations)

Output

Log Window Font

This menu allows the user to modify the current log window font type, size and appearance using the font Common dialog. It is strongly recommended to use a non-proportional font, such a New Courier, so that numeric column data is properly aligned in the log window.

Note that the default font and size of the log window may be permanently defined in the PROBEWIN.INI file.

Debug Mode

This menu toggles the program in and out of Debug mode. In Debug mode, the program generates an abundance of additional output to the log window for the purposes of debugging the program or viewing the analytical calculations in greater detail.

Debug mode is also useful to check the low level commands sent to the hardware interface. Note that in Debug mode the Form level events are not protected against unanticipated errors. Program CalcZAF normally runs in DebugMode for more analytical detail.

Extended Format

This menu toggles the output option for printout to the log window of element data. If unchecked, the program automatically wraps element data output to 8 elements per line. Note that wavescan data is wrapped 4 elements per line. If this option is checked, the program does not wrap element data to the log window or disk file (if Save To Disk option is enabled), instead the data is written out as far as necessary to the right.

Save To Disk Log

This menu toggles the output option to mirror all log window output to a user specified text file. This may be useful for importing text or data from the log window to another non-Windows program. The default extension is .OUT but another extension may be entered by the user.

If a large amount of output is to be generated, this option will capture all output to the log window so that it can be printed out afterwards. If the output file selected by the user already exists, the program offers the option to overwrite or append subsequent output to the already existing file.

View Disk Log

This menu invokes the file viewer specified in the PROBEWIN.INI file (default file viewer is NotePad) to view the previously or currently opened disk log file.

If the file is currently open, the program will first close the disk log file before viewing it. Note that because the default file viewer (NotePad) is somewhat limited, it may be necessary to specify a more robust file viewer such as TextPad or Word for Windows. See the section on editing the PROBEWIN.INI file above to change the default file viewer.

Open Link To Excel

This menu will create an OLE (Object Linking and Embedding) link to an Excel application (if it is available). This link is available to both the StartWin and ProbeWin applications for sending both intensity and quantitative data to an Excel spreadsheet. This feature requires Excel 8 (Office 97) or higher.

From CalcZAF, the user can send calculated data automatically to an Excel spreadsheet along with the column labels, by clicking the "Excel >>" button.

Close Link To Excel

This menu will close an open OLE (Object Linking and Embedding) link to an Excel application (if it is available).

Help

About CalcZAF

This menu displays the copyright notice, acknowledgments and contact information for users of CalcZAF 95/98/NT.

Help On CalcZAF

This menu opens this help file for hypertext help file viewing at the main table of contents. Note that both a searchable index and glossary are available for browsing by the user at anytime during a run. Each help topic may be printed.

In addition, context sensitive help (that is, help that is applicable to the current program context) is available for each window in all applications by simply pressing the F1 key.

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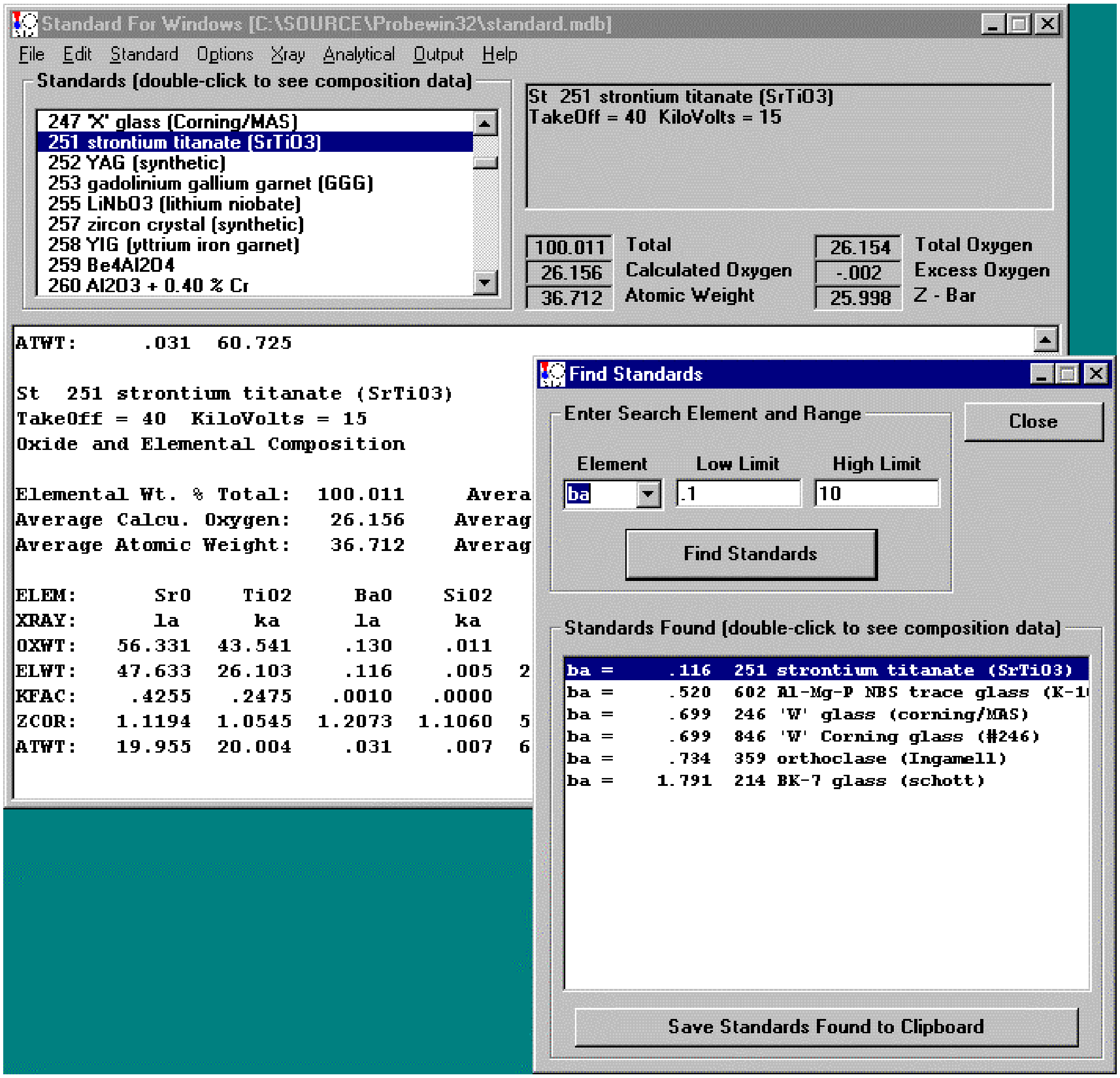
Standard

Overview



247 'X' glass (Corning/MAS)
251 strontium titanate (SrTiO3)
252 YAG (synthetic)
253 gadolinium gallium garnet (GGG)
255 LiNbO3 (lithium niobate)
257 zircon crystal (synthetic)
258 YIG (yttrium iron garnet)
259 Be4Al2O4
260 Al2O3 + 0.40 % Cr

St 251 strontium titanate (SrTiO3)
TakeOff = 40 KiloVolts = 15

100.011	Total	26.154	Total Oxygen
26.156	Calculated Oxygen	-.002	Excess Oxygen
36.712	Atomic Weight	25.998	Z - Bar

```
ATWT:      .031   60.725

St   251 strontium titanate (SrTiO3)
TakeOff = 40   KiloVolts = 15
Oxide and Elemental Composition

Elemental Wt. % Total:  100.011     Avera
Average Calcu. Oxygen:   26.156    Averag
Average Atomic Weight:   36.712    Averag

ELEM:      SrO     TiO2     BaO    SiO2
XRAY:       la       ka      la      ka
OXWT:   56.331   43.541    .130    .011
ELWT:   47.633   26.103    .116    .005   2
KFAC:    .4255    .2475   .0010   .0000
ZCOR:   1.1194   1.0545  1.2073  1.1060   5
ATWT:   19.955   20.004    .031    .007   6
```

Element: ba
Low Limit: .1
High Limit: 10

```
ba =      .116   251 strontium titanate (SrTiO3)
ba =      .520   602 Al-Mg-P NBS trace glass (K-1
ba =      .699   246 'W' glass (corning/MAS)
ba =      .699   846 'W' Corning glass (#246)
ba =      .734   359 orthoclase (Ingamell)
ba =     1.791   214 BK-7 glass (schott)
```

Program STANDARD.EXE maintains the standard composition database used by Probe for Windows for quantitative analysis. A menu item is provided in the Probe for Windows application to run Standard asynchronously.

The standard composition database is in Microsoft Access v. 3.5 format and can also be accessed using Microsoft Office Professional (although no modification of the tables should normally be attempted).

To import standard compositional data from older versions of Probe for Windows, first export the standard database to an ASCII file and then import the data using the Import menu item.

Standard for Windows stores all standard compositions as elemental concentrations. However, the user may elect to enter and display the composition in either oxide or elemental concentrations. In addition, the user may enter standard compositions based on a simple formula if creating a new standard composition.

Oxide standards are standards whose compositions are defined by cation concentrations and their corresponding cation and oxygen valence subscripts. Elemental standards are defined by elemental concentrations only. Whether a standard is entered and/or displayed as an oxide or elemental standard is determined by the value of the Enter Composition In and/or Display Composition As options in the File | Modify dialog. However, both oxide and elemental standards may be used to analyze either oxide or elemental calculated unknown samples in Probe for Windows in any combination.

Log Window

The main window of Standard for Windows is a scrollable text window which will record all output and calculations. This log window is a fully cut and paste enabled text window which can be used to transfer data or analysis results to any other Windows application such as a word processor or spreadsheet.

Simply click and drag the mouse to select text and use the <ctrl> c, <ctrl> x or <ctrl> v key combinations to copy, cut or paste text to or from the Windows Clipboard. Or use the Edit menu for these and other Clipboard functions.

The size of the log window buffer can be specified in the PROBEWIN.INI file and is limited only by the amount of memory available. If the Save Log to Disk File option is selected from the Output menu, then all log window output is mirrored to a text file, including any text manually entered by the user using the keyboard. The log window font and font size is specified in the PROBEWIN.INI file and may be changed during a run from the Output menu. To view a copy of the log file during a run, select the View Disk Log option from the Output menu.

All standards in the standard database are seen in the scrollable multi-select list box. Multi-select means that the list box supports the selection of more than one item. To select a range of items click the first item in the list and then holding the <shift> key, select the last item to be selected. To select individual items, click while holding the <ctrl> key down.

To see the compositional data for a particular standard, simply double click the standard in the list box. The standard composition and k-factors are output to the log window. Note that the k-factors calculated in Standard for Windows are based on the default operating voltage, take-off angle, x-ray lines, mass absorption coefficients and ZAF options for reference purposes only. Probe for Windows calculates actual standard k-factors based on the actual analytical conditions of the specific sample. An example of this output is given here :

```
St 160 Set 0 NBS K-412 mineral glass
TakeOff =  40 KiloVolts =  15
Oxide and Elemental Composition

Elemental Wt. % Total :   99.260     Average Total Oxygen :   42.780
Average Calcu. Oxygen :   42.782    Average Excess Oxygen :    -.002
Average Atomic Weight :   22.048    Average Atomic Number :   12.733

ELEM:     SiO2     FeO     MgO     CaO   Al2O3     MnO       O
```

```
XRAY:      ka      ka      ka      ka      ka      ka      ka
OXWT:   45.352   9.960  19.331  15.250   9.270    .099   -.002
ELWT:   21.199   7.742  11.657  10.899   4.906    .077  42.780
KFAC:    .1621   .0654   .0777   .1008   .0334   .0006   .1696
ZCOR:   1.3080  1.1836  1.5000  1.0817  1.4672  1.2043  2.5223
BETA:    .1632   .0631   .0732   .1023   .0312   .0004   .1634
ATWT:   16.766   3.079  10.653   6.040   4.039    .031  59.391
```

The keywords are defined here :

ELEM:	Oxide formula or elemental symbol
XRAY:	Defined x-ray lines for k-factor calculation
OXWT:	Oxide formula weight percent (using defined oxides)
ELWT:	Elemental weight percent
KFAC:	Normalized elemental k-factor (using default Phi-Rho-Z)
ZCOR:	ZAF correction factor (using default Phi-Rho-Z)
BETA:	Beta correction factor (using default alpha-factors)
ATWT:	Normalized atomic weight percent of element

Running Standard for the First Time

When Probe (or Standard) for Windows is run for the first time a demonstration standard database is automatically configured, for use with the demonstration probe data files JEOLEL.MDB and JEOLOX.MDB.

To create a site specific standard database, this demonstration standard database can be overwritten by clicking the File | New menus in the STANDARD program main window to create an empty standard database (click File | Close first, if necessary to close an already open database). Note that the demonstration probe data files JEOLEL.MDB and JEOLOX.MDB will be unable to run properly once this step is performed and should therefore be deleted afterwards.

After the new standard database has been created, the user can either begin to type in their new standard compositions by hand or import them from ASCII import files. The format of the standard import ASCII file can be seen in the supplied example files DHZ.DAT, ORE.DAT or SRM.DAT.

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Standard Menu Details

File

The File menu can be used to create a new Probe for Windows standard composition database, open an existing database, close the current database, import or export standard compositions from or to ASCII files, display database file information, print the log window or exit the program.

New

The New menu opens a dialog to select a new Probe for Windows standard database file. This option should only be used when creating a site specific standard composition database for the first time in order to overwrite the supplied demonstration database. The default standard database name must be STANDARD.MDB and this filename should be used in order that Probe for Windows can access this file for quantitative calculations. Once a site specific standard database has been created it will be automatically loaded each time the Standard for Windows program is run.

If the file already exists, the program will prompt the user whether to overwrite the existing file. After the new file has been created the program will display a file information dialog to allow the user the personalize the standard database title and description for the specific site.

The program will automatically append a .MDB extension to the file name if one is not supplied by the user. The .MDB file name indicates Microsoft Database and is a Microsoft Access v. 3.5 database file.

The user can also use this menu to create a new searchable standard database before importing the DHZ.DAT, SRM.DAT or ORE.DAT ASCII files. It is suggested to name the files DHZ.MDB, SRM.MDB and ORE.MDB respectively.

Open

The Open menu allows the user to select an existing Probe for Windows standard database file to add or modify standard composition data. The program will check that the specified file is of the correct format and warn the user if not. Probe for Windows generally uses a file extension of .MDB for it's standard database file.

The default standard database is STANDARD.MDB and this file is automatically accessed by Probe for Windows for quantitative calculations. Although the user may create as many additional standard composition databases as desired, Probe for Windows uses only the STANDARD.MDB file in the C:\Program Files\Probe for Windows directory for analysis of unknown samples.

Save As

The Save As menu allows the user to save the currently open Probe for Windows standard database file to another file name. The program will automatically append a .MDB extension if necessary.

Close

The Close menu allows the user to close the currently open Probe for Windows standard database file. This menu can be used in order to open another Probe for Windows standard database file. The program will ask the user to confirm the close operation and all compositions are automatically saved.

Import

The Import menu allows the user to import an ASCII file of standard compositions. This ASCII import file is usually named STANDARD.DAT and is primarily intended for users who wish to import ASCII standard composition data files into Probe for Windows. Please contact Advanced MicroBeam if your standard composition database is in another format and would like it imported into a Probe for Windows standard database.

Several example import files are supplied :

DHZ.DAT silicate analyses	(Deer, Howie and Zussman)
ORE.DAT sulfide analyses	(Dana's Mineralogy)
SRM.DAT SRM analyses	(NIST standard reference alloys and glasses)

The DHZ.DAT is a database of all the analyses in the student 1st edition of "Rock Forming Minerals" by Deer, Howie and Zussman (1st Edition). The DHZ database also includes the Harvard oxygen mineral standards (entered as standards #2761-2773) which are available from Carl Francis at the Harvard Mineralogical Museum. The ORE.DAT file is sulfide minerals from Dana's Mineralogy entered in ideal formulas. The SRM.DAT is a database of SRM (standard reference materials) alloys and glasses from the NIST SRM catalog.

The import process can be repeated as often as desired to import additional standards into the Probe for Windows standard database. Note that the program will abort the import process if a standard number already exists in the database.

Since almost any source can generate a suitable standard composition import file, a sample of a standard composition import file is shown here with a short explanation for each field:

```
 2003                        "Hortonolite, p. 4"
""
 -1  9
     "si"     "ti"     "al"     "fe"     "mn"     "mg"     "ca"     "h "     "o "
     1        1        2        1        1        1        1        2        1
     2        2        3        1        1        1        1        1        0
   15.912     .258     .482   32.402     .527   12.254     .579     .010   36.688
 2004              "Fayalite, Pantelleria, p. 4"
""
 -1  8
     "si"     "ti"     "al"     "fe"     "mn"     "mg"     "ca"     "o "
     1        1        2        1        1        1        1        1
     2        2        3        1        1        1        1        0
   14.285     .432     .048   47.339    2.656    2.093     .808   32.650
```

Two standard compositions are shown in the example above. The fields may be comma, tab or space delimited.

The first line contains the standard number (must be unique and range from 1 - 32768), and the standard name in double quotes (generally site specific standard numbers should use values under 2000 to avoid conflicts with the supplied NIST SRM, DHZ and Dana ORE sample databases). The second line contains the standard description field in double quotes. It may be empty. The third line contains the DisplayAsOxide (0 = do not display as oxide composition, non-zero = display as oxide composition) flag and the number of elements in the standard composition. The fourth line contains the element symbols in double quotes for each element in the standard composition. The fifth and sixth lines contain the number of cations followed by the number of oxygens for each element (required even for elements not displayed as oxides). The seventh line contains the composition in elemental weight percent for each element in the standard composition.

Note that all elements must be specified, even oxygen for silicate standards (even if the DisplayAsOxide flag is true).

Export

The Export menu allows the user to export an ASCII file of standard compositions. This may be useful for transferring compositional standard data to another application or simply to have a text file backup of the standard compositions. This text format is suitable for importing back into Standard for Windows after manually editing the entries if desired.

File Information

This menu displays and allows the user to change the run title and/or description fields for the currently open standard database.

Print Log

This menu allows the user to print entire contents of the log window or the selected contents of the log window.

Print Setup

This menu allows the user to change the default printer or printer setup options for the currently selected printer.

Exit

This menu exits the Standard for Windows program. Note that Standard for Windows automatically saves any changes to the standard database automatically as they are made using transaction processing methods so there is no chance of data loss.

Edit

Cut

This menu cuts the selected text from the log window to the Windows Clipboard.

Copy

This menu copies the selected text from the log window to the Windows Clipboard.

Paste

This menu pastes text from the Windows Clipboard to the log window.

Select All

This menu selects all text in the log window.

Clear All

This menu clears all text from the log window.

Standard

New

To create a new standard composition simply click the Standard | New menu. From the Standard Composition window enter each element and it's concentration by clicking on a blank element grid row. To change a previously entered element concentration or cation ratio for oxide entered or displayed standards, simply click an already existing element row.

The program will automatically specify a default standard number based on the next free standard number starting at 1.

If may be desirable to group the standards numbers using some sort of a numbering system. In this case it will be necessary to change the suggested standard number. Generally some thought should be given to arranging the various standards together into functional groups. For example, it might be helpful to reserve standard numbers 1 - 106 for the elemental metal standards based on atomic number. Or perhaps to reserve standard numbers 200 - 500 for silicate standards only.

Note that if an already existing standard number is entered, the program will prompt whether the existing standard should be overwritten. If not, select another standard number. Note also that standard numbers between 1 and 2000 should normally be used to avoid conflicts or confusion with the standard numbers in the DHZ.DAT, SRM.DAT and ORE.DAT files.

Also enter the standard name and optionally a description or comment field to indicate the standard source or other pedigree information.

If the standard is a simple compound, it may be easier to enter the composition as a formula string. Click the Enter Atom Formula Composition to enter the formula string, for example fayalite entered as fe_2sio_4. To enter a formula string note that the parser is able to handle multiple occurrences of an element and even the use of parentheses. For example: "ca2mg5si8o22(oh)2"

All standard compositions are saved to the standard database as elemental concentrations, therefore, be sure to enter the oxygen concentration ***if*** oxygen is present in the compound. This applies to ***all*** standards, even those which are entered and/or displayed as oxide concentrations. To facilitate the data entry for the oxygen concentration of standard compositions which are entered as oxide concentrations, the program will display the calculated oxygen of the cations entered thus far. Therefore, if oxygen is entered as the last element in the standard, it is a simple matter to specify the displayed oxygen concentration from the calculated oxygen of the cations.

Be sure to also enter excess oxygen (for example minerals that contain ferric iron), into the standard composition since the presence of the excess oxygen will affect the overall calculation of the standard k-factors and mean atomic number.

For example, to enter hematite (Fe_2O_3) as a composition, one must enter two atoms of Fe and three atoms of O. Using elemental weight percent entry, enter 69.94 wt. % Fe and 30.06 wt. % O. Using the default oxide entry, of Fe expressed as FeO and elemental O, that would be two molecules of FeO and one molecule of O, or 89.98 wt. % FeO and 10.02 wt. % O (the excess oxygen). However, if the cations for Fe are changed to two atoms of Fe and three atoms of oxygen, then one can input the composition as oxide simply by entering 100% Fe_2O_3.

Note that "oxide" displayed standards that contain fluorine and/or chlorine will sum greater than 100 %, since these halogens actually replace some of the oxygen that is already being included in the oxide formulas of the other cations.

The oxygen equivalent that is being included in the composition is usually small and can generally be ignored. However, for the most accurate analysis it is recommended that the standard containing fluorine and/or chlorine be entered as an elemental standard composition. That way, the oxygen can either be calculated by stoichiometry or measured directly along with the halogens. The only missing component then will be hydrogen present in water or hydroxyl which is usually less than 1 % of the total.

Formula Entry

The Formula Entry compositional string must be entered as a string of characters for element symbols and numbers for subscripts. For example :

Fe2SiO4 :

Enter as : "fe2sio4"

CaCO3

Enter as "caco3"

Note that subscripts of 1 need not be specified. If an element is present in the formula string more than once, the program will automatically sum the subscripts. Do not use fractional subscripts.

Note that because some formulas are "ambiguous" when parsed one character at a time, use both upper and lower case characters to eliminate confusing the formula parser. For example: "yba2cu3o7" will be interpreted as yttrium, boron and an undefined element "a". Instead, enter the string as "YBa2Cu3O7" and the formula will be parsed correctly.

Modify

This menu item allows the user to modify an existing standard name or composition without re-entering all the element symbols and concentrations. The user must enter the compositional changes in elemental or oxide weight percent when modifying an existing standard composition. Simply click on the element row in the grid to pull up the Enter Composition dialog. Enter the indicated elemental or oxide weight percent and click OK to save the changes.

This window may also be used to temporarily change the default x-ray line and cations for a specific standard for calculation purposes. This is often useful to explore the effect on the k-ratios of various operating voltages or selection of alternative x-ray lines.

Delete

Allows the user to delete an existing standard from the standards database file. Note that this is a permanent deletion from the standards database. The program will ask you to confirm the deletion. If it becomes necessary to use the standard again for quantitative analysis, it will have to be re-entered using the New menu item above.

Be careful about deleting any standards from the database. Remember, if a standard has been deleted that has been used in a quantitative run and later use that run as a setup or simply want to re-process the data again, the program will be unable to load that standard composition or even worse, might load the wrong composition if the standard was deleted and the same number was used for another standard composition.

Delete Selected

Allows the user to delete selected standards from the database. Each standard will require a confirmation before it can be deleted.

Be careful about deleting any standards from the database. Remember, if a standard has been deleted that has been used in a quantitative run and later use that run as a setup or simply want to re-process the data again, the program will be unable to load that standard composition or even worse, might load the wrong composition if the standard was deleted and the same number was used for another standard composition.

List Standard Names

This menu item will type out all standard numbers and names to the log window. This list can then be printed out or cut or copied and pasted to another document for reference purposes.

List Selected Standards

This menu item will type out the standard compositions and k-factors for the selected standards to the log window. To select a standard just click a standard in the standard list, to select additional standards, simply hold the <crtl> or <shift> keys and click additional standards. The <crtl> key selects or deselects individual standards, the <shift> key selects all standards in a range.

If the list of standards is very long, it may be necessary to select the Save to Disk Log menu so that the output is automatically saved to disk. This file can then be opened using the View Disk Log menu and printed out if desired.

List All Standards

This menu item will type out the standard compositions and k-factors for all standards in the standard database to the log window.

If the list of standards is very long, it may be necessary to select the Save to Disk Log menu so that the output is automatically saved to disk. This file can then be opened using the View Disk Log menu and printed out if desired.

Options

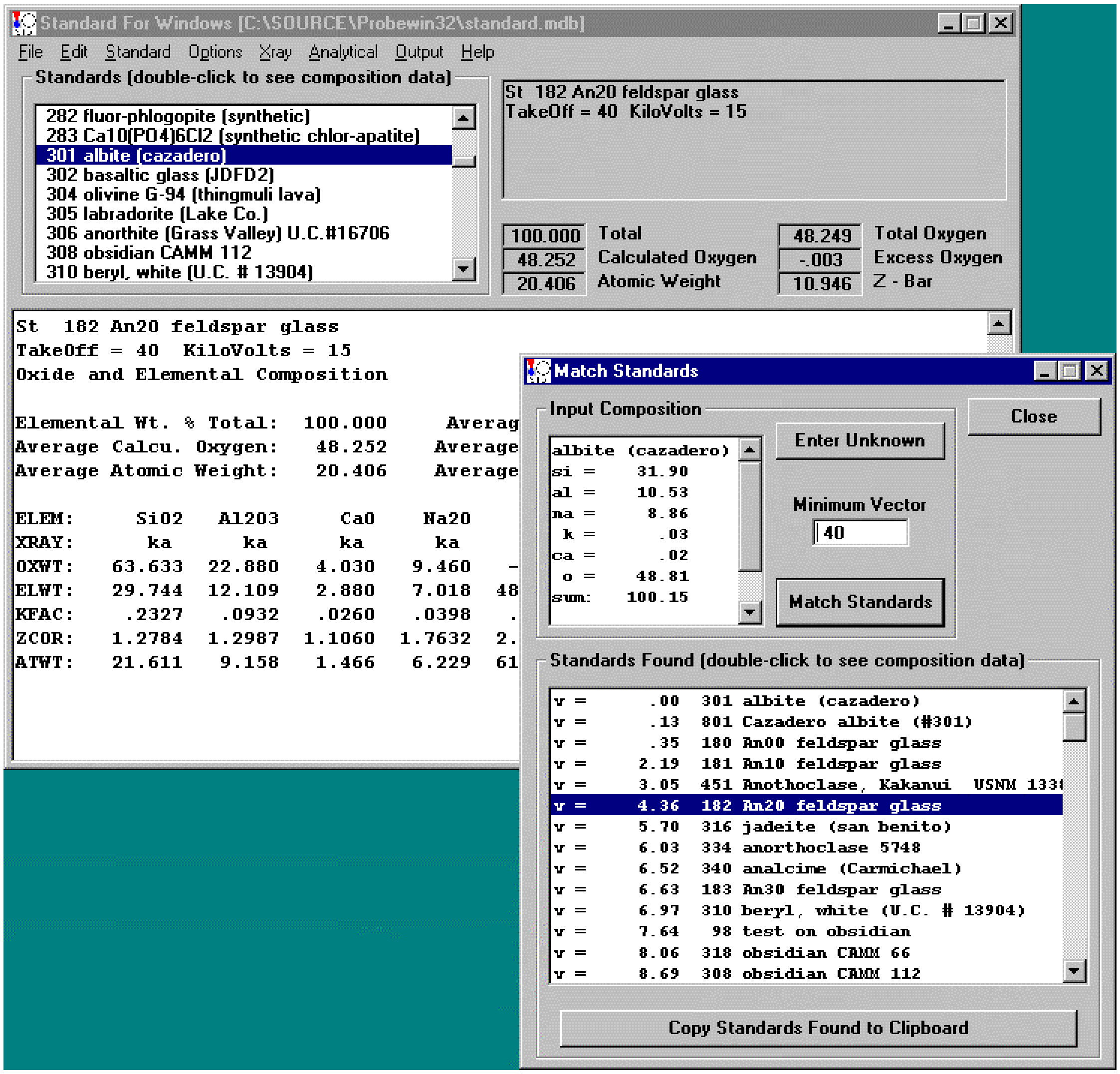
Find

Use this option to obtain a list of all standards that contain a specified element within a specified concentration range. To see the composition of the standards, simply double click the standard in the returned list and the composition will be output to the log window. This command is useful for determining, for instance, what standards contain between 10 and 20 weight percent Fe.

To copy the list of standards found to the Window clipboard, click the Copy to Clipboard button.

Match

This dialog is used to match a given standard or unknown composition to the standards in the standard database. Can be used to select the closest standard to an unknown sample for quantitative analysis or to suggest possible mineral identification.



Standards (double-click to see composition data)

```
282 fluor-phlogopite (synthetic)
283 Ca10(PO4)6Cl2 (synthetic chlor-apatite)
301 albite (cazadero)
302 basaltic glass (JDFD2)
304 olivine G-94 (thingmuli lava)
305 labradorite (Lake Co.)
306 anorthite (Grass Valley) U.C.#16706
308 obsidian CAMM 112
310 beryl, white (U.C. # 13904)
```

```
St  182 An20 feldspar glass
TakeOff = 40  KiloVolts = 15
```

100.000	Total	48.249	Total Oxygen
48.252	Calculated Oxygen	-.003	Excess Oxygen
20.406	Atomic Weight	10.946	Z - Bar

```
St  182 An20 feldspar glass
TakeOff = 40  KiloVolts = 15
Oxide and Elemental Composition

Elemental Wt. % Total:  100.000     Averag
Average Calcu. Oxygen:   48.252    Average
Average Atomic Weight:   20.406    Average

ELEM:      SiO2   Al2O3     CaO    Na2O
XRAY:       ka      ka      ka      ka
OXWT:   63.633  22.880   4.030   9.460   -
ELWT:   29.744  12.109   2.880   7.018  48
KFAC:    .2327   .0932   .0260   .0398   .
ZCOR:   1.2784  1.2987  1.1060  1.7632  2.
ATWT:   21.611   9.158   1.466   6.229  61
```

Input Composition

```
albite (cazadero)
si =     31.90
al =     10.53
na =      8.86
 k =       .03
ca =       .02
 o =     48.81
sum:    100.15
```

Minimum Vector: 40

Standards Found (double-click to see composition data)

```
v =      .00  301 albite (cazadero)
v =      .13  801 Cazadero albite (#301)
v =      .35  180 An00 feldspar glass
v =     2.19  181 An10 feldspar glass
v =     3.05  451 Anothoclase, Kakanui  USNM 133
v =     4.36  182 An20 feldspar glass
v =     5.70  316 jadeite (san benito)
v =     6.03  334 anorthoclase 5748
v =     6.52  340 analcime (Carmichael)
v =     6.63  183 An30 feldspar glass
v =     6.97  310 beryl, white (U.C. # 13904)
v =     7.64   98 test on obsidian
v =     8.06  318 obsidian CAMM 66
v =     8.69  308 obsidian CAMM 112
```

The dialog will display the last selected standard as a default composition to match to. To match to an arbitrary unknown composition, click the Enter Unknown button and enter a compositional string in weight percent.

The routine automatically re-calculates all analyses in mole or atomic percents for the vector fit using a sum of the square of the differences algorithm. The following is an example of the output from the "match" command after entering a composition of 70.51% of FeO and 29.49% of SiO_2 (fayalite) using the DHZ database :

```
Unknown composition:
    fe      si
   70.51   29.49     .00     .00     .00     .00     .00     .00
Sum =   100.00
STD =    5  Fayalite, East Greenland, p. 4          MATCH = 6.718
STD =    4  Fayalite, Pantelleria, p. 4             MATCH = 12.81
STD =   68  Grunerite, U.S.A., p. 151               MATCH = 30.25
STD =   50  Orthoferrosilite, p. 105                MATCH = 34.24
STD =  183  Titaniferous magnetite, p. 427          MATCH = 38.97
```

Once the list of sorted standards has been loaded (decreasing match vector) each standard may be viewed by simply double clicking it in the list. The output will be automatically directed to the log window.

To copy the list of standards matched to the Window clipboard, click the Copy to Clipboard button.

It may be most useful to open one of the supplied databases for matching an unknown composition depending on the circumstances. See the section on the File | Import menu.

Weight Percent Entry

The weight percent entry compositional string must be entered as a string of element and weight percent pairs. For example :

Albite :

ELEM:	SiO2	Al2O3	Na2O	O
ELWT:	32.132	10.289	8.769	48.810

Enter as : "si32al10na9o48"

YAG :

ELEM:	Y2O3	Al2O3	O
ELWT:	44.939	22.721	32.339

Enter as : " y45al23o32"

Lanthanum Hexaboride :

ELEM:	La	B
ELWT:	68.170	31.830

Enter as : " la68b31"

Note that numbers less than 1 percent must entered as 1 since the string parser does not support fractional weight percents using a decimal point.

Note that because some formulas are "ambiguous" when parsed one character at a time, use both upper and lower case characters to eliminate confusing the formula parser. For example: "yba2cu3o7" will be interpreted as yttrium, boron and an undefined element "a". Instead, enter the string as "YBa2Cu3O7" and the formula will be parsed correctly.

Modal Analysis

This menu item is used to perform a quantitative modal analysis of unknown compositions, based on a group of modal phases that are defined by selecting standard compositions from a standard database. Any database of standard compositions may be used to define the phases, since the modal groups thus defined are stored within each standard database and may be recalled for use with subsequent data sets.

Typically this procedure is performed on a large set of compositional data acquired using multiple traverses or gridded locations that are statistically representative of the material in question. The input file must contain the oxide or element symbols for each column of data enclosed in double quotes, in the first line of the data file. The file must not contain a totals column.

The number of matches and abundance of each phase in the group will be tabulated and the average and standard deviation of each phase calculated. A sample output is shown here for a silicate modal analysis :

```
Modal Group  1 silicate
```

```
Minimum Total =  95

Phase  1 plag
  Minimum Vector =  4
  End Member = Feldspar
     301 albite (cazadero)
     306 anorthite (Grass Valley) U.C.#16706

Phase  2 alka
  Minimum Vector =  4
  End Member = Feldspar
     301 albite (cazadero)
     374 orthoclase MAD-10

Phase  3 oliv
  Minimum Vector =  4
  End Member = Olivine
     263 Fe2SiO4 (synthetic fayalite)
     273 Mg2SiO4 (magnesium olivine) synthetic

Phase  4 opyx
  Minimum Vector =  4
  End Member = Pyroxene
     350 enstatite 25-25
     469 hypersthene, johnstown USNM #746

Phase  5 cpyx
  Minimum Vector =  4
  End Member = Pyroxene
     358 diopside (Chesterman)
     453 augite, kakanui USNM 122142

    Line  Vector   Phase          End  -  Member             Sum    SiO2     FeO     CaO   Al2O3     MgO    Na2O
       1     .30    opyx  Wo  8.  En 65.  Fs 27.       .   98.70   52.80   17.70    4.06     .44   23.51     .19
       2     .93    opyx  Wo  3.  En 47.  Fs 50.       .   98.48   50.08   29.90    1.44    1.23   15.78     .05
       3    2.14    opyx  Wo  2.  En 82.  Fs 16.       .   98.94   50.55   12.18    1.22     .62   34.37     .00
       4     .24    oliv  Fo 84.  Fa 16.       .       .   99.61   37.18   15.45     .09     .33   46.56     .00
       5     .98    opyx  Wo  1.  En 87.  Fs 13.       .   99.20   54.66    9.28     .29     .09   34.88     .00
       6  ------  ------       .       .       .       .   82.11   40.66    7.28     .21     .08   33.88     .00
       7     .05    oliv  Fo 84.  Fa 16.       .       .  100.08   38.55   15.79     .06     .55   45.13     .00
       8     .00    oliv  Fo 85.  Fa 15.       .       .  101.46   40.37   14.91     .08     .00   46.10     .00
       9     .05    oliv  Fo 83.  Fa 17.       .       .  100.00   38.62   16.39     .06     .00   44.93     .00
      10     .06    oliv  Fo 83.  Fa 17.       .       .  100.16   38.47   16.23     .03     .29   45.14     .00
      11     .02    oliv  Fo 96.  Fa  4.       .       .   99.99   41.07    4.30     .00     .56   54.06     .00
      12     .68    opyx  Wo  1.  En 87.  Fs 12.       .   99.08   55.33    8.44     .70     .16   34.45     .00
      13     .08    oliv  Fo 83.  Fa 17.       .       .   97.86   37.46   15.97     .01     .03   44.39     .00
      14     .07    oliv  Fo 84.  Fa 16.       .       .  100.50   38.51   15.58     .32     .40   45.69     .00
      15     .01    oliv  Fo 84.  Fa 16.       .       .  101.09   39.76   15.53     .05     .00   45.75     .00
      16     .12    oliv  Fo 84.  Fa 16.       .       .   98.92   37.73   15.36     .02     .03   45.78     .00
      17     .00    oliv  Fo 84.  Fa 16.       .       .  101.15   40.03   15.59     .01     .00   45.52     .00
      18    5.64  ------       .       .       .       .   97.77   48.90   29.60    7.96    3.86    6.87     .58
      19   17.73  ------       .       .       .       .   98.53   51.92   31.90     .00    1.85     .00   12.86
      20     .59    cpyx  Wo 42.  En 48.  Fs 10.       .   98.64   52.92    6.20   19.97    2.80   16.40     .35
      21  ------  ------       .       .       .       .   69.50   38.92    6.20   11.97    1.80   10.40     .21
      22     .17    oliv  Fo 83.  Fa 17.       .       .  100.64   37.94   16.66     .04     .02   45.98     .00
      23     .09    oliv  Fo 83.  Fa 17.       .       .   97.01   36.81   16.07     .26     .28   43.59     .00
      24     .05    oliv  Fo 84.  Fa 16.       .       .  100.08   38.55   15.79     .06     .55   45.13     .00
      25     .00    oliv  Fo 85.  Fa 15.       .       .  101.46   40.37   14.91     .08     .00   46.10     .00
      26     .02    alka  Ab100.  An   .  Or   .       .   98.65   67.84     .05     .00   19.65     .04   11.07
      27     .00    plag  Ab 85.  An 15.  Or   .       .  100.33   64.10     .17    3.26   22.66     .25    9.89
      28     .05    oliv  Fo 83.  Fa 17.       .       .  100.00   38.62   16.39     .06     .00   44.93     .00
      29     .06    oliv  Fo 83.  Fa 17.       .       .  100.16   38.47   16.23     .03     .29   45.14     .00
      30     .08    oliv  Fo 83.  Fa 17.       .       .   97.22   37.16   16.25     .05     .02   43.74     .00
      31     .00    plag  Ab 91.  An  9.  Or   .       .  100.44   66.12     .00    2.02   21.66     .00   10.64
      32     .07    oliv  Fo 83.  Fa 17.       .       .  100.39   38.57   16.57     .02     .00   45.23     .00
      33     .97    oliv  Fo 83.  Fa 17.       .       .   99.96   42.15   14.88    1.17    1.82   39.94     .00
      34     .08    oliv  Fo 82.  Fa 18.       .       .  100.27   38.28   17.31     .05     .00   44.63     .00
      35     .01    oliv  Fo 83.  Fa 17.       .       .   97.14   38.14   15.69     .23     .25   42.83     .00
      36    1.90    oliv  Fo 80.  Fa 20.       .       .   98.26   43.94   15.86    1.00    1.01   36.45     .00
      37     .07    oliv  Fo 82.  Fa 18.       .       .   99.87   38.17   17.35     .05     .00   44.30     .00
      38     .66    opyx  Wo  9.  En 76.  Fs 15.       .   96.47   50.60    9.91    4.57    4.07   27.32     .00
      39     .77    opyx  Wo  1.  En 83.  Fs 16.       .   98.26   53.16   11.34     .42     .59   32.75     .00

Results of Modal Analysis

InputFile    : C:\SOURCE\PROBEWIN\modal.dat
OutputFile   : C:\SOURCE\PROBEWIN\modal.out
Date and Time: 5/22/97 9:29:44 PM

Group Name   : silicate
Total Number of Points in File :  39
Valid Number of Points in File :  37
Match Number of Points in File :  35

Minimum Total for Valid Points :    95.00
Percentage of Valid Points :     94.9
Percentage of Match Points :     89.7
```

```
   Phase  #Match  %Total  %Valid  %Match  AvgVec
    plag       2     5.1     5.4     5.7     .00
                End  -  Member             Sum    SiO2     FeO     CaO   Al2O3     MgO    Na2O
Average:  Ab 88.  An 12.  Or   .       .  100.38   65.11     .09    2.64   22.16     .13   10.27
Std Dev:      4.      4.       .       .     .08    1.43     .12     .88     .71     .18     .53

   Phase  #Match  %Total  %Valid  %Match  AvgVec
    alka       1     2.6     2.7     2.9     .02
                End  -  Member             Sum    SiO2     FeO     CaO   Al2O3     MgO    Na2O
Average:  Ab100.  An   .  Or   .       .   98.65   67.84     .05     .00   19.65     .04   11.07
Std Dev:       .       .       .       .     .00     .00     .00     .00     .00     .00     .00

   Phase  #Match  %Total  %Valid  %Match  AvgVec
    oliv      24    61.5    64.9    68.6     .18
                End  -  Member             Sum    SiO2     FeO     CaO   Al2O3     MgO    Na2O
Average:  Fo 84.  Fa 16.       .       .   99.72   38.96   15.46     .16     .27   44.88     .00
Std Dev:      3.      3.       .       .    1.31    1.67    2.47     .30     .42    2.93     .00

   Phase  #Match  %Total  %Valid  %Match  AvgVec
    opyx       7    17.9    18.9    20.0     .92
                End  -  Member             Sum    SiO2     FeO     CaO   Al2O3     MgO    Na2O
Average:  Wo  4.  En 75.  Fs 21.       .   98.45   52.45   14.11    1.81    1.03   29.01     .03
Std Dev:      4.     15.     14.       .     .93    2.10    7.60    1.76    1.39    7.24     .07

   Phase  #Match  %Total  %Valid  %Match  AvgVec
    cpyx       1     2.6     2.7     2.9     .59
                End  -  Member             Sum    SiO2     FeO     CaO   Al2O3     MgO    Na2O
Average:  Wo 42.  En 48.  Fs 10.       .   98.64   52.92    6.20   19.97    2.80   16.40     .35
Std Dev:       .       .       .       .     .00     .00     .00     .00     .00     .00     .00
```

Creating an Input File

The easiest way to create an input file for the modal analysis procedure is to use the Plot! window in Probe for Windows to output a .DAT file of the oxide or elemental weight percent compositions to disk. Be sure to select output of column labels so that the modal analysis routine can identify the elements in the input file.

One can also use an input file from any other source, just so long as the oxide or element symbols are the first line of the file and the data is in weight percent. The weight percent data can be in any format since the file is read as unformatted ASCII. However, the element symbols in the first line must be enclosed within double quotes.

A sample input file is shown below :

```
"SiO2"   "FeO"    "CaO"    "Al2O3"  "MgO"    "Na2O"
 52.8     17.7     4.06       .44    23.51      .19
 50.08    29.9     1.44      1.23    15.78      .05
 50.55    12.18    1.22       .62    34.37     0.0
 37.18    15.45     .09       .33    46.56     0.0
 54.66     9.28     .29       .09    34.88     0.0
 40.66     7.28     .21       .08    33.88     0.0
 38.55    15.79     .06       .55    45.13     0.0
 40.37    14.91     .08       .00    46.10     0.0
 38.62    16.39     .06       .00    44.93     0.0
 38.47    16.23     .03       .29    45.14     0.0
 41.07     4.3     0.0        .56    54.06     0.0
 55.33     8.44     .70       .16    34.45     0.0
 37.46    15.97     .01       .03    44.39     0.0
 38.51    15.58     .32       .40    45.69     0.0
 39.76    15.53     .05       .00    45.75     0.0
 37.73    15.36     .02       .03    45.78     0.0
 40.03    15.59     .01       .00    45.52     0.0
 48.90    29.6     7.96      3.86     6.87      .58
 51.92    31.9     0.0       1.85     0.0     12.86
 52.92     6.2    19.97      2.8     16.4       .35
 38.92     6.2    11.97      1.8     10.4       .21
 37.94    16.66     .04       .02    45.98     0.0
 36.81    16.07     .26       .28    43.59     0.0
 38.55    15.79     .06       .55    45.13     0.0
```

Modal Analysis Options

Minimum Total- Unknown compositions that sum below this value will not be used in the point count analysis. A default minimum sum of 90% or 95% is suggested. Increase the minimum total value to reject points that sum lower.

Normalize Concentrations For Fit- This option is used to specify if the standard and unknown concentrations should be normalized to 100% before the vector fit is calculated. Of course, this applies only to those concentrations that have already met the Minimum Total tolerance.

Weight Concentrations For Fit- This option is used to specify if the element concentrations for the standards that define each phase should be weighted based on the average composition of the element in that phase. If the major elements in a phase should have a greater influence in determining the vector fit, then this option should be selected. If all concentrations, regardless of magnitude should have equal weight in the vector fit, this option should not be selected.

Minimum Vector- The tolerance for the match to a defined phase. A default value of 4.0 is suggested. If a closer match is desired for one or more phases in the group, decrease the vector value for that phase. The vector is calculated by simply taking the square root of the sums of the differences squared to each n-dimensional phase. Note that all weight percents, for both standards and unknowns, are normalized to 100% prior to the vector fit. The calculation is shown below for a one dimensional phase :

$$V = \sqrt{\sum_{i=1}^{n}(X_i - X_i')^2}$$

Where X_i and X_i' are the components (elements) to be compared

Mineral end-member- A mineral end-member calculation for one of the following phases, Feldspar, Olivine, Pyroxene and Garnet may also be specified.

Defining Modal Phases

When defining the phase compositions to match, try to avoid over-determining the phase. For example, in defining an olivine phase you should only use two standards (preferably forsterite and fayalite) since the range of composition is linear. For example, if you enter more than two compositions in the above linear compositional system (for instance an Fo50 composition standard) you could over define the numerical system because the solution to the unknown composition is no longer unique. Note that the routine will always give a correct result, however the calculation will become more unstable as it is over-defined.

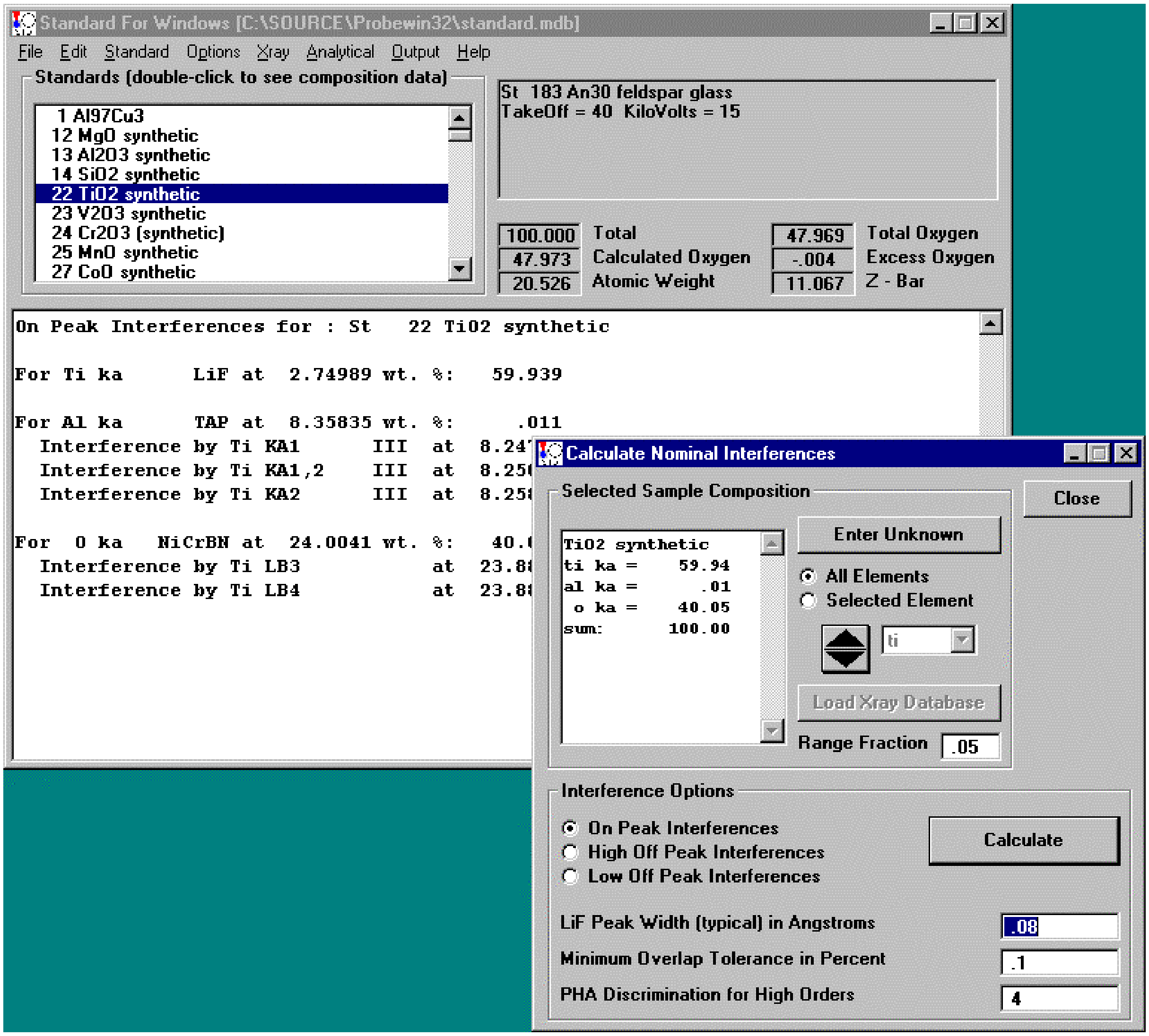
The entire printed output may be captured by selecting the Output | Save Log To Disk menu although an output file of the results for each input line is automatically saved. The output file will contain the vector fit, matched phase, end-member calculation (if selected), sum and composition of each line in the input file.

The printed summary lists the total number of analyses, the minimum total for a valid composition, number of valid points that sum above the minimum sum, the number of matched points and the percentage of points that were matched.

In addition, for each phase, the output will contain the phase name, the number of matches for that phase, the percent of matched points, valid points and total matched points for the matches in that phase. This is followed by the average end-member (if selected), weight percent sum and composition for that phase and the standard deviation for each element.

Interferences

This command will accept a composition (of either a standard number in the standard database or an unknown composition entered by the user) and calculate the approximate percent spectral interference overlap on all interfered lines based on the concentrations, the x-ray line intensities of all known lines, a user defined gaussian peak width, and a PHA discrimination factor for higher order lines.



1 Al97Cu3
12 MgO synthetic
13 Al2O3 synthetic
14 SiO2 synthetic
22 TiO2 synthetic
23 V2O3 synthetic
24 Cr2O3 (synthetic)
25 MnO synthetic
27 CoO synthetic

St 183 An30 feldspar glass
TakeOff = 40 KiloVolts = 15

100.000 Total
47.973 Calculated Oxygen
20.526 Atomic Weight
47.969 Total Oxygen
-.004 Excess Oxygen
11.067 Z - Bar

```
On Peak Interferences for : St   22 TiO2 synthetic

For Ti ka      LiF at  2.74989 wt. %:   59.939

For Al ka      TAP at  8.35835 wt. %:     .011
  Interference by Ti KA1       III  at  8.24
  Interference by Ti KA1,2     III  at  8.25
  Interference by Ti KA2       III  at  8.25

For  O ka   NiCrBN at  24.0041 wt. %:   40.
  Interference by Ti LB3            at  23.8
  Interference by Ti LB4            at  23.8
```

```
TiO2 synthetic
ti ka =     59.94
al ka =       .01
 o ka =     40.05
sum:       100.00
```

● All Elements
○ Selected Element
ti

Range Fraction .05

● On Peak Interferences
○ High Off Peak Interferences
○ Low Off Peak Interferences

LiF Peak Width (typical) in Angstroms .08
Minimum Overlap Tolerance in Percent .1
PHA Discrimination for High Orders 4

The percent interference is based on the actual concentrations of both the interfered and interfering elements. For example, if the concentration of the interfered element is stated as 5.3 wt. % and the percent interference is calculated to be 10%, then the apparent concentration that might be measured is 10% greater than the actual concentration, or 5.3 plus 0.53 (that is, 10% of 5.3) which equals 5.83 wt. %. Note that since this is only a nominal calculation, it should only be used as an indicator of possible problematic analytical situations.

The interference calculation uses the x-ray line and crystal defaults as defined in the ELEMENTS.DAT file (usually found in C:\Program Files\Probe for Windows). A sample interference output for a biotite standard is shown here :

```
 ******************************************************************************
 STD 112 biotite #3                               MZbar=   10.688 Zbar=   13.413
```

```
LMNT:   SiO2     Al2O3    FeO      MgO      CaO      Na2O     K2O      TiO2
OXWT:  38.620   10.720   18.130   14.010     .020     .690    9.210    2.290
ELWT:  18.052    5.674   14.093    8.448     .014     .512    7.646    1.373
K-FAC   .1435    .0407    .1206    .0571    .0001    .0027    .0696    .0120
Z-COR  1.2583   1.3931   1.1688   1.4808   1.0749   1.9290   1.0987   1.1443
MPCT:  34.560    5.653   13.568   18.690     .019     .599    5.257    1.541
APCT:  11.520    2.261    6.784    9.345     .010     .399    3.505     .514

LMNT:   MnO      BaO      Rb2O     Cl       F        H2O
OXWT:     .950     .110     .030     .020    4.020    2.660
ELWT:     .736     .099     .027     .020    4.020     .298
K-FAC   .0062    .0007    .0002    .0002    .0130    .0000
Z-COR  1.1881   1.3255   1.3704   1.1777   3.0936    .0000
MPCT:     .720     .039     .009     .030   11.378    7.939
APCT:     .360     .019     .006     .030   11.378    5.293

TOTAL WEIGHT PERCENT :  101.480            TOTAL OXYGEN FROM CATIONS :   40.469
AVERAGE ATOMIC WEIGHT :  21.844             AVERAGE MOLECULAR WEIGHT :   54.564

LiF peak width =       .080 Angstroms
Overlap tolerance =       .100 Percent
PHA discrimination factor =      4.000

For Si  ka at     7.1262       38.62 wt.% :

For Al  ka at     8.3401       10.72 wt.% :

For Fe  ka at     1.9373       18.13 wt.% :

For Mg  ka at     9.8900       14.01 wt.% :

For Ca  ka at     3.3595         .02 wt.% :
Interference by K  SKB^4    I    at     3.4040   is      1.2 % overlap.
Interference by K  SKB'''   I    at     3.4116   is       .1 % overlap.

For Na  ka at    11.9101         .69 wt.% :

For K   ka at     3.7424        9.21 wt.% :

For Ti  ka at     2.7497        2.29 wt.% :
Interference by Ba LA1      I    at     2.7767   is       .4 % overlap.

For Mn  ka at     2.1031         .95 wt.% :

For Ba  la at     2.7759         .11 wt.% :
Interference by Ti SKA3     I    at     2.7324   is       .1 % overlap.
Interference by Ti SKA3''   I    at     2.7367   is       .3 % overlap.
Interference by Ti SKA'     I    at     2.7399   is       .5 % overlap.
Interference by Ti SKA''    I    at     2.7464   is      1.8 % overlap.
Interference by Ti KA1      I    at     2.7490   is    270.0 % overlap.
Interference by Ti KA1,2    I    at     2.7502   is    486.0 % overlap.
Interference by Ti KA2      I    at     2.7527   is    230.7 % overlap.
Interference by Rb KA1      III  at     2.7771   is       .4 % overlap.
Interference by Rb KA1,2    III  at     2.7813   is       .6 % overlap.
Interference by Rb KA2      III  at     2.7897   is       .1 % overlap.

For Rb  la at     7.3183         .03 wt.% :

For Cl  ka at     4.7288         .02 wt.% :
Interference by Ba LB2      II   at     4.8092   is       .2 % overlap.

For F   ka at    18.3200        4.02 wt.% :
Interference by Fe LA2      I    at    17.5860   is      1.0 % overlap.
Interference by Fe LA1      I    at    17.5860   is      8.5 % overlap.
Interference by Mg SKB^7    II   at    18.4091   is       .1 % overlap.
Interference by Mn LB1      I    at    19.1034   is       .2 % overlap.
```

The default gaussian peak width is just a nominal value, increasing the width will produce greater overlaps. The PHA discrimination factor can adjust the contribution from higher order lines. The default of 4.0 will divide the intensity of each subsequent order by 4, before calculating the percent overlap. This means that each higher order line will be only

1/4 or 25 % of the intensity of the previous order. To decrease the contribution of higher order lines, increase the PHA discrimination factor.

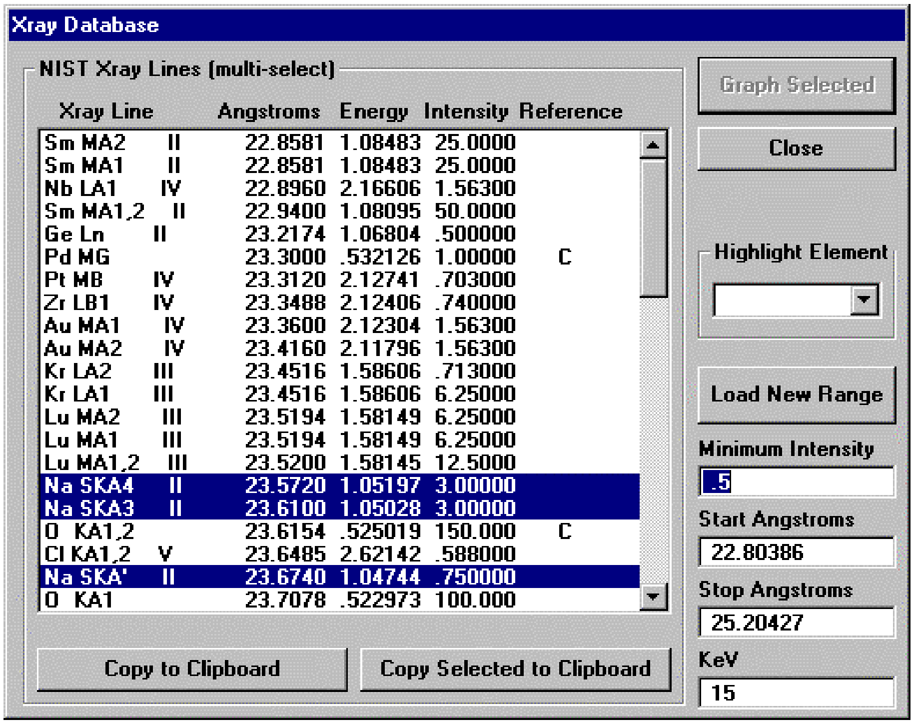
This calculation is for use only as a guideline and is not intended for use as a quantitative spectral interference correction. Probe for Windows contains special algorithms to empirically determine the actual spectral interferences based on measured overlaps.

The dialog will use the composition of the currently selected standard as a default composition. See the section on Weight Percent Entry under the Options | Match menu for instructions on entering an arbitrary unknown composition for calculation of nominal interferences.

X-Ray

X-Ray Database

This menu allows the user to view specified ranges of the NIST x-ray wavelength database. This database covers the wavelength range from approximately 0.5 to 200.0 angstroms and includes higher order reflections as well. Higher order reflections are reduced in intensity by 25% for each subsequent order to simulate the effect of PHA analysis.



Xray Line	Angstroms	Energy	Intensity	Reference
Sm MA2 II	22.8581	1.08483	25.0000	
Sm MA1 II	22.8581	1.08483	25.0000	
Nb LA1 IV	22.8960	2.16606	1.56300	
Sm MA1,2 II	22.9400	1.08095	50.0000	
Ge Ln II	23.2174	1.06804	.500000	
Pd MG	23.3000	.532126	1.00000	C
Pt MB IV	23.3120	2.12741	.703000	
Zr LB1 IV	23.3488	2.12406	.740000	
Au MA1 IV	23.3600	2.12304	1.56300	
Au MA2 IV	23.4160	2.11796	1.56300	
Kr LA2 III	23.4516	1.58606	.713000	
Kr LA1 III	23.4516	1.58606	6.25000	
Lu MA2 III	23.5194	1.58149	6.25000	
Lu MA1 III	23.5194	1.58149	6.25000	
Lu MA1,2 III	23.5200	1.58145	12.5000	
Na SKA4 II	23.5720	1.05197	3.00000	
Na SKA3 II	23.6100	1.05028	3.00000	
O KA1,2	23.6154	.525019	150.000	C
Cl KA1,2 V	23.6485	2.62142	.588000	
Na SKA' II	23.6740	1.04744	.750000	
O KA1	23.7078	.522973	100.000	

Minimum Intensity: .5
Start Angstroms: 22.80386
Stop Angstroms: 25.20427
KeV: 15

The x-ray list range (Start Angstroms and Stop Angstroms) and minimum search intensity (Minimum Intensity) of the x-ray list can be specified by the user. Note that since the list is limited to about 1000 items, the program will automatically increase the minimum intensity until the returned list of x-ray lines is less than 1000. To view x-ray lines of lesser intensity, simply reduce the x-ray range and click the Load New Range button.

Description of the X-ray Database (adapted from NIST documentation by C. Fiori)

The NIST x-ray database is based on 4985 (1st order) entries and includes all the measurable x-ray lines, satellites and absorption edges from under 100 eV to over 120 keV. Additionally, most of the x-ray lines and satellites are assigned a relative intensity (relative to the alpha-1 line in each family). The data base was assembled primarily from four sources:

1.) B.L. Doyle, W.F. Chambers, T.M. Christensen, J.M. Hall and G.H. Pepper "SINE THETA SETTINGS FOR X-RAY SPECTROMETERS", Atomic Data and Nuclear Data Tables Vol. 24, No 5, 1979.

2.) E.W. White, G.V. Gibbs, G.G. Johnson Jr. and G.R. Zechman "X-RAY WAVELENGTHS AND CRYSTAL INTERCHANGE SETTINGS FOR WAVELENGTH GEARED CURVED CRYSTAL SPECTROMETERS" Report of the Pennsylvania State Univ., 1964.

3.) J.A. Bearden "X-RAY WAVELENGTHS AND X-RAY ATOMIC ENERGY LEVELS" Rev. Mod. Phys., Vol. 39, No. 78, 1967.

4.) J.A Bearden and A.F. Burr,"REEVALUATION OF X-RAY ATOMIC ENERGY LEVELS", Rev. Mod. Phys., Vol. 31, No. 1, 1967.

Each x-ray line or edge series as a function of atomic number was fit to a fourth degree polynomial. The fit was subtracted from the appropriate data and the residuals plotted and examined. In this way rogue entries could be identified and corrected. The resulting data base is considered to be sufficiently accurate for any application involving the Si (Li) x-ray detector and single crystal wavelength spectrometers.

Note that the last entry in the x-ray database window gives a code for the source of the entry. If the column is blank the source is reference 2. If the column contains the letter "C" the source is reference 1. If the letters "BB" appear, the source is reference 4. The letters "W,F" mean that reference 2 was used but the relative transition probability has been adjusted by Fiori. Reference 3 was used as a check since it is the source of many of the entries of reference 1.

In column 3 the notation KA1,2 means the entry is the weighted sum of the KA1 and KA2 in the ratio 2 to 1. For low atomic number the entries are not self consistent since the data is from different sources. If the column begins with the capital letter S then the entry is a satellite line due to doubly ionized atoms. The relative transition values for these entries are only valid for electron excited specimens, and are, at best, estimates.

Th following are Siegbahn to shell-transition notation conversions:

```
KA   = KA1+KA2+KA3
KA1,2 = (2*KA1+KA2)/3
KA1  = K-L3
KA2  = K-L2
KA3  = K-L1
KB   = SUM(KBn)
KBX  = Metal
KB1  = K-M3
KB1' = KB1+KB3+KB5
KB2  = (K-N3)+(K-N2)
KB2' = K-N3
KB2'' = K-N2
KB3  = K-M2
KB4  = K-N4)+(K-N5)
KB5  = K-M4)+(K-M5)
KB5' = K-M5
KB5'' = K-M4
Kd1  = K-O3
Kd2  = K-O2
LA   = LA1+LA2
LA1  = L3-M5
LA2  = L3-M4
LB1  = L2-M4
LB10 = L1-M4
LB15 = L3-N4
```

```
LB17 = L2-M3
LB2  = L3-N5
LB3  = L1-M3
LB4  = L1-M2
LB5  = L3-O4)+(L3-O5)
LB6  = L3-N1
LB7  = L3-O1
LB9  = L1-M5
LG1  = L2-N4
LG11 = L1-N5
LG2  = L1-N2
LG3  = L1-N3
LG4  = L1-O3
LG4' = L1-O2
LG6  = L2-O4
LG8  = L2-O1
Ll   = L3-M1
Ln   = L2-M1
Ls   = L3-M3
Lt   = L3-M2
Lu   = L3-N6)+(L3-N7)
Lv   = L2-N6
MA1  = M5-N7
MA2  = M5-N6
MB   = M4-N6
MG   = M3-N5
MG2  = M3-N4
MZ1  = M5-N3
MZ2  = M4-N2
Md   = M2-N4
Me   = M3-O5
```

Emission Table

This menu lists a table of emission energies (or angstroms) for all elements and their major analytical x-ray lines. An option is provided for printing the data as angstroms or electron volts.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries using the CalcZAF program.

Edge Table

This menu lists a table of absorption edge energies (or angstroms) for all elements and their major analytical x-ray lines. An option is provided for printing the data as angstroms or electron volts.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries using the CalcZAF program.

Fluorescent Yield Table

This menu lists a table of fluorescent yield fractions for all elements and their major analytical x-ray lines.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries using the CalcZAF program.

MAC Table

This menu lists a table of MACs (mass absorption coefficients) for the specified element and it's major analytical x-ray lines. Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries using the CalcZAF program. The program will automatically load the values based on the selected default MAC table. See the Analytical | ZAF Selection section for a description of the MAC table choices.

Analytical

Empirical MACs

This menu allows the user to selectively load empirical MACs (mass absorption coefficients) from a supplied ASCII file. These MACs are loaded from the EMPMAC.DAT file in the Probe for Windows directory.

See the documentation or help section under Probe for Windows Menu Details for further information on using this feature.

The selections made here will be reflected in the calculation of ZAF correction factors and k-ratios in the Standard for Windows program. This may be used to determine the effect of various mass absorption coefficients on the correction factors for the standard compositions.

ZAF Selections

This menu allows the user to modify or display the ZAF or Phi-Rho-Z quantitative matrix correction options for all standards. The selections made here will be reflected in the calculation of ZAF correction factors and k-ratios in the Standard for Windows program.

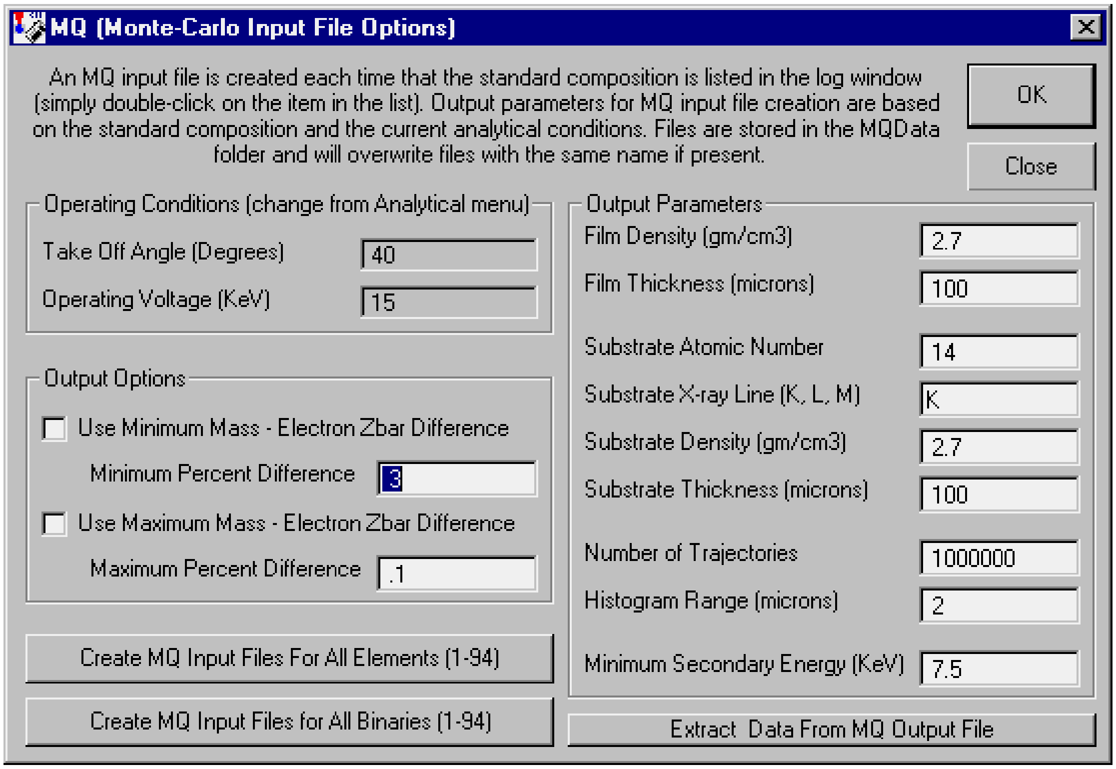
Please refer to the section on ZAF Selections for under the Probe for Windows Menu details for further information on selecting these options.

Operating Conditions

This menu allows the user to modify or display the accelerating voltage, takeoff angle or beam current for calculation purposes. No changes to the microprobe operation are performed. These parameters are not saved to the Standard database and are only intended for evaluating their effect on the various correction factors calculated by Standard for Windows. Of course, only the kilovolts and takeoff parameters have any effect on the calculations.

MQ (Monte-Carlo) Options

This menu displays a dialog that is used in conjunction with the NIST (National Institute of Standards and Technology) monte-carlo electron-solid interaction program (MQ.EXE). This software allows modeling of backscatter and generated and emitted x-ray intensities from bulk (infinitely thick) and thin film compositions and was written by Robert Myklebust and Dale Newbury at NIST. The letter "Q" in the "MQ" files names indicates the version of the NIST monte-carlo software. This version is current as of Sept. 1999.



MQ (Monte-Carlo Input File Options)

An MQ input file is created each time that the standard composition is listed in the log window (simply double-click on the item in the list). Output parameters for MQ input file creation are based on the standard composition and the current analytical conditions. Files are stored in the MQData folder and will overwrite files with the same name if present.

Operating Conditions (change from Analytical menu)

Take Off Angle (Degrees): 40
Operating Voltage (KeV): 15

Output Options

☐ Use Minimum Mass - Electron Zbar Difference
Minimum Percent Difference: 3
☐ Use Maximum Mass - Electron Zbar Difference
Maximum Percent Difference: .1

Output Parameters

Film Density (gm/cm3): 2.7
Film Thickness (microns): 100
Substrate Atomic Number: 14
Substrate X-ray Line (K, L, M): K
Substrate Density (gm/cm3): 2.7
Substrate Thickness (microns): 100
Number of Trajectories: 1000000
Histogram Range (microns): 2
Minimum Secondary Energy (KeV): 7.5

The options (and underlying routines) in the Probe for Windows MQ Options dialog are designed to be used only with the "Q" version of the NIST software. Changes in the input or output format of previous or subsequent versions of the NIST monte-carlo software could affect the ability of the program to create properly formatted input files or parse output files. The MQ program and source code is provided with the Probe for Windows installation, with permission of the authors, to ensure compatibility.

Creating MQ Input Files From Standard Database Compositions

Whenever the Standard program lists data for a standard (whether the user double-clicks in the standard list or uses the List All or Selected Standards), the program will automatically output a properly formatted input file for use with the MQ software.

The compositions, default parameters and conditions in the input file created by Standard may be modified by editing the MQ Options dialog fields before the input files are created. All MQ input files created by his dialog are written to a sub-directory called MQData.

To use the input file simply issue the following command from the Windows command prompt:

```
mq < filename.inp
```

where "filename.inp" is the name of the input file created by Standard. The redirection symbol "<" causes MQ to read input from the ASCII file rather than from the keyboard. Because MQ is a console type FORTRAN program, be sure that input files do not contain spaces or other invalid characters.

Using the MCARLO.BAT Batch File

For best results a batch file can also be created that automatically deletes the output file, copies the input file, runs MQ and copies the output file back to a sub directory. An example batch file (included in the installation) can be seen here:

```
REM  This Batch file (MCARLO.BAT) MUST be executed from a data sub-directory!!!
REM  Usage: CALL ..\MCARLO "inputfile"          (without extension)
REM  (use double quotes if spaces exist in filename)
```

```
copy ..\cdonly.dat ..\cdonly.bat > nul
cd >> ..\cdonly.bat
copy %1.inp ..\mcarlo.inp
cd..
del mcarlo.dat
mq < mcarlo.inp
call cdonly
copy ..\mcarlo.dat %1.dat
del ..\cdonly.bat
```

Note that the MCARLO.BAT batch file shown here must reside in the directory containing the MQ executable, but must be run or called from a sub-directory of that directory. This sub-directory should be the directory containing MQ input files and will therefore generally contain the MQ input files previously created by the Standard program.

The batch file shown above uses a small file called CDONLY.DAT. This file is an ASCII file that contains only the characters "cd ", that is the letters "cd" and a space. This is used to create a small batch file that contains the return path to the sub-directory and is executed once MQ has finished running.

The MCARLO.BAT batch file shown above must therefore be called from the command line as seen here:

```
..\MCARLO "inputfile"
```

Where the "..\" indicates the parent directory. The MCARLO.BAT batch file will automatically process each input file (*.INP) and save the output to an output file with that same name but a different extension (*.DAT).

Multiple Batch Processing
Or the MCARLO.BAT file may be called from another batch file (from the MQ input file sub-directory) as seen here for automating multiple MQ calculations:

```
CALL ..\MCARLO "inputfile1"
CALL ..\MCARLO "inputfile2"
CALL ..\MCARLO "inputfile3"
CALL ..\MCARLO "inputfile4"
CALL ..\MCARLO "inputfile5"
```

A multiple call batch file is automatically created for each type of input file to facilitate extended runs of many compositions. Calls to pure element input files (see next section) for each KeV condition are contained in a batch files called 15-ELEMENT.BAT (in this case for 15 KeV input files). Calls to the complete set of binary compositions (see next section) are contained in a batch file called 15-BINARY.BAT (again for 15 KeV input files). Calls to input files created for compositions in the standard database are contained in a batch file called 15-STANDARD.BAT. All these batch files will be written by default to the MQ-DATA sub-directory.

Creating MQ Input Files For Pure Elements or Binary Compositions

Two buttons on the lower left are used to create a set of MQ input files for each pure element in the periodic table and/or each binary in the periodic table. Once a set of pure element output files have been run through MQ (about 6 hours each for a 1,000,000 trajectory calculation), the "Extract Data From MQ Output File" button may be used to create a spreadsheet containing generated k-ratios and backscatter yields for output files from compound standard compositions.

Parsing MQ Output Files

The "Extract Data From MQ Output File" button can be used to parse one or more MQ output files into a tab delimited data file that can then be used for further processing. First the program will ask the user to select the folder containing the output files previously calculated for all the pure elements in the periodic table. Although the program will appear to ask for a specific input file, simply select any pure element output file from the folder that contains the elemental output files.

Next it will prompt the user to select an output file from a compound composition. The program will then parse the compound composition output file and then automatically parse each pure element output file for each element in the compound to calculate elemental k-ratios and backscatter coefficients. The results for this and all subsequent compound output files selected (for this session), are automatically appended to a tab-delimited data file, which can then be easily imported into any plotting or spreadsheet program for further processing.

The data that is currently extracted from the MQ output files is not necessarily complete. If it is desired to automatically extract additional information from the output files, further modifications to the parsing routines can be made. Please contact Advanced Microbeam to discuss your specific needs.

Output

Log Window Font

This menu allows the user to modify the current log window font type, size and appearance using the font Common dialog.

Note that the default font and size of the log window may be permanently defined in the PROBEWIN.INI file.

Debug Mode

This menu toggles the program in and out of Debug mode. In Debug mode, the program generates an abundance of additional output to the log window for the purposes of debugging the program or viewing the analytical calculations in greater detail.

Extended Format

This menu toggles the output option for printout to the log window of element data. If unchecked, the program automatically wraps element data output to 8 elements per line. If checked, the program does not wrap element data to the log window or disk file (if Save To Disk option is enabled).

Save To Disk Log

This menu toggles the output option to mirror all log window output to a user specified text file. This may be useful for importing text or data from the log window to another non-Windows program. In addition, this option may also be used to capture large amounts of output.

This disk log may be viewed using the Output | View Disk Log menu and therefore printed out as a hardcopy if desired.

View Disk Log

This menu will cause the current disk log (if already opened in this session) to be closed and viewed using the specified file viewer in the PROBEWIN.INI file. The default file viewer is Windows NotePad, however another file viewer such as TextPad or Word for Windows might be substituted in the PROBEWIN.INI file "FileViewer" keyword.

Output Electron and X-ray Ranges

This menu, when checked, will output calculated electron and x-ray ranges based on a range of densities based on the composition of the standard.

Calculate Alternative Z-bars

This menu, when checked, will output an assortment of alternative Z-bars and various fractional models based on the composition of the standard.

Calculate Continuum Absorption

This menu, when checked, will output information on continuum absorption calculations for a variety of x-rays (0.01 sin theta above the characteristic line) for the standard composition.

Calculate Charge Balance

This menu, when checked, will perform a simple calculation of charge balance, based on the current cations and oxide ratios and the composition of the standard.

Help

About Standard

This menu display the copyright notice, acknowledgments and contact information for users of Probe for Windows.

Help On Standard

This menu opens this help file for hypertext help file viewing starting at the main table of contents.

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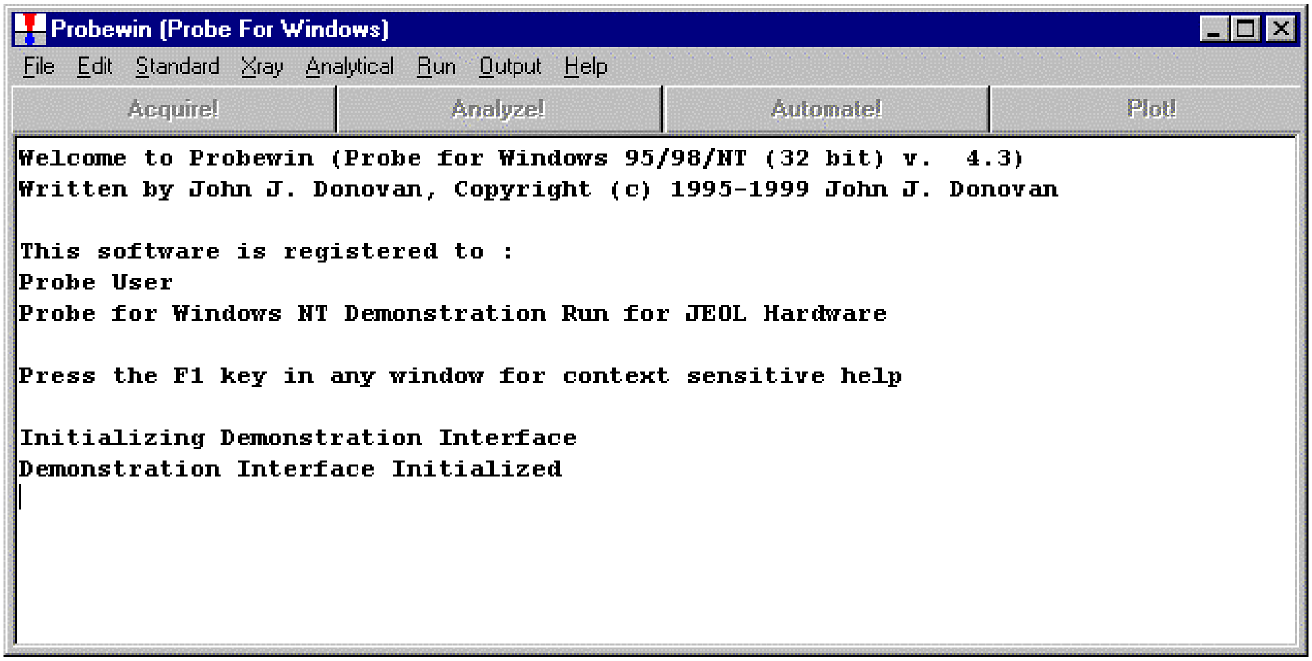
Probe for Windows

Overview

The main program for data acquisition and analysis is PROBEWIN. The menu for this program is found in the Probe for Windows menu group under the Start button. Click the menu to start Probe for Windows NT.



```
Welcome to Probewin (Probe for Windows 95/98/NT (32 bit) v.  4.3)
Written by John J. Donovan, Copyright (c) 1995-1999 John J. Donovan

This software is registered to :
Probe User
Probe for Windows NT Demonstration Run for JEOL Hardware

Press the F1 key in any window for context sensitive help

Initializing Demonstration Interface
Demonstration Interface Initialized
```

When Probe for Windows NT is started it will ask first whether to attempt to interface to the microprobe hardware. If data acquisition or automation is desired, then click Yes for confirmation of the spectrometer, stage and crystal positions. If only re-processing previously acquired x-ray intensities, click No.

If the users clicks Yes, the program will attempt to communicate with the microprobe hardware based on the interface type defined in the PROBEWIN.INI file. Note that if the specified hardware is not actually present, this could cause the program to "hang". Be sure to click No if only processing microprobe data off-line.

To open an existing Probe for Windows database file, click File | Open. To open a new Probe for Windows database file, click File | New. To browse available Probe for Windows data files, click the Find File menu item.

Log Window

The main window of Probe for Windows is a scrollable text window which will record all acquisition and analysis results. This log window is a fully cut and paste enabled text window which can be used to transfer data or analysis results to any other Windows application such as a word processor or spreadsheet.

Simply click and drag the mouse to select text and use the <ctrl> c, <ctrl> x or <ctrl> v key combinations to copy, cut or paste text to or from the Windows Clipboard. Or simply use the Edit menu for these and other Clipboard functions.

The size of the log window buffer can be specified in the PROBEWIN.INI file and is limited only by the amount of memory available. If the Save Log to Disk File option is selected from the Output menu, then all log window output is mirrored to a text file, including any text manually entered by the user using the keyboard. The log window font and font size is specified in the PROBEWIN.INI file and may be changed during a run from the Output menu. To view a copy of the log file during a run, select the View Disk Log option from the Output menu.

Menus

The Probe for Windows log window contains a main menu for easy access to many program functions. Most of these functions are file dependent and are only enabled if a Probe for Windows database file is opened.

Menu items always available are the Standard | Standard Database and X-Ray menus. The Standard Database menu item launches the Standard for Windows application as a separate process, while the X-Ray Database menu item opens the NIST x-ray database as a modal window in Probe for Windows. The other X-Ray menus list various x-ray data to the log window.

Buttons

The Probe for Windows log window also contains four buttons for easy access to modeless windows that may be open at the same time. A modeless window is defined as a window that may remain open while allowing the focus to shift to another window. Probe for Windows has four main modeless windows plus a number of smaller windows such as Move, Locate and Position Database modeless windows.

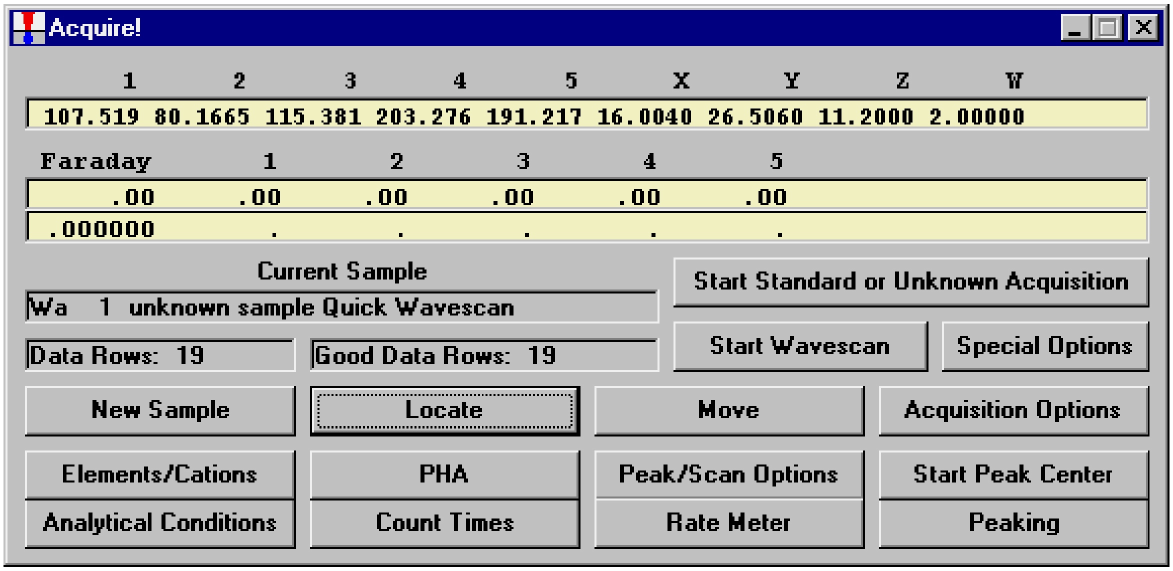
To activate an already open window, simply click the window. If you can't see one of the four main modeless windows because it is hidden by another window, simply click the corresponding button on the Probe for Windows log window form, and it will be brought forward. The four buttons will in turn activate the Acquire!, Analyze!, Automate! or Plot! windows. To close a modeless window, simply double click the upper left corner of the window, however these windows will retain there size and position if left open or at least minimized for the duration of the run.

To conserve screen space, it might be wise to minimize windows that are not actively being used at a particular time. To do this, simply click the minimize button in the upper right corner of the window.

Probe Window Details

Acquire!

The Acquire! window is used to manually create new standard, unknown or wavescan samples, and to manually acquire x-ray intensity data for them. Other buttons from this window allow the operator to set up all acquisition parameters for the current or subsequent sample (the program will not allow acquisition parameters to be modified on samples that already contain data).



```
      1       2       3       4       5       X       Y       Z       W
 107.519 80.1665 115.381 203.276 191.217 16.0040 26.5060 11.2000 2.00000

 Faraday         1         2         3         4         5
      .00       .00       .00       .00       .00       .00
 .000000         .         .         .         .         .

                Current Sample
Wa    1  unknown sample Quick Wavescan

Data Rows:  19              Good Data Rows:  19
```

When the Acquire! window is first opened in a new run, the program will prompt the user that the nominal beam current may be acquired. The nominal beam current is used to provide a scaling factor for the beam drift correction (see below under beam drift correction). When the instrument conditions are properly configured (kilovolts and beam current), click Yes to start the nominal beam current acquisition. To skip the acquisition of the nominal beam current until the next time the Acquire window is activated, click No, or to not acquire the nominal beam current simply click Cancel. In this last instance, the program will use load the nominal beam current as zero for the demo and AM interfaces or load the default beam current from the PROBEWIN.INI file for all other interfaces. To disable the beam drift correction or to manually change the nominal beam current, click the Count Times button and enter the new value in cps for the demo or AM interfaces or in nA for all other interfaces.

Manual standard and unknown sample data can be acquired on a line by line basis, while manual wavescan samples will automatically acquire a complete set of wavescan data for each element in the wavescan sample. If more than one element is assigned to a given spectrometer, then the program, will automatically acquire the additional elements as soon as the previous element wavescan is completed.

At the top of the Acquire! window the current x, y, z stage coordinates, stage rotation (W-motor) index (for JEOL 733 only), and the spectrometer/scanner positions are displayed as label fields. During data acquisition, real time counts, respective count times, the elements being acquired and the faraday beam counts can be observed as well.

In addition, the current sample name and the number of data points appended to it are displayed just above the New Sample button.

The current sample is, by definition, the last sample created by either the user or the automated procedures. This current sample is the only sample to which data can be acquired and appended to.

Note that wavescan samples can only have data acquired and appended to them once. Be sure that the current sample is the sample to which data should be appended to. Otherwise be sure to create a new sample using the New Sample button.

New Sample

Click the New Sample button to start a new sample for the currently open run. This will open the New Sample dialog box.

The new sample (standard, unknown or wavescan) is created based on the **last unknown sample** and the current real-time acquisition parameters specified by the user. Exceptions: if a wavescan sample already exists, the new wavescan is based on the last wavescan, or if no unknown samples exist, the new sample is based on the last standard sample, or if no unknown or standard samples exist, then the new sample is based on the last wavescan sample. The main reason for this is because if the last sample is a standard that was acquired using the "Quick" standard automation option, then all the unknown analyzed elements may not be present.

This means that unless a previously saved sample setup is selected from the Load Sample Setup button, all sample parameters for the new sample are based on the **last unknown sample**. Any additional modifications to the real-time parameters, such as kilovolts, beam current, beam size, count times, PHA, peak positions, etc. are applied after the **last unknown sample** setup has been loaded.

It is important to keep in mind that because all assigned sample parameters such as standard assignments, interference assignments, calculation options, specified elements, etc. are loaded from the last unknown sample, if any changes to the sample assignments are *not* made to the last unknown sample, then these sample assignments will *not* be carried forward to the next new sample that is created.

To change the analytical setup, it is required to either change the analytical setup of the last unknown sample (if it contains no data) or create a new unknown sample and modify it. Note that count time can be changed at any time, even for samples that already contain data.

The New Sample dialog allows the user to create a new standard, unknown or wavescan sample. A new sample name and a sample description can be entered. The lower-most portion of the new sample dialog box lists which standards are currently in the run. In order for a standard sample to be created, it must first be added to the run. This is done by first clicking the Standard | Add Standards To Run menu item from the Probe for Windows log window menus.

Once the sample type is selected and the name is entered, the user can simply click OK and the new sample will be created. If this is the first sample for a new run, the program will create a sample with no analyzed elements. The user may add analyzed elements using the Elements/Cation button described below.

Load Element Setup

To add or remove elements from the sample, or to save or load element setups to or from the element setup database, click the Load Element Setup button.

This dialog provides a method to save and load element setups that have been calibrated for peak positions and PHA parameters for use in creating new analytical setups. If the element setup is saved from a standard sample that has had data acquired for it, the intensity data is automatically saved to the element setup database for evaluation purposes. Note that element setups can be saved anytime, even when not interfaced to the microprobe hardware, from the Analyze! window using the Elements/Cations and Load Element Setup buttons.

Probe for Windows NT also includes the complete wavescan and peaking parameter data for each element to be saved and recalled from the setup database. However, note that element setup data imported from the 16 bit version of Probe for Windows will not contain any wavescan or peaking parameter data and therefore those fields will be blank, although when loaded into a new sample, those blank fields will be replaced with appropriate defaults.

The element setup database can be browsed using the data cursor in the upper right of the window, or all occurrences of a specific element can be browsed by entering the element symbol in the Search Element field. To return the browser to display all elements, simply delete the element symbol from the Search Element field.

The browser displays element setups in the order that they were saved to the setup database. To go to the most recent setup, click the extreme right end of the data cursor. The number of element setups currently in the database is shown also.

Although element setups can be deleted, it may be desirable to keep all element setups for possible evaluation of position or intensity trends for microprobe performance records.

Load Sample Setup

To save or load sample setups to or from the current run, click the Load Sample Setup button. This button allows the user to load sample setups previously saved **within the current run** for later use in acquisition of new samples.

For example, it might be useful to create several sample setups within a run, if it is known that several different phases will be analyzed, each of which are significantly different in their elemental compositions. In other words, it is possible to create one sample setup containing the elements Fe, Ni and Cr as the analyzed elements and another sample setup containing the elements Al, Mg, and Cu as the analyzed elements, all within the same run.

These setups can be referenced for use with the automation actions for tremendous flexibility during the automated acquisition of different phases within a single run by using the Setups button in the Automate! window during the digitization of position samples.

However, if the sample pointed to by the sample setup contains any data, the element setup cannot be changed later on. For this reason it might be best to only reference samples that do not contain any data lines.

Note that the sample setup is actually a pointer to the row number of the selected sample. Any changes in the selected sample parameters will be automatically included when the sample setup is selected later on. This row number may be viewed for all samples in the current run from the Run | List Sample Names menu.

Sample setups may be loaded (or saved for the current sample), if it does not contain any data, by clicking the Elements/Cations button from the Acquire! window and then clicking the Load Sample Setup button. A previously created sample can be saved as a sample setup by using the Elements/Cations button from the Analyze! window.

When saving a sample setups always enter a generic name to indicate the purpose of the sample setup. It may be best to name the sample setup something descriptive, such as, "olivine setup" or "volatile calibration setup" so that the correct sample setup may be easily remembered.

Load File Setup

To load the entire sample setup from another run file, including the standard list and MAN assignments, click the Load File Setup button. With this dialog, it is possible to load an entire sample setup from another run for immediate use or modification.

Because this dialog provides a listing of the sample setup from each filename clicked, it is an easy matter to browse through all available runs to find a suitable sample setup to load.

Note that loading a file setup will overwrite all current run parameters for the Run, Sample and Element database tables. Only the Nominal Beam and Volatile Element correction sample assignments are ignored from the file setup.

After, the file setup (based on the last unknown from the specified file) has been loaded, the program will ask whether to also load the standard intensities. Select Yes, to load the standard intensity data beginning with the last standard and going backwards to the beginning. The standard intensity data will be time stamped with the current date and time. Note that if a standard intensity has already been loaded, the program will not load that particular standard intensity.

Load Multiple Setups

This button is used to load multiple sample setups based on sample setups that have been previously saved sample setups within the current run. All considerations for Samples Setups apply for Multiple Setups (see Sample Setups above). To create sample setups for digitizing position samples, use the "Elements/Cations" button in the Analyze! Window.

When more than one sample setup is specified by the user, the program will automatically combine the specified sample setups into a single sample.

This is useful in several situations, for example, when performing major and trace element acquisition it may be desired to acquire the major elements at one set of column conditions (kilovolts, beam current and/or beam size) and the trace elements using a different column condition (kilovolts, beam current, etc.).

In this case, each element will be evaluated and if different column conditions are required, all elements with similar column condition will be acquired before the program attempts acquiring elements with a different column condition. This is analogous to using the "Combine Multiple Setups" check box in the Automate! window.

The way the program actually runs "combined" column condition sample as follows: first the program looks at the first spectrometer used and finds the first element on that spectrometer. What the first element is, of course depends on the acquisition order option used. It might be channel order, ascending or descending angstroms or user defined order. Whatever conditions that "first" element uses, that is the first condition that is run.

After all elements with that condition have been run, then the program looks for the next element (not already acquired) on the first used spectrometer and runs that condition and so on. So keep this is mind when creating "combined" column condition setups.

Elements/Cations

Clicking the Elements/Cations button opens the analyzed and specified elements dialog box. The analyzed and specified dialog box lists the currently selected elements for analysis. If this is the first sample of a new run then no analyzed elements are present. To edit or add elements, one of the following procedures may be performed :

> **1. Click the Load Element Setup button to load previously saved element setups from the element setup database. Element setups in samples created before the current sample may be saved as element setups from the Elements/Cations button in the Analyze! window.**

2. Click the Load Sample Setup button to load a previously saved sample setups in the currently open run. Samples created before the current sample may be saved as a sample setup from the Elements/Cations button in the Analyze! window.

3. Click the grid row of the element of interest or an empty row to add new elements. This action activates the element properties dialog box. One then selects an element by either typing the element symbol or by clicking the drop down list of all elements and scrolling to and highlighting the desired element.

Default settings for the analyzed x-ray line, cations, and oxygen proportions are loaded automatically. These can be edited via the drop down menu associated with each based on the user's preferences. Leave the x-ray line field blank to indicate an unanalyzed element (specified, by difference or stoichiometry). The defaults for these fields can be modified in the ELEMENTS.DAT file in the Probe for Windows directory. Note that Probe for Windows will retain any modifications of these defaults for the duration of the run.

Special note: if the "UseMultiplePeakCalibrationOffset" parameter is set in the PROBEWIN.INI file and the fit coefficients have been properly calibrated using the Peak/Scan Calibrate Parameters dialog in program StartWin, the program will load "corrected" spectrometer peak positions in the Elements/Cations dialog when the user selects an element and x-ray line. See the Calibrate Peak Center help in the StartWin section for more details.

The choice of background measurement either off-peak or MAN can be specified here. Off-peak correction types include linear, average, high only, low only, and exponential. MAN backgrounds are based on a calibration curve fitted to appropriate standards that do not contain the element of interest (Donovan and Tingle, 1996). The MAN calibration is assigned from the Analytical | MAN Fits menu after the standards to be used in the MAN fit have been acquired.

Note that because the MAN flags may be specified differently for standards and unknowns, the MAN flags specified here will be saved for only the current sample type. This means that if the current sample type is an unknown, then the MAN flags defined in the Element/Cation Properties dialog will only be applied to unknown samples. To define the MAN flags for both the standards and unknowns, select them from the Acquisition Options window by clicking the Acquisition Options button in the Acquire! window.

Off-Peak Backgrounds

The off-peak background correction measures the background on the sample of interest (i.e., your standard or unknown) with the spectrometer adjusted to a position on each side of the analytical peak.

Probe for Windows can individually off-peak correct each point analysis for standard and unknown samples. If the element concentration is very small, it may be necessary to use the off-peak corrected sample method. The program is very flexible in that it allows you to use all MAN or all off-peak background corrections or any combination of the two methods on each element of an acquisition.

Note that since the off-peak positions are saved on a sample by sample basis, one must always start a new standard or unknown sample after changing the off-peak positions or adjusting them graphically using the mouse from a wavelength scan from the Plot! window. Probe for Windows uses a linear slope calculation as the default off-peak background correction. Because of this, unsymmetrically offset background position measurements can be easily handled. One can even position both of the off-peak measurements on the same side of a peak and extrapolate the background correction.

In addition, the user can also specify that the program calculate the off-peak counts using the average of the off-peak counts, or only the high or only the low off-peak counts. Finally, the user can specify an exponential slope calculation for curved backgrounds. If one changes the off-peak background type for an element, it may be necessary to change it for both the unknown samples, as well the standards, depending on the analytical situation.

Off Peak Background Correction Types

Probe for Windows allows the user to select the off-peak type for the background correction. The default slope calculation is the most accurate, except in cases where the user has moved both off-peak positions to the same side of the analytical peak and adjusted the off-peaks excessively close together resulting in a large extrapolation. The off-peak correction types are as follows :

1. = linear slope interpolation or extrapolation
2. = average of both high and low off-peak counts
3. = use only the high off-peak counts
4. = use only the low off-peak counts
5. = use exponential off-peak calculation (based on exponent)
6. = slope-high (based on high off-peak and slope coefficient)
7. = slope-low (based on low off-peak and slope coefficient)
8. = polynomial (based on high and low off-peak and three coefficients)

These off-peak background correction types may be assigned on a sample by sample basis either on or off-line. Note that the actual values of the polynomial fit coefficients are dependent on the absolute magnitude of the count intensities. For this reason it is important to avoid changing the value of the nominal beam current once any polynomial fit coefficients have been assigned to samples in the run. The calculations for linear slope and exponential calculation are shown below :

Linear Slope Calculation

$$I_{off} = I_{low} + (I_{high} - I_{low}) \bullet \frac{(P_{on} - P_{low})}{(P_{high} - P_{low})}$$

where : I_{off} = the off-peak count correction
I_{high} = the x-ray counts for the high off-peak position
I_{low} = the x-ray counts for the low off-peak position
P_{high} = the high off-peak position
P_{low} = the low off-peak position
P_{on} = the on peak position

Exponential Slope Calculation

$$I_{off} = \frac{ce^{-aP}}{P^N}$$

where : I_{off} = the off-peak background intensity
P = the spectrometer position
c = a fit parameter
a = a fit parameter
e = the exponential
N = a user specified exponent

See the Model Background button the Graph dialog. This button is accessible when a wavescan sample is displayed from the Plot! Window.

Same Side Off-Peaks

Beware of placing same-side off-peaks too close together. A large extrapolation can cause a very large error in the off-peak background correction if the background standard deviation is large. The program will print a warning if it feels that the same-side off-peak extrapolation is excessive. In cases where the same side off-peak extrapolation is excessive, the user may want to use the average of the off-peak counts. Of course, averaging the off-peak counts is really only applicable in situations where the background slope is zero.

MAN Backgrounds

MAN (mean atomic number) background corrections can be utilized when the P/B is sufficiently high or when the use of fixed spectrometers are required. The user can specify the standards used to calibrate the background fit for the polynomial MAN iteration of an analyzed sample. The MAN background correction method works because the primary cause of background (aside from trace contamination and peak over-lap interferences) is the average atomic number of the sample. Therefore a function can be fit to appropriate standard data and applied to the analysis of unknown samples. Since the atomic number depends on the composition and that is unknown to begin with, the MAN background is not applied until the analytical calculation iteration. Remember :

- MAN backgrounds can be used whenever the ratio of x-ray peak to background is high (>10:1), and this is usually the case with WDS microanalysis

- MAN backgrounds must be used for any fixed spectrometers

- Trace element concentrations require the use of off-peak measurements

Spectrometer Number and Analyzing Crystal

The spectrometer used to analyze the element should be specified based on the crystal arrangement of the microprobe. Enter a number or use the drop down menu option. This automatically loads a default crystal type, on-peak and off-peak positions that may be modified.

Interferences

The High and Low Off-Peak Interference buttons can be used when both the potential interfering and interfered elements are already present in the sample setup to determine the approximate magnitude of the off-peak interferences. This calculation is based on a gaussian peak shape and various crystal defaults and is intended as a guideline only for off-peak position entry.

The program assumes 100% of the interfering element and 0.1% of the interfered element in each binary pair calculation. For example, if the percent interference is calculated as 10%, then that indicates that instead of measuring 0.1 wt. % of the element, it is likely that a concentration of 10% greater than 0.1 wt. % or 0.11 wt. % will be measured. Note that since this is only a nominal calculation, it should only be used as an indicator of possible problematic analytical situations.

PHA

The PHA parameter settings (baseline, window, inte/diff (integral or differential) mode, gain, bias, and deadtime) can be specified here. Defaults are loaded from the configuration files and can be modified at any time.

Note that if the UseEmpiricalPHADefaults flag is set in the [Software] section of the PROBEWIN.INI file, then the PHA defaults loaded, are actually calculated from coefficients stored in the EMPPHA.DAT file instead being loaded from the SCALERS.DAT file.

The PHA parameters entered here will be used to configure the microprobe hardware, if supported by the interface type during the acquisition. The PHA settings for the current sample (if it contains no data) can also be modified from the PHA button in the Acquire! window.

The current PHA settings from the microprobe can also be read (if supported by the hardware and specified in the PROBEWIN.INI file) by clicking the Get PHA button.

Detector Parameters

If the proper microprobe interface support is present for detector control, the detector slit size, slit position and mode (usually FPC or SPC) may also be specified for automatic control during the analysis. If the microprobe interface does not support automated detector parameter control, these fields can be used simply to document the parameters. See the documentation on the DETECTORS.DAT file in the Configuration Files section above.

Integrated Intensity Options
If the element is acquired on a tunable spectrometer, the program offers an option acquire the integrated intensity over the peak instead of simply the peak intensity. If this option is checked, the program will start scanning at the low or high off-peak limit using the Initial Step Size specified and scan to the other off-peak limit.

The actual step size is modified based on the acquired intensity so that as the intensity increases, the step size decreases to the Minimum Step Size specified. This improves statistics where it is needed the most. The default step size is 1/10th of the off-peak range and the default minimum step size is 1/100th of the off-peak range.

The program accumulates the intensities over the off-peak range and this is stored as the on-peak data. The off-peak intensities are also measured normally and the integrated background is calculated based on the background type selected and the off-peak intensities. All off-peak options may be used with the integrated intensity option. The reported integrated intensities are in units of counts per second times spectrometer units.

If an element is acquired using the integrated intensity option, the standard intensities for that element must also be acquired using the integrated option.

Specified Area Peak Factors (fixed composition APF)
As an alternative to the "calculated" Area Peak Factors, Probe for Windows offers the use of "fixed" composition Area Peak Factors. These are single values that are applied to the element intensity on an emitter basis (as opposed to calculated based on the actual absorber concentrations in the matrix).

These factors may be useful in certain situations where the compositional range is limited and binary Area Peak Factors are not available for some or all elements in the matrix (e.g., Na, K or Ca in an oxygen matrix). This option may be turned off or on in the Analysis Option dialog under the Analytical menu.

Load Element Setup

This button opens the element setup database dialog. Here the user can add element setups to the new sample or save element setups to the setup database. Generally, one usually saves standard sample element setups for elements that are assigned to that standard to the setup database. Later, one can retrieve these calibrated setups for use in creating new sample setups for acquisition in another run.

The element setup database can be browsed using the data cursor in the upper right of the window, or all occurrences of a specific element can be browsed by entering the element symbol in the Search Element field. To return the browser to display all elements, simply delete the element symbol from the Search Element field.

The browser displays element setups in the order that they were saved to the setup database. To go to the most recent setup, click the extreme right end of the data cursor. The number of element setups currently in the database is shown also.

Load Sample Setup

To save or load sample setups to or from the current run, click the Load Sample Setup button. This button allows the user to load sample setups previously saved **within the current run** for later use in acquisition of new samples.

For example, it might be useful to create several sample setups within a run, if it is known that several different phases will be analyzed, each of which are significantly different in their elemental compositions. In other words, it is possible to create one sample setup containing the elements Fe, Ni and Cr as the analyzed elements and another sample setup containing the elements Al, Mg, and Cu as the analyzed elements, all within the same run.

These setups can be referenced for use with the automation actions for tremendous flexibility during the automated acquisition of different phases within a single run by using the Setups button in the Automate! window during the digitization of position samples.

However, if the sample pointed to by the sample setup contains any data, the element setup cannot be changed later on. For this reason it might be best to only reference samples that do not contain any data lines.

Note that the sample setup is actually a pointer to the row number of the selected sample. Any changes in the selected sample parameters will be automatically included when the sample setup is selected later on. This row number may be viewed for all samples in the current run from the Run | List Sample Names menu.

Sample setups may be loaded (or saved for the current sample), if it does not contain any data, by clicking the Elements/Cations button from the Acquire! window and then clicking the Load Sample Setup button. A previously created sample can be saved as a sample setup by using the Elements/Cations button from the Analyze! window.

When saving a sample setups always enter a generic name to indicate the purpose of the sample setup. It may be best to name the sample setup something descriptive, such as, "olivine setup" or "volatile calibration setup" so that the correct sample setup may be easily remembered.

Analytical Conditions

Clicking the Analytical Conditions button opens the analytical conditions dialog box. The operator can then edit the kilovolts, beam current, and beam size settings for the selected sample. In addition, if the hardware interface is supported, the user may specify a column condition string to indicate the desired analytical conditions of the instrument.

If the hardware support for these parameters is not present, then all parameters (other than kilovolts) are simply available for documentation purposes only. Note also that if the instrument has the ability to check the current analytical conditions, then the program will make sure that the analytical condition have not changed since they were last set. If the analytical conditions have changed by an amount greater than that specified by the "OperatingVoltageTolerance" and "BeamCurrentTolerance" in the PROBEWIN.INI file [hardware] section, then the program will force the analytical or column condition to be reloaded.

Analytical Conditions versus Column Conditions

In the first method, (using specified values for kilovolts, beam current and beam size) the user directly specified the analytical conditions. In the second method, a specified string is used to indicate a column condition that is stored internally in the instrument. In this latter case, because the actual value of the operating voltage (kilovolts) in the column condition string is ambiguous, the user should be sure that the single value fields are also correctly specified based on the column condition string specified.

For example, the SX50 supports the use of internally stored column conditions that are indicated by a four character string. Therefore, if the column condition option is selected, and the column condition string is specified as "hv15" (indicating a column condition at 15 keV), then the user should also be sure that the kilovolts field is correctly specified as 15 keV. Failure to properly specify the kilovolts field when using column condition strings will cause the ZAF or Phi-Rho-Z matrix corrections to be inaccurate. The beamcurrent field is also used in some cases to check for beam drift.

For accurate documentation purposes it is also preferred that the beam current and beam size fields are also properly specified when using column condition strings. If, as in the above example, the "hv15" column condition string also includes a beam current of 10 nA and a beam size of 10 um, then those values should also be specified in the single values fields even though are not actually used in the acquisition procedure. This maintains the accuracy of the documented sample conditions.

Note that because samples at different operating voltages can be acquired in a single run, standards at the corresponding voltages, must also be acquired for quantitative calculations.

Faraday Stage

A new feature is support for a specimen based faraday cup that is specified by stage coordinates. See the [Faraday] section of the PROBEWIN.INI file documentation for information on how to specify the default location of the stage faraday cup. The Faraday Stage button will bring up a dialog box that allows the user to update the faraday cup stage location in the program and make a manual measurement. During standard or unknown acquisitions, the software will move to the faraday stage location make the measurement and then return the original stage location for the next analysis.

Locate

Clicking the Locate button opens the locate sample positions dialog box.

All acquired sample positions can be accessed from this dialog. Double-clicking on a sample entry displays the stage coordinates for each line acquired. To move to a sample position, highlight a position sample and click Go. This will move the stage to the first undeleted stage coordinate of the sample selected. If a specific line coordinate is selected from the position grid, then clicking Go will move to that specific coordinate (deleted or not).

By clicking the Copy Position Coordinates to Clipboard, the sample position coordinates shown, will be copied to the Windows clipboard for pasting into another application if desired.

PHA

Clicking the PHA (pulse height analysis) button opens the PHA parameters dialog box.

Here all currently analyzed elements are listed detailing their respective baseline, window, inte/diff (integral or differential) mode, gain, bias, deadtime factors. To edit any of these values, simply click the element row to be edited and the element PHA parameters window will appear.

If the proper microprobe interface support is present for detector control, the detector slit size, slit position and mode (usually FPC or SPC) may also be specified for automatic control during the analysis. See the documentation on the DETECTORS.DAT file in the Configuration Files section above.

The PHA parameters dialog provides buttons to both set (write) PHA parameters to the microprobe and get (read) the PHA parameters from the microprobe (if the necessary hardware interface is available).

Note that if a gain and bias hardware interface is available, and the bias (detector high voltage) values on a given spectrometer are different for multiple elements, the acquisition will pause for two seconds before proceeding with the acquisition. For this reason the program will warn the user if the bias values are different between elements on a single spectrometer.

In addition a graphical PHA distribution may be acquired, based on the PHA count time and intervals specified.

The PHA distribution should be adjusted by first adjusting the bias until the region of flat response is found. This "plateau" will provide the most stable count intensities since any small changes in the bias will not cause a change in the detector count rate. Next the gain of the PHA amplifier is adjusted to place the PHA distribution is the approximate center of the amplifier range. If the argon escape peak is close to the signal peak, the gain should be adjusted so that the escape peak is either completely included or completely excluded from the PHA distribution.

Count Times

Clicking the Count Times button opens the count times dialog box. This dialog also allows the user to modify the beam current integration time and the nominal beam current.

This dialog displays the current analyzed elements and the counting time for on-peak, off-peak, wavescan, peaking and quick scans. Each element may utilize a different count time for each on and off-peak measurement. The wavescan count time is applied to wavescan samples, the peak scan count time is applied to the peaking pre-scan acquired before a spectrometer peak center is run (if that option was selected) and the quickscan count time is applied to wavelength quick scans.

The count times for a range of elements may be specified by first clicking the Update Selected Element check box and then clicking and dragging a range of elements in the element list. Any changes to the count times dialog will be applied to all selected element in this case.

The Max Counts is used to specify a statistics based count time. By default, Max Counts is set to 100,000,000 (100 million total counts), so that it will normally not effect the acquisition. However, it may be desirable to specify a specific maximum count that is to be acquired for each element.

For example, the count time and max counts could be configured so that each element will count for 30 seconds or 10000 total counts, whichever comes first. The advantage of this method is that on samples with high count rate elements, the actual time required to acquire data is shortened, while low count rate elements are counted for a full 30 seconds. In this way also, the statistical significance of all element count rates with the same Max Counts is more comparable.

The Unknown Count Time Factor is also displayed, which is actually used for trace element analysis in two different ways. The first method is as a factor applied to the default count time for acquiring unknown sample elements relative to the count time used for the standards. This is especially useful in trace element analysis when the standard only requires a 10 second count time but the unknown requires say, 100 seconds for the desired detection limit. By simply specifying an Unknown Count Time factor of 10, this can be achieved.

The other way in which the Unknown Count Time Factor can be used is when the Use Alternating On and Off Peak Acquisition flag is checked in the Acquisition Options window. In this case the total acquisition time is calculated as above but in addition, the program will automatically divide up the acquisition of on and off-peak count times into alternating intervals based on the Unknown Count Time Factor for each element.

For example, if the Use Alternating On and Off Peak Acquisition option is checked and the on peak count time for an element is 10 seconds and the off-peak count time is 5 seconds and the Unknown Count Time Factor is set to 3 then the program will first acquire 10 seconds on peak followed by 5 seconds on each off-peak, in three separate repetitions. The counts are automatically totaled at the completion of all repetitions. This option is especially useful in situations where the sample measurement could be affected by extended acquisition times due to beam damage, carbon contamination or surface charging.

Note that in both instances, the Unknown Count Time Factor entered, must be an integer value.

Beam Current Measurement and Beam Drift

The integration time for beam current measurements and the nominal beam (in cps for AM hardware or nA for other hardware) can also be specified here.

The nominal beam current is used as a scaling factor for the beam drift correction. Any non-zero value will suffice, however to have the count rates reported close to the actual count rates, a nominal beam current should be used that is similar to the actual beam current used during the acquisition.

The beam count time is in seconds for AM hardware and number of averaged beam current integrations for other hardware.

For aperture beam current measurement probes (ARL SEMQ) the beam is always measured (counted) simultaneously with the x-ray counting and therefore is normalized based on the nominal beam counting time. Because the measured beam current is therefore the portion of the beam that is intercepted by the beam current aperture support housing, one must take special precautions to avoid changing the geometry of the electron optics during the quantitative run. This usually means making no changes to the condenser lens, gun bias, or filament alignment. However, since the objective lens is usually below the beam current aperture, the electron beam focus can normally be adjusted with no detrimental effects to the quantitative calibration. The advantage of the aperture beam current method is that since the beam current and x-ray counts are simultaneously acquired, any non-linear change in the beam current is automatically corrected for by the beam drift correction.

On the other hand, microprobes which use a faraday cup (JEOL and Cameca) for the beam current measurement, require an extremely stable and regulated beam current since the beam current measurement occurs after the x-ray count acquisition. The benefit of the faraday cup beam current measurement method is that the electron optics geometry (i.e., sample current) can be changed during the quantitative run without affecting the beam drift calibration.

The beam drift correction calculation is shown here :

$$I_C = I_U \frac{B_N \frac{T_U}{T_N}}{B_U}$$

Where :

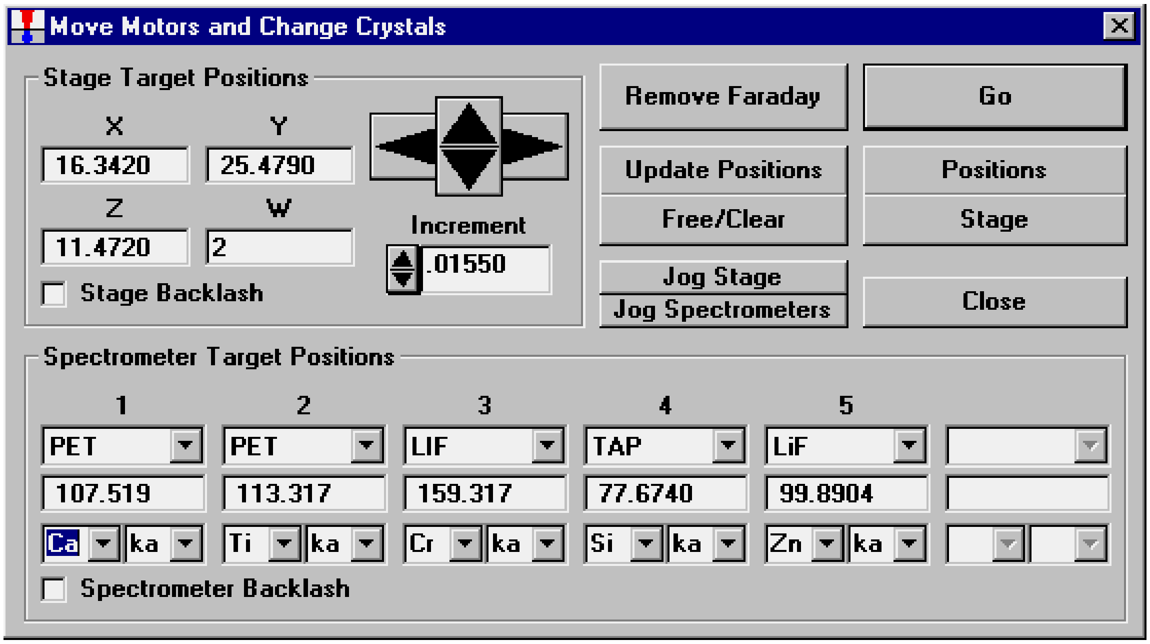
I_C is the beam drift corrected unknown intensity
I_U is the uncorrected unknown intensity
B_N is the nominal beam current
T_U is the count time for the unknown intensity
T_N is the count time for the nominal beam current measurement
B_U is the beam current for the unknown intensity

If you do not want a beam drift correction (it is recommended to always use a beam drift correction for the most quantitative work), one can either enter a nominal beam current of zero or disable the beam drift correction from the Analytical | Analysis Options menu.

Move

Clicking the Move button opens the move motors and change crystals dialog box. If crystal flipping is not supported by the interface hardware, then the crystal lists are disabled.

To manually flip a crystal when automated crystal flipping is not available, first drive to the crystal flip position (using the JOYWIN application if necessary) and manually flip the crystal. Then, after moving to a valid spectrometer position, exit the program and restart the application and confirm the new crystal positions in the Confirm dialog.



Stage Target Positions

X: 16.3420
Y: 25.4790
Z: 11.4720
W: 2
Increment: .01550
☐ Stage Backlash

Spectrometer Target Positions

1	2	3	4	5	
PET	PET	LIF	TAP	LiF	
107.519	113.317	159.317	77.6740	99.8904	
Ca ka	Ti ka	Cr ka	Si ka	Zn ka	

☐ Spectrometer Backlash

The operator can adjust the stage coordinates as well as the spectrometer motors positions and crystal types.

The large four-way directional arrows can be used to increment the stage by the micron increment number listed in the cell below. The value of the micron increment can also be adjusted up or down using the spin controls or edited by hand.

One can set stage, spectrometer and crystal targets by entering the positions manually or by selecting an element and x-ray symbol from the spectrometer target position lists. *Special note: if the "UseMultiplePeakCalibrationOffset" parameter is set in the PROBEWIN.INI file and the fit coefficients have been properly calibrated using the Peak/Scan Calibrate Parameters dialog in program StartWin, the program will load "corrected" spectrometer peak positions in the Move dialog when the user selects an element and x-ray line. See the Calibrate Peak Center help in the StartWin section for more details.*

This modeless window can be accessed using the Move button on either the Acquire! or Automate! windows. Note that during automated acquisitions this window will be automatically unloaded to prevent hardware access conflicts.

The Stage and Spectrometer backlash check boxes in the Move window are applied only to stage or spectrometer motion from the Move window. Exception: if the stage backlash option is unchecked in the Move window, no stage backlash will be applied during the automation actions. See the Acquisition Options dialog (accessed from the Acquire! window) to enable or disable stage and/or spectrometer backlash for sample acquisitions.

Faraday In/Out or Beam Blanked/Unblanked

The faraday cup on faraday equipped microprobes can be inserted or removed and the beam blank state can be toggled by the user using the Faraday In/Out or Beam Blanked/Unblanked button.

The insert and remove faraday delay times can be specified in the PROBEWIN.INI file. This may be useful to give the hardware sufficient time to complete the operation before proceeding.

An additional Faraday/Beam Blank button is provided in the Stage BitMap window used for graphical stage moving (Stage button).

Update Positions

This button when clicked, updates the Move dialog for the current stage and spectrometer positions for those interfaces that do not constantly poll the microprobe hardware. Normally, the Advanced MicroBeam hardware accesses the hardware constantly to update the Move dialog, while other slower interfaces do not.

Jog Stage

Clicking this button causes the stage motors to jog by an amount specified in the PROBEWIN.INI file backlash factors. Use this button to remove any stage backlash that may be present when manually digitizing stage positions. The jog is based on the current stage positions for all stage motors.

Note that the stage backlash flag must be set (checked), in order to use the sample specific stage backlash flags as specified in the Acquisition Options windows for automated samples.

Jog Spectrometers

Clicking this button causes all spectrometers to jog by an amount specified in the PROBEWIN.INI file backlash factors. Use this button to remove any spectrometer backlash that may be present when manually adjusting the spectrometer positions. The jog is based on the current spectrometer positions for all spectrometer motors.

This flag is duplicated in the Acquisition Options window, which can be accessed by clicking the Acquisition Options button in the Acquire! window.

Free/Clear

This button stops and deactivates all current stage and spectrometer motor motion. It is equivalent to clicking the Cancel button on the Automation Status window except that a motor de-energize is also performed on the Advanced MicroBeam hardware.

If a serial interface (SESAME or TRACOR) is specified, the program will instead clear the serial buffers to re-set any line errors.

Positions

Clicking this button opens the Position Database dialog box. This list of position samples is similar to that seen in the Automate! position list.

All currently digitized position sample stage coordinates, (standards, unknowns, and/or wavescans) for the position database can be displayed. Double-clicking on any entry will display the stage coordinates for that position sample.

One can move to any selected position sample by clicking the Go button on this window. The program will move to the first undeleted stage position for the selected sample. If a specific position coordinate is selected in the lower coordinate display grid, the program will move to that specific position.

The Re-Load will refresh the position sample list in case it was modified by another dialog.

The currently selected position sample coordinates can be updated using the Update button based on the current stage position.

The Edit button will allow the user to manually edit certain fields for the position samples including the position sample name fiducial set and setup number.

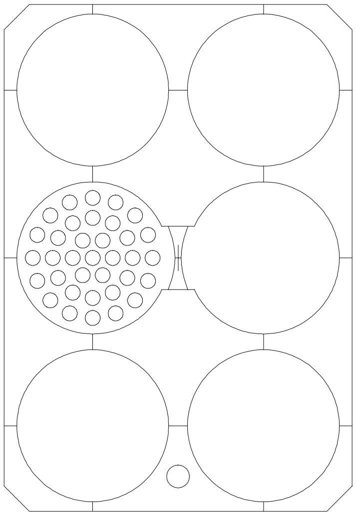
Stage

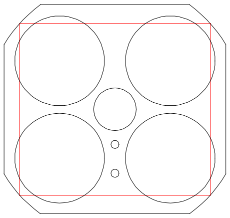
The Stage button will open a Stage Bit Map window to allow the user to move to a stage position by simply clicking on a specific area on an image of the stage. More than one image file (up to MAXBITMAP%) can be specified in the

PROBEWIN.INI file under the "Standards" section. To select another stage bit map simply select the file from the list box. The current stage position is indicated as a red circle.

The frame containing the stage bit map list box, cursor position and Faraday/Beam Blank button can be dragged to a different area of the window if the present position covers an area of interest.

To re-size the Stage Bit Map window simply drag any corner of the window to the desired size and shape. To enable the Stage button or add additional Stage Bit Maps files to the program, see the [Standard] section in the PROBEWIN.INI file. Some sample Stage Bit Map files are shown here for the Cameca SX50 and ARL SEMQ respectively:





Close

Clicking this button will close the Move dialog box and return to the Acquire! window.

Go

Clicking the Go button activates the motors to send all stage and/or spectrometers to their listed target positions. If the crystal positions were changed, the crystals will also be flipped before the spectrometers are driven to their final positions (if supported by the hardware interface).

Since the Go button is the default button in this window, typing <enter> from the keyboard will also cause the motors (and crystals) to move.

Peak/Scan Options

Clicking the Peak/Scan Options button displays the spectrometer peak center and wavescan parameters for each element.

All currently analyzed (tunable spectrometer) elements are displayed, along with their values for the current sample, for on and off-peaks, wavescan limits, peakscan limits, peaking parameters.

Click an element row to edit any peak and scan parameter. Clicking any element row opens the peak and scan properties dialog box. Spectrometer positions may be entered in either spectrometer units or angstrom units and either in absolute position units or relative offsets.

The Move To On Peak button will actually move the current spectrometer to the on-peak position of the current element. The Update On Peak button will update the on-peak (and all other offset positions) to the current position of the spectrometer. This is useful when manually tuning a spectrometer position. The Move To On Peak button will also automatically set the PHA parameters if supported by the hardware.

Note that there is an option to have the stage position "bumped" during the wavescan acquisition. Because the spectrometers are usually operated in an asynchronous mode, the stage is incremented for the specified microns at each specified interval (0-60 seconds). For example, if the user has set the stage increment value to 5 microns and the stage increment interval to 10 seconds, then the program will increment the stage by 5 microns every 10 seconds, during the wavescan acquisition

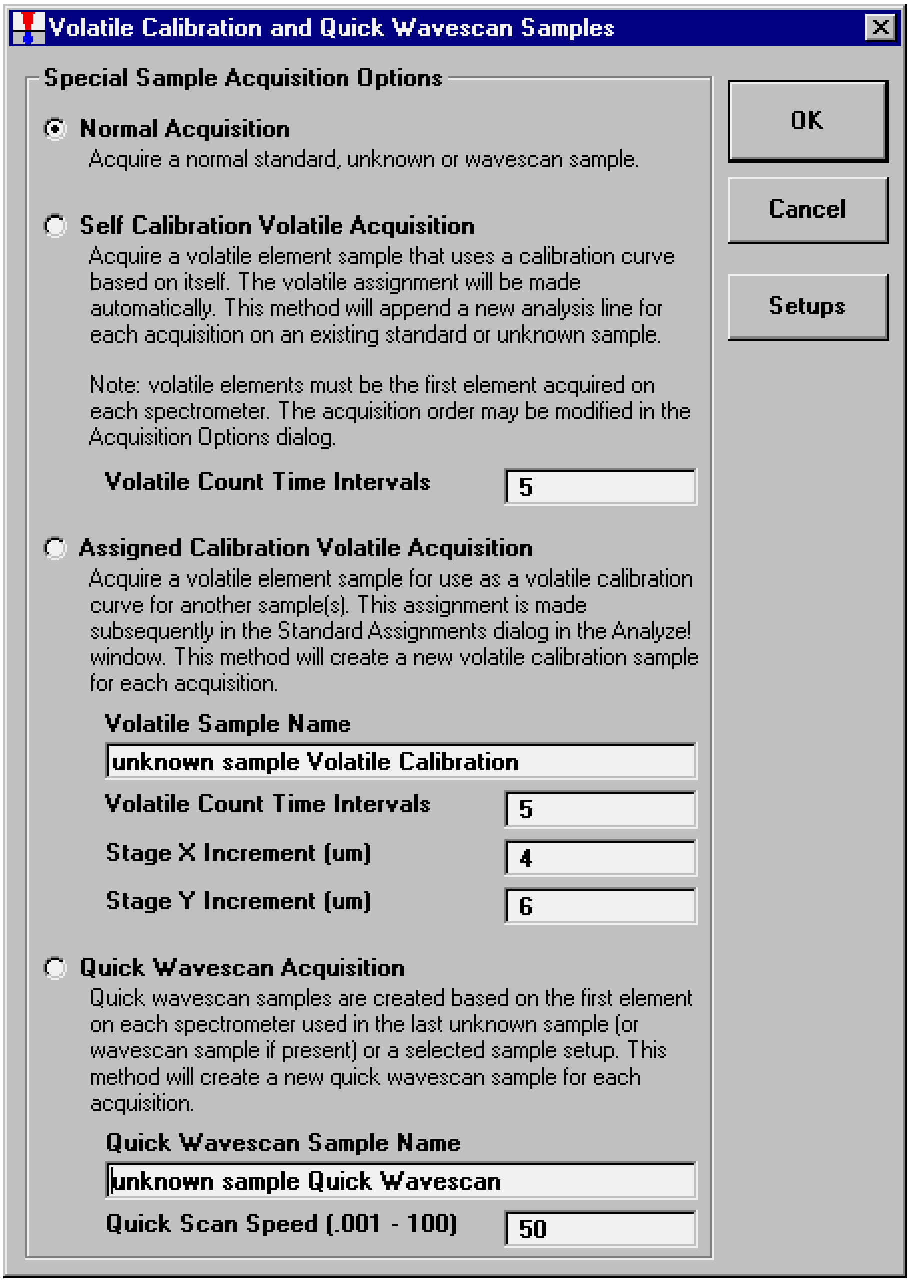
Rate Meter

Clicking the rate meter button opens the Rate Meter Display window. The faraday cup will be removed to show a real time graphical display of each spectrometer and its associated counts per second numeric value for the currently selected spectrometer motor position.

The rate meter dialog is disabled automatically during a sample acquisition to prevent hardware conflicts.

Special Options

Clicking the Special Options button opens the Volatile Calibration and Quick Wavescan Samples dialog. There are two different methods that may be used in Probe for Windows for the correction of volatile elements in unknown samples and another method for the acquisition of "quick" wavescan samples.



Special Sample Acquisition Options

● **Normal Acquisition**

Acquire a normal standard, unknown or wavescan sample.

○ **Self Calibration Volatile Acquisition**

Acquire a volatile element sample that uses a calibration curve based on itself. The volatile assignment will be made automatically. This method will append a new analysis line for each acquisition on an existing standard or unknown sample.

Note: volatile elements must be the first element acquired on each spectrometer. The acquisition order may be modified in the Acquisition Options dialog.

Volatile Count Time Intervals: 5

○ **Assigned Calibration Volatile Acquisition**

Acquire a volatile element sample for use as a volatile calibration curve for another sample(s). This assignment is made subsequently in the Standard Assignments dialog in the Analyze! window. This method will create a new volatile calibration sample for each acquisition.

Volatile Sample Name: unknown sample Volatile Calibration

Volatile Count Time Intervals: 5

Stage X Increment (um): 4

Stage Y Increment (um): 6

○ **Quick Wavescan Acquisition**

Quick wavescan samples are created based on the first element on each spectrometer used in the last unknown sample (or wavescan sample if present) or a selected sample setup. This method will create a new quick wavescan sample for each acquisition.

Quick Wavescan Sample Name: unknown sample Quick Wavescan

Quick Scan Speed (.001 - 100): 50

Volatile element calibration corrections can only be applied to elements that are the first element acquired on a given spectrometer. The acquisition order of the elements may be modified in the Acquisition Options dialog.

"Quick" wavescan samples are spectrometer wavescans that automatically cover the entire range of each spectrometer available in the run.

Self Calibration Volatile Acquisition

In the self calibration method, the software acquires the on-peak data for the volatile element during the normal sample acquisition process for unknown samples.

Click the Self Calibration Volatile Acquisition option so that the next sample acquisition will be acquired using the self calibration method. The number of count time intervals used is specified in the Volatile Count Time Intervals text field. Up to 50 intervals may be used in a volatile acquisition.

For example, if the user enters 5 in the Volatile Count Time Intervals text field, and the default on-peak count time is 10 seconds, then the software will automatically count five 2 seconds intervals for the on-peak elements that are first in spectrometer order. Off-peak acquisitions are not affected.

To view the volatile calibration data, click the Standard Assignments button in the Analyze! window and select the element to view. Click the Display Volatile Fit to view the data for all lines in the sample. Note that the volatile calibration caption will indicate a self calibration correction. That is, the unknown number used for the calibration is itself.

Assigned Calibration Volatile Acquisition

Another method for volatile element calibration corrections is the assigned calibration method. In this method an unknown sample is automatically acquired one element at a time by clicking the Assigned Calibration Volatile Acquisition option to allow the volatile element effect to be calibrated precisely on a single sample. This sample will be started automatically using the sample name in the Volatile Sample Name field the next time a sample acquisition is started.

The count time is adjusted based on the Volatile Count Time Interval parameter. For example, if the user enters 5 in the Volatile Count Time Intervals text field, and the default on-peak count time is 10 seconds, then the software will automatically count five 2 seconds intervals for the on-peak elements that are first in spectrometer order. Off-peak acquisitions are not affected.

The stage is automatically moved in X and Y for each element to ensure that a fresh sample spot is utilized for the volatile calibration.

The volatile element calibration can be assigned to any element in a sample or range of samples provided that it was acquired as the first element on that spectrometer. See the Analyze! window Standard Assignments button for the volatile element calibration display and assignment.

Setups

Pressing the Setups button opens the sample setup window. Here an available sample setup can be selected and used as the basis for the assigned volatile calibration sample. This feature is intended for assigned calibration volatile corrections when it is desired to acquire a subset of the actual elements to which the volatile correction will be assigned.

Quick Wavescan Acquisition

Clicking the Quick Wavescan Acquisition option will start a quick wavescan sample the next time a wavescan is started.

A sample name and scan speed percentage can be specified. The program will move each spectrometer currently being used to it's extreme limit and continuously scan the spectrometer while acquiring count data at the same time. This is a very useful feature if an EDS detector is not available or WDS resolution is required.

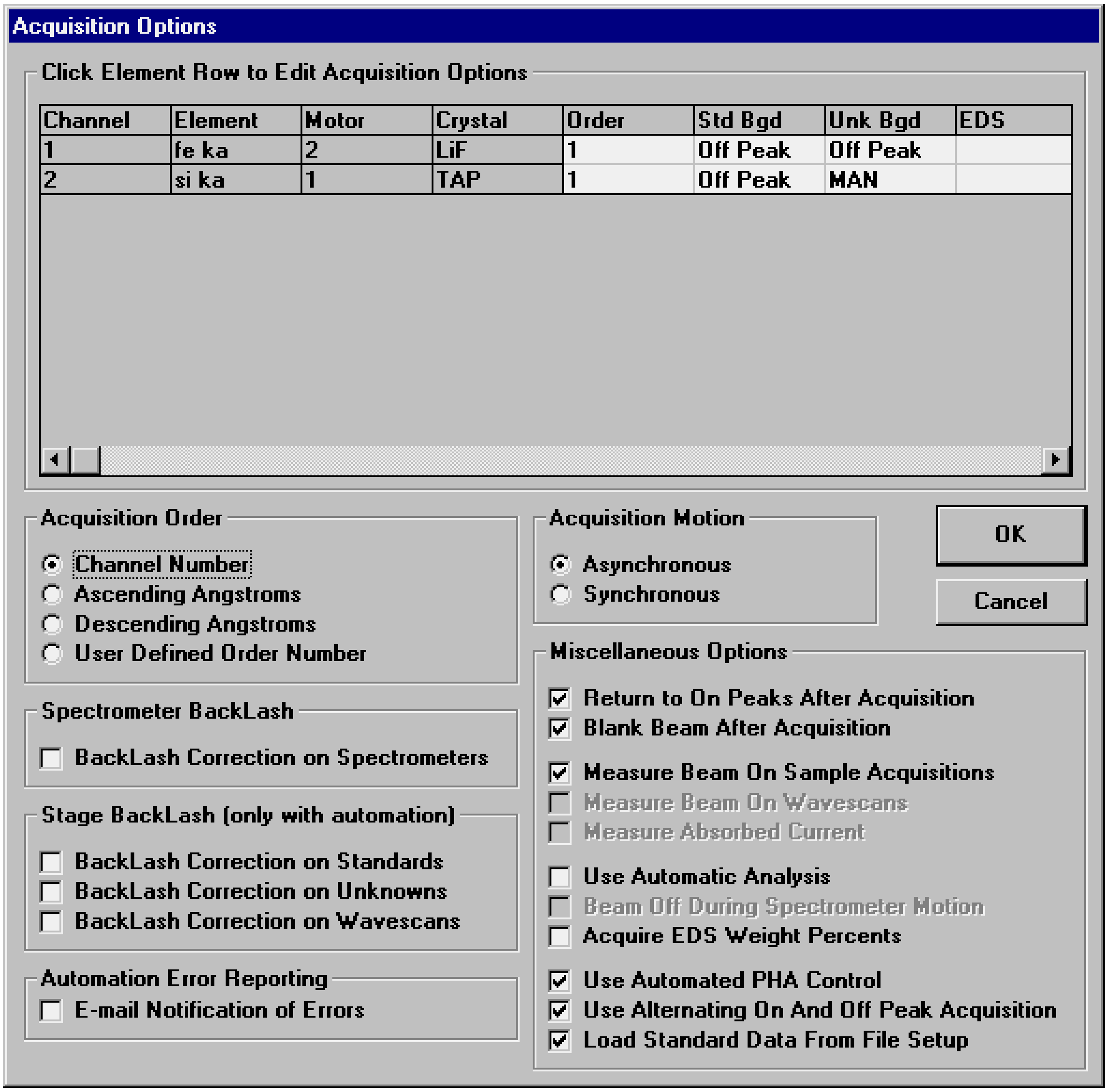
The count time used for the quickscan is specified in the Count Times dialog. Note that the program uses the element configuration for the first acquired element (order=1) on each spectrometer in the sample to create the quickscan sample.

Setups

Pressing the Setups button opens the sample setup window. Here an available sample setup can be selected and used as the basis for the quickscan.

Acquisition Options

Clicking the Acquisition Options opens the Acquisition Options dialog box.



Acquisition Options

Click Element Row to Edit Acquisition Options

Channel	Element	Motor	Crystal	Order	Std Bgd	Unk Bgd	EDS
1	fe ka	2	LiF	1	Off Peak	Off Peak	
2	si ka	1	TAP	1	Off Peak	MAN	

Acquisition Order

- ● Channel Number
- ○ Ascending Angstroms
- ○ Descending Angstroms
- ○ User Defined Order Number

Spectrometer BackLash

- ☐ BackLash Correction on Spectrometers

Stage BackLash (only with automation)

- ☐ BackLash Correction on Standards
- ☐ BackLash Correction on Unknowns
- ☐ BackLash Correction on Wavescans

Automation Error Reporting

- ☐ E-mail Notification of Errors

Acquisition Motion

- ● Asynchronous
- ○ Synchronous

Miscellaneous Options

- ☑ Return to On Peaks After Acquisition
- ☑ Blank Beam After Acquisition
- ☑ Measure Beam On Sample Acquisitions
- ☐ Measure Beam On Wavescans
- ☐ Measure Absorbed Current
- ☐ Use Automatic Analysis
- ☐ Beam Off During Spectrometer Motion
- ☐ Acquire EDS Weight Percents
- ☑ Use Automated PHA Control
- ☑ Use Alternating On And Off Peak Acquisition
- ☑ Load Standard Data From File Setup

All elements analyzed in the current run are listed along with their order of analysis and background type (for both standard and unknown samples). The acquisition order for the elements on each spectrometer may be specified by channel number, ascending or descending angstroms or user-defined order number.

Stage and spectrometer backlash flags for sample acquisitions can be set. Asynchronous or synchronous spectrometer motion can be specified. Note that the sample specific stage backlash flag seen here are only used when performing automated sample confirmation or acquisition. These sample specific backlash flags will be ignored if the Stage Backlash option is not checked in the Move window before the automation is started.

Note that the program will attempt to determine if a backlash is required based on the last direction of motion for each motor. If the backlash factor is positive and the last motion was from a higher to a lower position and the size of the motion was larger than the backlash size, then a backlash will not be performed on the next occurrence of the backlash adjustment.

Other options include beam blanking after each acquisition, returning to on-peaks after acquisition, and beam current measurement modes. If an absorbed current interface is available, the measurement can be turned on or off here also.

The acquisition options for each element can be edited by clicking the desired element row. The spectrometer order number (if user defined was selected) and background type for both standards and unknowns can be specified. If entering the spectrometer order number as user defined, be sure to set the acquisition order for all elements on that spectrometer before exiting the Acquisition Options dialog.

The Use Automatic PHA Control option only applies to those interfaces that support computer control of the baseline, window, gain, bias, integral/differential and/or deadtime parameters. If any of these parameters are under computer control and this option is checked (the default) then the computer will control them for acquisition and automation purposes. If this option is not checked then these parameters will not be set by the program and must be set manually outside the program.

The Use Alternating On And Off Peak Acquisition flag will cause the program to acquire the on and off peak intensities alternately based on the number of repetitions determined by the Unknown Count Time factor parameters in the Count Times dialog. This option applies only to unknown samples. This flag is ignored for Volatile Assigned Calibration acquisition and also for elements (acquisition order = 1) that are acquired using the Volatile Self Calibration acquisition.

The Load Standard Data From File Setup is used to specify whether standard intensity data from a file setup should automatically be imported during automated acquisition using the File Setup option. When a file setup is imported from the new Sample dialog, the user is offered a choice whether or not to load the standard intensity data.

EDS Acquisition

Probe for Windows supports various EDS interfaces for combined WDS and EDS acquisition. To utilize this feature the Acquire EDS Weight Percents check box must be checked and the elements to be acquired using EDS must be already suitably configured using the specific EDS package and selected from the Acquisition Properties window (click the element row in the Acquisition Options window).

To add an element for EDS acquisition, first add the element to the current sample as a specified (blank x-ray line) from the Elements/Cations window. Then, from the Acquisition Options | Properties window click the EDS Acquisition Option check box. If the EDS element is properly configured, the program will list the Acquisition Option under EDS as Yes.

During the acquisition, the program will initiate an EDS acquisition, using the EDS software interface defined in the PROBEWIN.INI file, and when the EDS weight percent value is returned by the EDS interface, it will be stored in the probe database for use in the analytical calculations.

To include these EDS acquired elements in the analytical calculations, simply go to the Calculation Options window from the Analyze! window and make sure that the EDS Data Options has the Use EDS Element Data option selected.

Start Peak Center

Clicking Start Peak Center starts a manual peak center (and peak pre-scan if selected) on the elements selected from within the Peaking button window. If more than one element on the same spectrometer was selected, then the program will automatically peak center them one at a time.

Select the elements to be peak centered and the peak center method from the Peaking dialog accessed from the Acquire! window.

Peaking

Clicking the peaking button opens the peak center window. All currently analyzed elements are listed. The Peak Center method can be specified. A choice of Interval Halving, Parabolic and ROM based (if supported by the hardware) is available. The default peak center method can be specified from the PROBEWIN.INI file. With all methods the program will perform a peak to background measurement to determine if the peak is statistically significant enough to attempt a peak center.

To options are offered, the first an option move the spectrometers to their on-peak positions if selected in the element list. This option is useful for moving the spectrometers to calibrated peak positions for x-ray mapping purposes.

The other option forces the program acquire a "pre-scan" wavescan just prior to the peak center procedure to allow the user to select an initial peak center start position. This last option is particularly useful in cases where the nominal spectrometer position is badly out of mechanical calibration and the actual peak position is some distance from the theoretical peak position.

The Plot Selected Peak Center button will display graphically, the last peak center attempted for the selected element. The points displayed are numbered in the order acquired. Therefore, for example, if the parabolic peak center was performed, and an optimum peak center was obtained, there will be four points displayed: the three (on, hi, lo) for the parabolic fit and a fourth new on-peak based on the calculated peak center.

> The **Interval Halving** method is the most accurate, but the slowest. It is based on a peak crawl that changes direction and halves the step size whenever the count intensity drops by more than a standard deviation.
>
> The **Parabolic** method peak center uses a moving three point fit on the peak top that is fit to a 2nd order polynomial. The centroid of the parabola is returned as the new peak center. This method is the fastest, if the spectrometer position is already close to the actual on-peak position.
>
> The **ROM Based** method peak center is available if the hardware interface supports a ROM peak center. The actual method used for the ROM Based peak center depends on the microprobe hardware.

The program will automatically select elements in the element list that were not peaked in the previous peak center when the window is re-opened again. Note that the element list is multi-select so that any combination of elements can be peaked. If more than one element on the same spectrometer was selected, then the program will automatically peak center them one at a time. The Plot Selected Peak Center button may be used to display the last peak center count-position data for the selected element.

An option for performing a spectrometer prescan before the peak center and an option for moving to the selected element on-peaks when closing the dialog are possible.

The move to on-peak option is useful for moving a spectrometer (and setting the PHA conditions) to it's on-peak position for imaging purposes. Remember : the elements to peak center (or move to on-peaks) must be highlighted before closing the dialog.

Spectrometer Peak Center Scans

All spectrometer peak center procedures may be prefaced with a spectrometer peak scan to allow the user to manually specify the nominal peak center position. This spectrometer peak scan is performed using the number of peak scan steps as defined in the Peak/Scan Options dialog. The count time for each peak scan point is based on the peak count time (divided by 4) as defined in the Count Times dialog.

Initial Peak-To-Background Measurements

The program always performs a peak-to-background and peak intensity check before the actual peak center procedure. The peak-to-background is calculated by measuring the count rate at the nominal peak position and the current off-peak positions using the peak count time for each position. The measured count data must meet the required minimum peak-to-background and minimum peak count intensity as defined in the Peak/Scan Options dialog, before performing the actual peak center procedure.

If performing the peak center from the Automate! dialog, the program will not proceed with acquisition of sample data if one or more spectrometer fails to complete a peak center procedure for any reason.

Spectrometer Peak Center Procedure

Three peak center procedures are used by Probe for Windows. They are the Interval Halving, the Parabolic Fit and the ROM based peak center procedures.

Interval Halving

Interval halving is a peak center procedure based on a peak crawl starting at the nominal peak position. The step size of the peak crawl is halved and the direction reversed whenever the count rate drops by more than a single standard deviation (square root of the intensity). The interval halving procedure is slow but extremely precise.

The initial peak step interval is always in a positive direction and the peak interval size is based on the Peaking Start Size as defined in the Peak/Scan Options dialog. Note that the default peaking start and stop sizes from the SCALERS.DAT file are modified by the following expression :

$$S_{actualstart} = S_{start} \bullet \sqrt[3]{\frac{2d_{OnPeak}}{2d_{LiF\,200}}}\,(*2.0)\,\sqrt{\frac{P_{LoLim} + ABS\,(P_{HiLim} - P_{LoLim})}{P_{OnPeak}}}$$

Where : $S_{actualstart}$	is the actual calculated step size
S_{start}	is the spectrometer default start or stop size
P_{HiLim}	is the spectrometer high limit
P_{LoLim}	is the spectrometer low limit
P_{OnPeak}	is the spectrometer on-peak position
$2\ d_{OnPeak}$	is the 2d spacing of the analyzing crystal
$2\ d_{LiF\,200}$	is the 2d spacing for LiF (4.0267)

From this expression one can see that the actual start and stop sizes are unchanged at the spectrometer high limit when an LiF crystal is used, and are increased for lower spectrometer positions and larger crystal 2d spacings. The calculated start and stop sizes are also doubled when the crystal 2d spacing is larger than 30 angstroms (LDE crystals).

When the interval step size has been halved to a value less than the peaking stop size, the spectrometer is considered to be peak centered and the current position is saved as the new peak position.

If the number of intervals measured exceeds the "Maximum Peaking Cycles" as defined in the Peak/Scan Option dialog, the program terminates the peak center procedure with an error.

Parabolic Fit

The parabolic peak center procedure is based on a moving three point triplet that attempts to fit an inverted parabola. The three positions measured are the middle, high and low positions. The middle position is initially the nominal peak position and the high and low positions are offset from the middle position based on the value of the Peaking Start Size, which is calculated using the process described in the interval peaking procedure above. Ideally the Peaking Start Size calculation should be attempt to yield an offset which places the high and low positions at a position which produces approximately 1/2 the maximum intensity of the peak, for a more precise parabolic fit.

The peak center procedure then determines if the high and low position intensities are at least one standard deviation less than the middle position intensity. If the three intensities meet this criteria, the program calculates the centroid of a parabola based on the count-position data triplet. The results of the centroid peak fit can be viewed by placing the program into "Debug Mode" under the Output menu.

If the data cannot be fit to a parabola, the middle position is shifted by an amount equal to 1/2 of the Peaking Start Size and the middle, high and low position intensities are measured again.

If the number of count-position data triplets measured exceeds the "Maximum Peaking Cycles" as defined in the Peak/Scan Option dialog, the program terminates the peak center procedure with an error.

ROM Peaking

The ROM peaking procedure is available for those interfaces that support an integrated ROM based peaking procedure. These are the JEOL, Sesame, Tracor, SX100 and SX50 interface types.

The actual peaking procedure utilized by each interface is somewhat different but usually based on a parabolic fit of some variety. The program passes the "Peaking Start Size" to each interface specific ROM Peaking routine where is it modified if necessary.

JEOL 8900 Direct (InterfaceType=2)- The "Peaking Start Size" is not used. Instead a peak parameter of 1 (fine scan) is always used.

Sesame (InterfaceType=3)- The "Peaking Start Size" is not used by the Sesame interface. The function simply sends the "PEAK" command for the specified motor, at the current position.

Tracor (InterfaceType=4)- The Tracor peak center procedure uses a two step peak center (an initial peak center based on the "Peaking Start Size" and a final peak center based on the "Peaking Stop Size"). The total number of steps is hard-coded since the PAC cannot update this system parameter while another spectrometer is being peaked. The number of TRACOR motor steps per peak scan point is calculated from the number of motor steps per l-unit conversion factor (from the MOTORS.DAT file), the "Peaking Start Size", (or) the "Peaking Stop Size" and the number of peak scan steps (hard-coded to 40). The following expression is used to calculate this value, and assumes a TRACOR peak scan width equal to ten times the Peaking Start Size :

$$sp = M * P * 10 / N$$

where	sp	is the Tracor ROM peak center scan motor steps per point
	M	is the motor steps to units conversion factor (from MOTORS.DAT)
	P	is the Peaking Start Size (initial) or Peaking Stop Size (final)
	N	is the number of peak scan steps (hard-coded to 40)

The Tracor step count time is calculated from the Peaking Count Time (divided by four). Note that the PAC Crystal Factor variable must be set to 1 on all spectrometer-crystals for this procedure to perform correctly.

After the initial Tracor peak center has completed, the procedure moves the motor position to the calculated centroid returned from the PAC, then a final Tracor peak center is performed, before the new peak intensity is measured.

SX100 (InterfaceType=5)- The "Peaking Start Size" is modified to produce a number between 0 and 4 by utilizing the following expression :

$$pw = (M_{high} - M_{Low}) * P / 3000000$$

where	pw	is the SX100 peak center width
	M_{high}	is the motor high limit
	M_{low}	is the motor low limit
	P	is the Peaking Start Size

SX50 (InterfaceType=6)- The "Peaking Start Size" is modified to produce a number between 0 and 4 by utilizing the following expression.

$$pw = (M_{high} - M_{Low}) * P / 3000000$$

where	pw	is the SX50 peak center width
	M_{high}	is the motor high limit
	M_{low}	is the motor low limit
	P	is the Peaking Start Size

If the SX50 peak center width is less than or equal to 2, the SX50 driver sends the "PE" command for the specified motor. Otherwise, if the SX50 peak center width is greater or equal to 3, the SX50 driver sends the "PE WIDE" command instead.

JEOL 8900 Rlogin (InterfaceType=7)- The "Peaking Start Size" is not used. Instead a peak parameter of 1 (fine scan) is always used.

Final Peak-To-Background Measurements

After a successful peak center procedure the peak-to-background is re-calculated by simply measuring the on-peak count rate at the new peak position. This result can be compared with the initial peak-to-background to evaluate the peak center procedure performance.

Start Sample Count

Clicking the Start Sample Count button starts a standard or unknown sample acquisition based on the current sample. Data is automatically appended to the current sample which is displayed in the Acquire! window. Be sure that current sample is the correct sample to avoid appending data to the wrong sample.

The program will check the sample parameters before starting an acquisition to make sure that the sample is a valid sample for the configured hardware. For example, if the sample uses Fe ka LiF on spectrometer 2, but the current crystal on spectrometer 2 is PET and the crystal flip interface is not enabled, the program will warn the user that the crystal must be flipped manually before starting the acquisition.

Start Wavescan

If the current sample is designated as a wavescan sample, then clicking this button opens the Wavescan Acquisition window and automatically starts a wavescan sample acquisition.

All elements in the wavescan sample will be scanned, even if more than one element is on a spectrometer. This provides an easy method to scan all elements in a quantitative sample for off-peak interferences.

During the acquisition the wavescan graph may be viewed in greater detail, simply click the wavescan graph to toggle the display size. The wavescan window contains fields to display the mouse cursor position in spectrometer, angstrom and count units. In addition, options for loading the NIST x-ray database window based on the x-ray range of the wavescan graph clicked and a button to save the graph data to an ASCII file are available.

Once the wavescan has been acquired, the user can view the off-peak interferences, by using the Plot! window to display the wavescan sample graphically.

Note that the current sample must be a wavescan sample without any data already acquired. To start a new manual wavescan sample use the New Sample button from the Acquire! window.

Analyze!

Analyze

This button is used to convert previously acquired x-ray count data to elemental, oxide, atomic or formula results. The samples currently selected in the sample list will be analyzed and the analysis output is directed to both the Analyze! window and the log window.

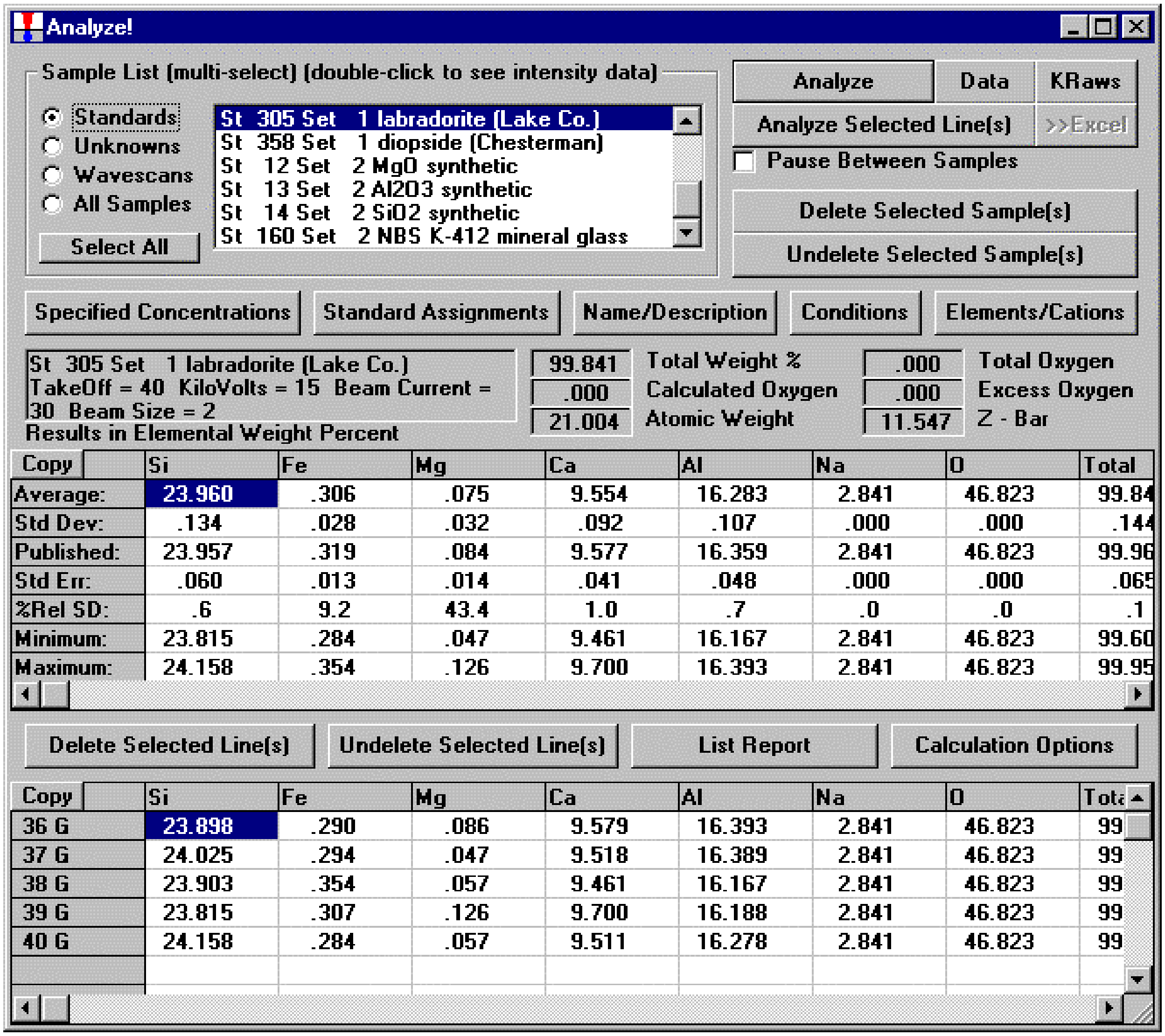
The Analyze! window will display the totals, calculated oxygen (if any) and z-bar and atomic weight along with the last specified results in the following order :

- Elemental Weight Percents
- Oxide Weight Percents
- Atomic Weight Percents
- Formula Atoms

All calculations (including homogeneity statistics and mineral end-members are also output in the above order to the log window as text. To reduce the detail level of the output, see the options in the Analytical | Analysis Options menu. To see a complete output of all intermediate steps of the analysis calculations use the Output | Debug Mode menu to turn on debug printout and re-run the analysis.

If more than one sample is selected for analysis, it may be desirable to check the Pause Between Samples check box. If this is selected, then the program will automatically pause after each analysis until the user clicks the Cancel or Next buttons on the Analysis Status window. This allows the user to view the results of each analysis and delete data points if necessary before proceeding to the next analysis. This is especially useful when analyzing the standards as unknowns after a standardization has been acquired to check for problems in the standard calibration data.

The Copy buttons in the two grids can be used to copy the displayed results into the Windows Clipboard buffer, from which it can be pasted into other applications as tab delimited text.



● Standards
○ Unknowns
○ Wavescans
○ All Samples

St 305 Set 1 labradorite (Lake Co.)
St 358 Set 1 diopside (Chesterman)
St 12 Set 2 MgO synthetic
St 13 Set 2 Al2O3 synthetic
St 14 Set 2 SiO2 synthetic
St 160 Set 2 NBS K-412 mineral glass

☐ Pause Between Samples

St 305 Set 1 labradorite (Lake Co.)
TakeOff = 40 KiloVolts = 15 Beam Current = 30 Beam Size = 2
Results in Elemental Weight Percent

99.841 Total Weight %
.000 Calculated Oxygen
21.004 Atomic Weight
.000 Total Oxygen
.000 Excess Oxygen
11.547 Z - Bar

Copy	Si	Fe	Mg	Ca	Al	Na	O	Total
Average:	23.960	.306	.075	9.554	16.283	2.841	46.823	99.84
Std Dev:	.134	.028	.032	.092	.107	.000	.000	.144
Published:	23.957	.319	.084	9.577	16.359	2.841	46.823	99.96
Std Err:	.060	.013	.014	.041	.048	.000	.000	.065
%Rel SD:	.6	9.2	43.4	1.0	.7	.0	.0	.1
Minimum:	23.815	.284	.047	9.461	16.167	2.841	46.823	99.60
Maximum:	24.158	.354	.126	9.700	16.393	2.841	46.823	99.95

Copy	Si	Fe	Mg	Ca	Al	Na	O	Tota
36 G	23.898	.290	.086	9.579	16.393	2.841	46.823	99
37 G	24.025	.294	.047	9.518	16.389	2.841	46.823	99
38 G	23.903	.354	.057	9.461	16.167	2.841	46.823	99
39 G	23.815	.307	.126	9.700	16.188	2.841	46.823	99
40 G	24.158	.284	.057	9.511	16.278	2.841	46.823	99

Error Messages

Several problems can occur when performing analyses of quantitative samples. If data for a standard is missing, the program will print "No count data for standard __ for __". This means that either that the standard was not acquired at all or that the standard assignments were changed to a channel that was not acquired due to a "quick" standardization. Simply acquire data for the specified element on the specified standard or re-assign the standard to correct the problem.

If the message "No count data for interference standard __ for __" is seen, a similar problem exists. Note that standards used in the interference corrections should not be run as "quick" standards to avoid this problem. If the warning message persists, you may be assigning an interference correction where none exists. Check your wavelength tables to be sure. If you find no interference, then remove the interference correction for the element. Remember, interferences are quite dependent on spectrometer resolution (slit width) and PHA settings (for higher order lines) and so the magnitude or presence of the interference may change from run to run.

Problems With the Analysis

Quantitative analysis is always beset with many difficulties and it is often difficult to pinpoint the cause of "bad" analyses. More often than not, the effect is the sum of more than one problem. However, there are several things that can be checked. First of all start by examining the standards. It is in situations like this that the use of secondary standards can really be helpful in determining what is going on. If the primary and secondary standards do not agree with each other, there is a problem.

First of all, are the standards really "good" standards? That is, have their compositions been determined by a reliable analytical method? In general, major element standard concentrations need to be determined using classical wet chemistry methods. Second, are the concentrations entered into the STANDARD.MDB database correct and without typographical errors? Third, was the right standard actually acquired? It is easy to get lost at 300-400 magnifications when using a standard mount which contains many standards.

Some other things to consider :

1. Is the operating voltage correct specified? Is the correct x-ray line tuned for each element? Check the on-peak position offsets from the Peak/Scan Option dialog and see if they are reasonable. The program will usually type a warning if the actual and calculated peak positions are very different. Be sure that the spectrometer is not tuned on a nearby line of another element if using multi-element standards.

2. Be sure that no bad data points are in the standards samples used for the quantitative calibration. The best way to check for this is to analyze each standard and examine the results to look for points with obviously bad or low totals (epoxy, bad surface polish, bad carbon coat, etc.). If a bad point is found, one can delete it. Remember, one can always undelete data later on. A deleted point is simply not used in calculating the analytical calculations but is still present. A good rule of thumb is to only delete points that have low totals since generally most of the problems mentioned above will result in fewer x-ray counts. Avoid deleting points just to get better agreement between the primary and secondary standards. Points that have high totals should not be arbitrarily deleted. It may be necessary to look for other problems such as points with low totals in the primary standards.

3. Look for interferences on the analyzed elements. One easy way to do this is to use the Interferences button in the Element Parameters window (see the Elements/Cations button in the Acquire! or Analyze! window). One can also perform a wavelength scan and display possible interfering peak markers. If the element causing the interference is present in significant concentrations in your unknowns, and is not being analyzed for, it may be necessary to add the interfering element to the run by creating a new sample with the interfering element as an analyzed element. Be sure that the proper standards are available to use for the interference correction.

4. If none of the above suggestions seem to help, try acquiring the standards again. Probe for Windows uses an automatic standard drift corrections which can make a significant difference in situations where one or more of the standard intensities are drifting. Note that since the program will perform an automatic drift correction not only on the standards, but also the interference standards and the MAN background standards, it might be also be necessary to run additional sets of those standards or MAN standards.

5. In the case of trace or minor elements, also check to see that none of the off-peak positions are interfered with by another peak. This can cause a reduction in the on-peak counts, sometimes enough to result in a negative k-ratio. Always run at least one wavelength scan on a sample, using the same count time as your quantitative analyses, and if a peak is seen interfering with the off-peak marker, use the Low and/or High buttons in the Graph Data window to select a new off-peak position that is not interfered with.

Description of the Analysis Printout

When the analysis is printed out you will see the sample type (st or un), sample number and name printed on the first line. If the sample is a standard, then the sample set is also shown. The microprobe takeoff angle and the operating voltage is shown.

The next line indicates the total number of lines (or data points) that are contained in the sample. One may have 1 to 50 points in each sample. In the same line are the number of points that have status of "G" for good, meaning that this many points are undeleted. A sample with all points deleted will have the number of "G" points equal to 0 and will not be used in any calibrations or recalculations and therefore cannot be quantitatively analyzed, unless using the Analyze Selected Lines button.

Next, the average elemental totals and the total, calculated and excess oxygen is displayed. The calculated oxygen is the amount of oxygen calculated by cation stoichiometry (if selected) and the excess oxygen is the difference between the measured and oxygen calculated if the Display As Oxide option was selected. This excess oxygen is often very useful in determining if the selected cation ratios are correct, especially for iron bearing oxides.

Next is the atomic weight and average Z-bar for the sample. This is followed by the average number of ZAF iterations needed to converge each data point for the sample and the MAN iterations, which are the iterations required to converge the MAN background, interference, APF (Area Peak factor) and volatile element corrections. The Z-bar is defined as the sum of the weight fraction of the atomic numbers of all elements in the sample including specified elements.

Following this are the weight percents and standard deviation of any elements specified for the sample by fixed concentration, difference or stoichiometry.

The specified element type is listed for each specified element. The type "SPEC" means that the element concentration is truly specified. That is, either specified by the user or (for standard samples) loaded from the standard composition database. The other types are "DIFF" for element by difference, "CALC" for element by stoichiometry to stoichiometric oxygen and "RELA" for an element by stoichiometry to another element.

The next two lines indicate the type of background correction used for each element ("MAN" for mean atomic number corrected and "OFF" for off-peak corrected elements) and the average count times for each element in the sample. Remember that the count times for each element can be different for each line in the sample. To see the actual counting time details use the Data button in the Analyze! window with the Debug Mode menu checked.

Note that if fixed spectrometers are used in the run, or a MAN background corrected sample was analyzed, the program will also indicate the magnitude of the absorption correction to the continuum background counts in the line labeled "%ABS" if the element is MAN corrected.

The analyzed element symbols are printed next and listed below are the weight percent calculated for each data point for each element. The elemental weight results are then followed by the average, "AVER" of each element column, the standard deviation, "SDEV" and the standard error, "SERR". The standard deviation is basically the range of the results and the standard error is essentially the precision of the average. Finally the percent relative standard deviation, "%RSD" of the results are printed which is simply the standard deviation divided by the average times 100.

Note that one can perform analyses of samples which are not unknowns, e.g. it is possible to have the program analyze a standard as though it were an unknown. Therefore, if the sample is a standard sample, the program next lists the published, "PUBL" weight percent value for the element as entered in the default standard database. If the element is not found in the standard database it is shown as "n.a." or "not analyzed". The next line lists the percent variance, "%VAR" from the published value for each element compared to the actual measured average for the standard. This can provide a valuable check on the quality of your analyses for secondary standard elements. That is, element channels which are not assigned as the primary calibration for that element. After this, is the line labeled "DIFF" which is a simple difference between the "AVER" and "PUBL" values.

The actual primary standard used for each element is displayed on the next line "STDS" since the standard assignments can be made on a sample by sample basis.

Note that sometimes the average analyzed value of a standard that is assigned, appears to be not exactly the value shown in the "PUBL" line. It seems reasonable that they should always be exactly the same, but remember that because PROBE treats all samples (standards and unknowns) as unknowns when performing an analysis, you may see a small discrepancy if the standard contains other analyzed elements that are also not assigned. This is because Probe for Windows calculates the correction factors for an analysis of a sample, based on the actual analyzed composition of the sample, not the theoretical composition in the STANDARD database. Of course, for the analytical calibration, PROBE for Windows uses the database composition for the calculation of standard k-factors.

Below this are shown the average standard k-factors "STKF" or the standard beta-factors "STBE" if using alpha-factors, the average standard counts "STCT" followed by the normalized unknown k-ratio, "UNKF", the unknown count rate "UNCT", the unknown background counts "UNBG" (either MAN or off-peak measured", the ZAF correction factor, "ZCOR" if using ZAF or Phi-Rho-Z or the "UNBE", unknown beta-factor if using alpha-factors, and the average raw k-ratio, "KRAW", which is the normalized and background corrected unknown counts divided by the standard counts.

The "KRAW" value shown is corrected for all corrections except the matrix correction. These include the deadtime, count time, beam drift, MAN or off-peak background, and the quantitative interference and APF (area peak factor) corrections. The peak to background ratio for each element is shown in the "PKBG" line. A peak to background of close to 1.0 means that no peak was present.

Below this are listed the percent correction to the counts for any interference corrections that were performed on the sample in the line labeled "%INT".

Finally, if the volatile element extrapolation was selected, the program will show the percent change in the x-ray counts due to the volatile element extrapolation in the line labeled "%VOL" and if any area peak factors (APF) were selected, the program will print the sum of the APFs (1.00 indicates no APF correction) for each element affected in the line labeled "APF:".

If the user elected to calculate the detection limits and/or sample statistics, the program will then print those calculations.

Following this are formula and atomic percent calculations if they were selected. All sample calculation options can be assigned to a single sample or a range of samples.

Example of Analysis Printout

```
St  358 Set   1 diopside (Chesterman)
TakeOff = 40  KiloVolts = 15  Beam Current = 30  Beam Size = 2
Number of Lines:  5 Number of 'Good' Lines:  5
Current Date and Time: 12/14/98 2:27:03 PM

Elemental Wt. % Total:   99.916     Average Total Oxygen:      .000
Average Calcu. Oxygen:     .000    Average Excess Oxygen:      .000
Average Atomic Weight:   21.657    Average Atomic Number:    12.230
Average ZAF Iteration:     4.00    Average MAN Iteration:      2.00

Results in Elemental Weight Percents

SPEC:         O
TYPE:      SPEC

AVER:    44.316
SDEV:      .000

ELEM:        Si      Fe      Mg      Ca      Al
BGDS:       LIN     LIN     LIN     LIN    LIN7
TIME:     10.00   10.00   10.00   10.00   10.00

ELEM:        Si      Fe      Mg      Ca      Al   SUM
    41   25.850    .060  11.323  18.372    .000  99.921
    42   25.984    .032  11.086  18.551    .004  99.974
    43   25.853    .039  11.223  18.609    .017 100.057
    44   25.862    .046  11.239  18.369    .061  99.893
    45   25.847    .034  10.974  18.542    .022  99.735

AVER:    25.879    .042  11.169  18.489    .021  99.916
SDEV:      .059    .011    .139    .111    .024
SERR:      .026    .005    .062    .050    .011
%RSD:        .2    26.3     1.2      .6   115.0
```

```
PUBL:   25.925    .047  11.192  18.489    .016  99.985
%VAR:     -.18  -10.09    -.21     .00   30.46
DIFF:    -.046   -.005   -.023    .000    .005
STDS:       14     263      12     358      13

STKF:    .4101   .5004   .4738   .1693   .4353
STCT:   4105.3  5026.8  4758.6  1692.8  4354.5

UNKF:    .2089   .0004   .0770   .1693   .0001
UNCT:   2091.2     3.5   773.7  1692.8     1.1
UNBG:     17.3    30.0    26.3    23.7    18.0

ZCOR:   1.2388  1.1999  1.4499  1.0919  1.4223
KRAW:    .5094   .0007   .1626  1.0000   .0003
PKBG:   122.39    1.12   30.47   72.64    1.07

Results in Oxide Weight Percents

SPEC:        O
TYPE:     SPEC

AVER:     .066
SDEV:     .085

ELEM:     SiO2     FeO     MgO     CaO   Al2O3   SUM
    41  55.303    .077  18.777  25.706    .000  99.921
    42  55.590    .042  18.383  25.957    .008  99.974
    43  55.309    .050  18.611  26.038    .032 100.057
    44  55.329    .059  18.637  25.702    .115  99.893
    45  55.296    .044  18.197  25.944    .042  99.735

AVER:   55.365    .054  18.521  25.869    .039  99.916
SDEV:     .126    .014    .230    .155    .045
SERR:     .056    .006    .103    .069    .020
%RSD:       .2    26.3     1.2      .6   115.0

Results in Atomic Percents

SPEC:        O
TYPE:     SPEC

AVER:   60.035
SDEV:     .060

ELEM:       Si      Fe      Mg      Ca      Al   SUM
    41  19.942    .023  10.094   9.931    .000 100.000
    42  20.049    .013   9.884  10.030    .004 100.000
    43  19.935    .015  10.000  10.055    .013 100.000
    44  19.956    .018  10.021   9.932    .049 100.000
    45  19.982    .013   9.803  10.045    .018 100.000

AVER:   19.972    .016   9.960   9.999    .017 100.000
SDEV:     .046    .004    .116    .062    .019
SERR:     .021    .002    .052    .028    .009
%RSD:       .2    26.2     1.2      .6   115.0

Results Based on 6 Atoms of o

SPEC:        O
TYPE:     SPEC

AVER:    6.000
SDEV:     .000

ELEM:       Si      Fe      Mg      Ca      Al   SUM
    41   1.994    .002   1.009    .993    .000   9.998
    42   2.004    .001    .988   1.003    .000   9.996
    43   1.994    .002   1.000   1.006    .001  10.003
    44   1.995    .002   1.002    .993    .005   9.996
    45   1.994    .001    .978   1.002    .002   9.977

AVER:    1.996    .002    .995    .999    .002   9.994
SDEV:     .005    .000    .012    .006    .002
SERR:     .002    .000    .006    .003    .001
%RSD:       .2    26.3     1.2      .6   115.0
```

Data

Click this button to see the raw count rate data for the sample. The count data will also be printed to the log window. The upper grid will display the count statistics for the sample averaged data including the standard deviation, one sigma, percent relative standard deviation, minimum and maximum values.

The one sigma values represent a predicted standard deviation based on the count rate and the counting time used for the acquisition. It is calculated by dividing the square root of the count rate by the square root of the average counting time. Therefore when counting at one second count times, the one sigma value will be exactly equal to the square root of the count rate. For sample sets using a 10 second counting time, the one sigma value will be smaller than the simple square root by the square root of 10 or approximately 3.162.

Use the one sigma value to evaluate whether a given average count rate represents a homogeneous set of sample data. Standard deviations larger than the one sigma value indicate the possibility of actual compositional variation within the data set. The percent relative standard deviation is simply the standard deviation divided by the average count rate.

Use the Copy buttons to copy this data as tab delimited data to the Windows Clipboard for pasting into another application. The data will automatically formatted as tab delimited text.

The log window will contain additional information including the stage positions for each point in the sample, the on and off-peak positions (absolute and relative offset, the average on-peak and high and low off-peak count times, and for unknown samples only, the Unknown Count Factor multiplier (UNFAC:) and Maximum Counts (MAXCT:), and finally the Volatile Calibration Assignment (VOL#:) which is equal to the unknown sample number for the volatile self calibration or equal to another unknown sample number in the case of the assigned volatile calibration.

This is followed by the background acquisition type of Mean Atomic Number (MAN), or off-peak (OFF), spectrometer motor number, crystal type, and acquisition order and the MAN or off-peak corrected counts for the sample.

After this, the calculated off-peak counts are also printed out along with the background calculation type (if applicable) of Linear interpolation (LINEAR), high off-peak only (HIGH), low off-peak only (LOW), average of high and low (AVER) and exponential interpolation (EXPON).

Example of Data Printout

```
St  358 Set   1 diopside (Chesterman)
TakeOff =  40   KiloVolts =  15    Beam Current =  20   Beam Size =  5
Number of Lines:  5 Number of 'Good' Lines:  5

Stage (or Beam Deflection) Coordinate Positions:
        X        Y        Z        W               X        Y        Z        W
  146G  -6791.0  -6774.0  91.0000  .000000   147G  -6785.0  -6774.0  91.0000  .000000
  148G  -6779.0  -6774.0  91.0000  .000000   149G  -6773.0  -6774.0  91.0000  .000000
  150G  -6767.0  -6774.0  91.0000  .000000

Last (Current) On and Off Peak Count Times ("----" indicates default max count):
ELEM:    si ka   al ka   fe ka   mg ka   cr ka   ti ka   mn ka   ca ka  BEAM
BGD:       OFF     OFF     OFF     OFF     OFF     OFF     OFF     OFF
MOTOR:       1       1       2       4       3       3       2       5
CRYST:     TAP     TAP     LIF     TAP     LIF     LIF     LIF     PET
ONTIM:   20.00   20.00   20.00   20.00   20.00   20.00   20.00   20.00
HITIM:   10.00   10.00   10.00   10.00   10.00   10.00   10.00   10.00
LOTIM:   10.00   10.00   10.00   10.00   10.00   10.00   10.00   10.00

On-Peak (off-peak corrected) or MAN On-Peak X-ray Counts (and Faraday Current):
ELEM:    si ka   al ka   fe ka   mg ka   cr ka   ti ka   mn ka   ca ka  BEAM
BGD:       OFF     OFF     OFF     OFF     OFF     OFF     OFF     OFF
MOTOR:       1       1       2       4       3       3       2       5
CRYST:     TAP     TAP     LIF     TAP     LIF     LIF     LIF     PET
ORDER:       1       2       1       1       1       2       2       1
  146G  8340.8     5.4     1.7  3305.2     -.4     -.3      .4  2335.1  20.063
  147G  8331.9     6.9     1.9  3287.4     -.3     -.3      .3  2350.0  20.059
```

```
  148G  8347.0     4.4      .9  3289.6      .8     -.7     2.4  2347.0  20.067
  149G  8355.2     7.9     2.3  3303.1      .5      .1      .9  2346.5  20.081
  150G  8340.0     4.3     1.9  3274.8      .1      .0     1.5  2369.2  20.072

AVER:   8343.0     5.8     1.8  3292.0      .1     -.2     1.1  2349.6  20.068
SDEV:      8.7     1.6      .5    12.4      .5      .3      .9    12.4    .009
1SIG:     20.4      .5      .3    12.8      .1      .1      .2    10.8
SERR:      3.9      .7      .2     5.6      .2      .1      .4     5.5
%RSD:       .1    27.4    29.4      .4   401.5  -137.7    79.6      .5

Off-Peak (calculated) X-ray Counts (and Absorbed Current):
ELEM:    si ka   al ka   fe ka   mg ka   cr ka   ti ka   mn ka   ca ka  BEAM
TYPE:   LINEAR  LINEAR  LINEAR  LINEAR  LINEAR  LINEAR  LINEAR  LINEAR
  146G    35.2    22.8     4.2    16.5     2.6     1.6     3.4    11.9    .000
  147G    34.3    22.3     5.0    15.9     3.0     1.6     3.7    13.4    .000
  148G    32.9    22.1     4.6    16.4     2.0     1.7     2.5    13.3    .000
  149G    33.7    20.7     4.4    17.2     2.2     1.3     3.3    14.1    .000
  150G    34.2    23.1     4.6    15.1     2.5     1.3     2.7    13.8    .000

AVER:     34.0    22.2     4.6    16.2     2.5     1.5     3.1    13.3    .000
SDEV:       .9      .9      .3      .8      .4      .2      .5      .8    .000

Raw Hi-Peak X-ray Counts (and Absorbed):
ELEM:    si ka   al ka   fe ka   mg ka   cr ka   ti ka   mn ka   ca ka  BEAM
  146G    32.1    19.3     4.0    15.5     2.9     1.6     3.6    11.5    .000
  147G    31.3    19.4     5.3    15.8     3.2     1.1     3.4    13.0    .000
  148G    28.2    18.6     4.7    14.9     2.1     1.5     2.0    14.9    .000
  149G    29.4    18.1     4.1    17.5     2.2     1.3     3.4    15.0    .000
  150G    31.5    19.5     4.7    14.8     2.6     1.6     2.6    12.8    .000

AVER:     30.5    19.0     4.6    15.7     2.6     1.4     3.0    13.4
SDEV:      1.6      .6      .5     1.1      .5      .2      .7     1.5

Raw Lo-Peak X-ray Counts (and Absorbed):
ELEM:    si ka   al ka   fe ka   mg ka   cr ka   ti ka   mn ka   ca ka  BEAM
  146G    40.1    26.5     4.6    17.8     2.2     1.6     3.1    12.3    .000
  147G    39.0    25.5     4.2    16.0     2.7     2.1     4.3    13.8    .000
  148G    40.0    26.0     4.5    18.3     2.0     2.0     3.3    11.7    .000
  149G    40.2    23.5     5.1    16.9     2.2     1.4     3.1    13.1    .000
  150G    38.4    26.9     4.5    15.4     2.4     1.1     2.9    14.8    .000

AVER:     39.5    25.7     4.6    16.9     2.3     1.6     3.4    13.1
SDEV:       .8     1.3      .3     1.2      .3      .4      .5     1.2
```

KRaws

Click this button after performing a quantitative analysis and the raw k-ratios will be displayed in the Analyze! grid. This button is disabled until an analysis is performed.

The raw k-ratio is defined as the unknown count rate divided by the standard count rate. The calculation uses background corrected count rates.

Note that Probe for Windows also corrects the raw k-ratios for MAN backgrounds, on-peak interferences, APF (Area Peak factors) and volatile element corrections. This provides very useful information for importing into other recalculation programs (film thin analysis software for example).

Use the Copy buttons to copy this data as tab delimited data to the Windows Clipboard for pasting into another application.

Example of K-Raw Output

```
Line    si ka Off      fe ka Off      mg ka Off      ca ka Off      al ka Off
 41 G    .50845         .00100         .16490         .99360        -.00042
 42 G    .51184         .00054         .16137        1.00339         .00007
```

```
 43 G     .50878         .00065         .16337        1.00663         .00027
 44 G     .50874         .00076         .16369         .99344         .00098
 45 G     .50920         .00057         .15963        1.00294         .00036
```

Analyze Selected Line(s)

To analyze a subset of the data lines in the selected sample, first select the lines in the data grid and then click this button. The results, average and statistics of only the selected lines will be displayed even if they were previously deleted.

The lines not analyzed are temporarily flagged as deleted and are therefore eliminated from the analytical calculations and averages.

Delete Selected Sample(s)

Click this button to delete the currently selected samples. The program will flag each data line in the sample as "B" for bad. This will prevent the data in this sample from being used in any analytical calculation or calibrations (if the sample is a standard).

However, if the Analyze Selected Lines option is used, even the deleted lines will be analyzed if they were selected using this option.

Any deleted line or sample can be undeleted by using the Undelete buttons described below. An undeleted line is displayed with a "G" flag for good.

Undelete Selected Sample(s)

Click this button to undelete the currently selected samples. The program will flag each data line in the sample as "G" for good. This will allow the data in this sample to be used in any analytical calculation or calibrations (if the sample is a standard).

Specified Concentrations

Probe for Windows allows the user to specify concentrations of elements not analyzed for. Consider that in order for the program to compute matrix corrections properly, the program must know the concentration of all significant element concentrations. This means that the program should be able to compute a total of at least 90-95% to calculate matrix effects properly. Element concentrations which you have previously measured or estimated but are not analyzing for, can be manually specified.

This feature is especially useful when adding excess oxygen from Pressure-Temperature (PT) calculations or H2O for hydrous phases. The effect of absorption by oxygen upon Si and Al x-rays is not insignificant and must be considered for the most accurate results.

To specify the concentration of an element not analyzed for in an unknown sample (standard samples will have elements not analyzed for, loaded automatically from the standard database), first select the samples to assign the specified concentrations to from the Analyze! sample list box.

First make sure the element(s) to be calculated as a specified element, are in the sample setup by clicking the Elements/Cations button. If the element is not in the sample element list, add it as a specified element by clicking any empty element row and typing the element in the element field. Be sure to select an x-ray line of none by clicking a blank x-ray line. Then click OK or type <enter> to accept the element.

Then go back to the Specified Concentrations button and click the specified element. Enter the composition and click OK or type <enter>.

Remember that the specified concentrations for unknown samples will be automatically loaded into each new unknown sample that is created based on the last unknown sample.

Standard Assignments

The standard assignments dialog is used to select the primary standard for the calibration of the quantitative analysis. Each analyzed element must have a standard assigned to it. This dialog is also used to specify the quantitative correction of on-peak interferences and the volatile element correction. Remember that although the program will automatically load the standard assignments based on the last unknown sample, the standard assignments may be assigned at any time on a sample by sample basis.

The program will load a default set of standard assignments the first time this dialog is opened. These default standard assignments are simply based on the standard containing the highest concentration of each element. It will sometimes be necessary to modify these defaults standard assignments depending on the analytical situation.

Generally when assigning standards for use in the analytical calibration, it is best to use primary standards which contain a major concentration of the element. Standards not assigned but also containing the element are secondary standards and are valuable for checking the quality of the calibration. This can be seen by examining the "%VAR" line in the analysis printout.

Although standards can be re-assigned at any later time, the user should be aware that the use of "quick" standards may restrict one's ability to re-assign standards off-line since the required data may not have been acquired. When running on-line however, you can re-run the affected "quick" standards after changing the standard assignments to obtain the necessary calibration data.

Virtual Standard Intensities

A new option is the use of "virtual" standard intensities. These are calculated intensities based on interpolation and matrix corrected similar (atomic numbers) elements measured on the same crystal, spectrometer and voltage as the unknowns. This may be useful in cases where a physical standard is not available (e.g., Ar).

See the Calculate Virtual Standard Intensity menu under the Analytical menu for the interactive dialog for use in calculating and storing these parameters.

Spectral Interferences

Probe for Windows allows the user to select a fully quantitative correction for spectral interferences. To help inform the user of possible on-peak interferences, the program will give the user the option of displaying all possible interferences based on the current set of analyzed and interfered elements. However, remember that the program can only correct for interferences if both the interfered and interfering elements are analyzed for. In addition, an interference calibration standard must be acquired that contains a major concentration of the interfering element and none of the interfered element or any other elements that interfere with the interfered element.

The interference correction used by Probe for Windows is similar to that used by some investigators (Snetsinger, Bunch and Keil, 1968), but is quantitative and more general, and can be applied easily to the analysis even if the spectral line type of the interfering element is not known.

To specify an interference simply select the element that is interfered with from the Standard and Interference Assignments window and select the interfering element from the element list of analyzed elements. If the interfering element is not already an analyzed element, it must be added to the sample setup using the Elements/Cations button in the Acquire! window. Next select a standard to be used for the interference calibration from the list of standards in the run. If the standard is not currently in the run, it must be added to the run using the Standards | Add Standards To Run menu item and have data acquired for it.

Standards which will be used in the interference corrections should not be run as "quick" standards. Data for the interfered channel is necessary to calibrate the correction which may not be present with a "quick" standard. If you find that you need to apply an interference correction which has already been run as a "quick" standard, you can simply re-run the standard again, this time though ***not*** as a "quick" standard. It is not necessary to delete the previous "quick" standard as the program will ignore channels of standards which contain zero counts.

Note that once a sample setup has had interferences assigned, the program will automatically acquire the necessary intensity data even if the quick standard option has been selected.

As an example of the interference assignment, consider the following : when analyzing for iron and manganese, you may notice that when high concentrations of manganese are present, that small amounts of iron will also appear to be present due to an interference of Mn Kβ with the Fe Kα analytical line. To correct for this interference you need a standard that contains manganese but does not contain any iron. Often, you can assign the same standard that is used for the primary standard calibration, also for the interference calibration, in this case, a MnO synthetic crystal. You could also use any other manganese standard not containing appreciable iron such as a synthetic manganese silicate.

Note that if you needed to analyze for Fe, but Mn was not present in any significant quantity, you could justifiably ignore the interference, since you would never see it. What can be considered an insignificant interference is something that needs special consideration.

One useful suggestion is to use either the Standard for Windows program's Interference menu item or, even better, use the Interferences button in the standard assignment dialog to provide an approximate calculation of the interference. The program will calculate the interference based on a gaussian peak shape by assuming a worst case of 0.1% of the analyzed element and 100% of each of the other analyzed (and possibly interfering elements).

Note that in the above case, Mn interferes with Fe, but the reverse is not true : Fe does not interfere with Mn. However in some cases both elements interfere with each other. Probe for Windows can also handle this situation because the interference correction is an iterated solution. For example : consider the case of Ti Kα and Ba Lα. Both lines interfere with each other and both lines are used for quantitative analysis. To correct for this double Ti and Ba interference, the analyst will be required to analyze for both Ti and Ba. In addition, two interference standards will be required. To correct for the interference on Ti, a standard will be needed that contains Ba, but no Ti. To correct for the interference on Ba, a standard will be needed that contains Ti, but no Ba. In practice, if you used TiO_2 for the Ti and barite ($BaSO_4$) for Ba, as the analytical standards, you can also use Barite as the Ti interference standard and TiO_2 as the Ba interference standard.

The actual form of the quantitative interference within the ZAF iteration loop is given below :

$$C^{u}_{A} \approx \frac{C^{s}_{A}}{[ZAF]^{s}_{l_A}} [ZAF]^{u}_{l_A} \frac{I^{u}(l_A) - \dfrac{[ZA]^{\bar{s}}_{l_A}}{C^{\bar{s}}_{B}} \dfrac{C^{u}_{B}}{[ZA]^{u}_{l_A}} I^{\bar{s}}_{B}(l_A)}{I^{s}_{A}(l_A)}$$

Where the following notation has been adopted :

C^{j}_{i}	is the concentration of element i in matrix j
$[ZAF]^{j}_{\lambda_i}$	is the ZAF (atomic #, absorption and fluorescence) correction term for matrix j (Z and A are for wavelength λ_i and F is for the characteristic line at λ_i for element i)
$I^{j}_{i}(\lambda_i)$	is the measured x-ray intensity excited by element i in matrix j at wavelength λ_i.
$\bar{s}$	refers to an interference standard which contains a known quantity of the interfering element B, but none of the interfered element A.

The quantitative interference corrections in Probe for Windows allow the user to correct for up to 4 interfering elements per channel. Since the solution is iterated the program can correct for interferences when two (or more) analytical lines interfere with each other. See Donovan, et al., (1993) for more details on the derivation of the quantitative interference correction used here.

Note also that you can use a standard for the analytical calibration, even if the element channel used for the calibration is interfered with, as long as you correct for the interference as described above. In the case of a standard that contains interfered elements, the correction of the standard counts is simple, since the composition, is by definition, already known from the standard database. Therefore, in the case of standards at least, an iterated procedure is not required.

Area Peak Factors and Standard Assignments

Another consideration in assigning standards, concerns the use of Area Peak Factors (APF) for correction of light element peak shift and shape changes. When analysis of light elements is to be performed (typically oxygen, nitrogen, carbon and boron but possibly heavier elements such as silicon, aluminum and sulfur), you need to plan the use of APF corrections for the most accurate results. The actual APF values selected will depend the standard assigned for calibration of the light element.

For example, when analyzing for C Kα and using Fe_3C as your carbon standard, your APF value for C Kα in a Cr_7C_3 matrix might be 0.80. However, if instead, you re-assigned your carbon standard to TiC, you must use APF values relative to TiC rather than Fe_3C. If they are available in the Empirical APF database (EMPAPF.DAT), just change the APF values for C Kα. Otherwise you would need to calculate the Fe_3C APF values relative to TiC. Some typical Fe_3C APF values for C Kα, recalculated relative to TiC and Cr_7C_3, are shown in the table below :

APF Relative To :	Fe_3C (APF/1.000)	TiC (APF/0.868)	Cr_7C_3 (APF/0.80)
Fe_3C	1.000	1.152	1.25
TiC	0.868	1.000	1.085
Cr_7C_3	0.80	0.92	1.00

Checking Secondary Standards

Once the primary standards are assigned it is recommended to analyze the standards to check for internal consistency. For this, the acquisition of secondary standards is preferred. Secondary standards are standards in which no elements (or only some) are not assigned as the primary standard for the analyzed elements.

From the Analyze! window, first select all the standards. Then check the Pause Between Sample check box. Then click the Analyze button. The program will pause between each sample analysis to allow the user to view the results. Look over the results carefully. Standards whose x-ray intensities have been assigned as the standard for that element will normally look close to perfect since they are by definition the primary standard. The other unassigned elements are treated as unknowns and by observing the "%VAR" (percent variance) line, one can get an idea of the quality of the standardization by examining these secondary standards.

For example, assume that you are analyzing for Si, Al, Na and K and your assigned standards are SiO_2 for Si, $NaAlSi_3O_8$ (albite) for Al and Na, and $KAlSi_3O_8$ (orthoclase) for K. In this case SiO_2 is your primary standard for Si, and both albite and orthoclase can serve as secondary standards to check the quality of the Si calibration since they are measured for but not assigned as calibration standards for Si. In addition, the orthoclase standard can be used as a secondary standard in the example above, to check the Al calibration. Therefore, orthoclase is the primary standard for K, but is a secondary standard for both Si and Al, and albite is the primary standard for Al and Na, but is a secondary standard for Si only, while SiO_2 is the primary standard for Si only. Note that a pure standard such as synthetic SiO_2 can be used to check the quality of the background correction on the Al, Na and K channels in this example, since pure SiO_2 should not contain those elements in significant quantities.

While looking over the standard results, look carefully for "bad" points. By this, it is meant points whose total compositions are anomalously low. These could be points where the beam was on epoxy or perhaps the surface was poorly polished. In any case you will see this as a low total. These points may be deleted.

Note that points whose totals are high, on the other hand, should not be deleted arbitrarily. High totals indicate that something else is wrong. Check for low total points on another standard or for the presence of spectral interferences.

Volatile Element Corrections

If the volatile element extrapolation option is selected the program can automatically correct for changes in composition over time. This is often the case for volatile elements such as sodium or potassium, but the extrapolation correction can also be applied to any degradation (or enhancement) of the x-ray intensities over time due to other causes such as sample damage, carbon contamination, etc. This correction is especially useful for samples which are too small to utilize a defocused beam and allows the user to run higher sample currents to improve the analytical sensitivity.

Two different methods are utilized by probe for Windows to correct for volatile element loss, first is an assigned method where an unknown sample is acquired and then later assigned for use as a calibration curve for one or more samples. The other is a "self" calibration where intermediate intensities are saved as the data is acquired and later used a the basis for calculated the volatile correction.

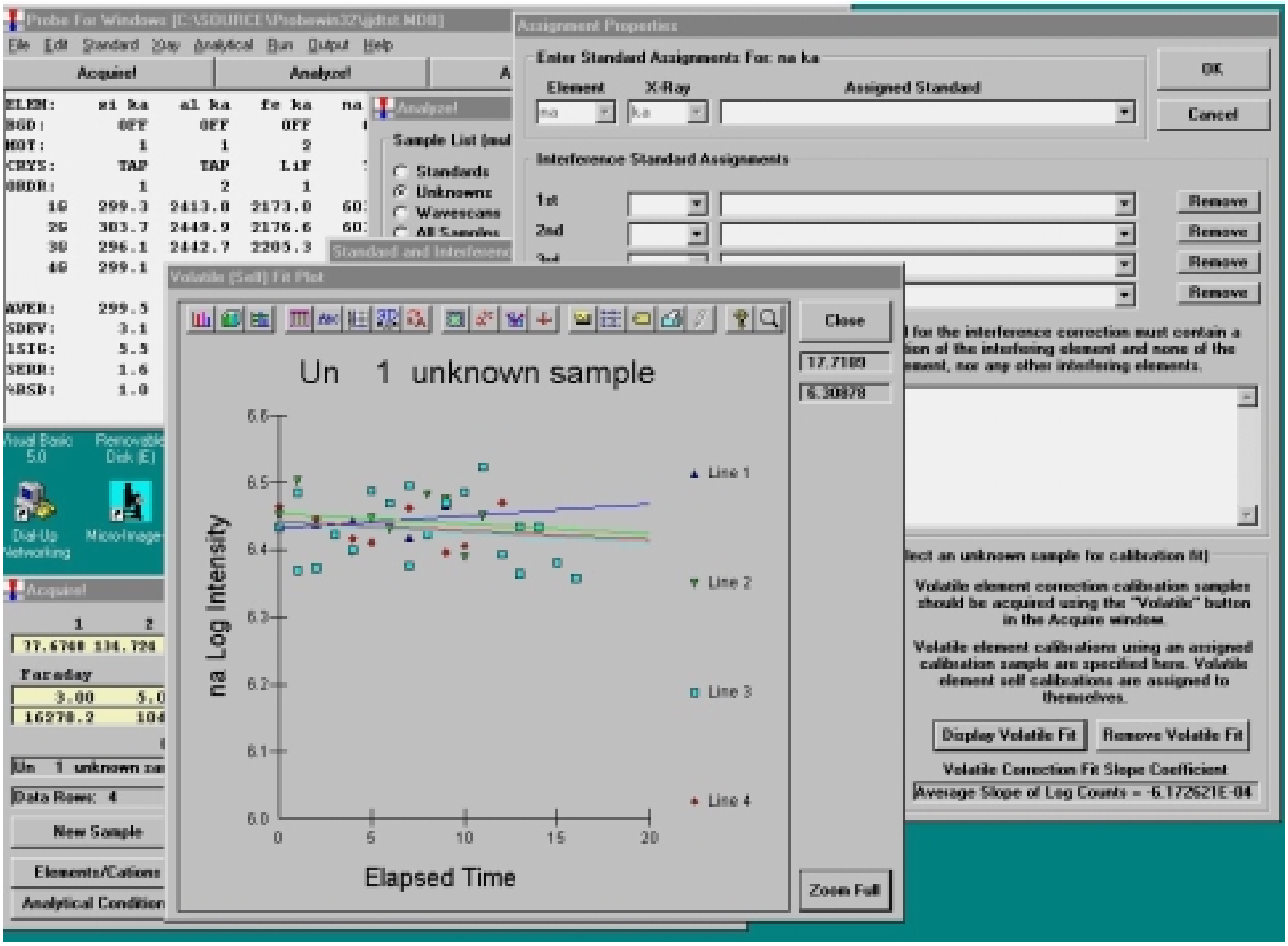
Mathematical Method

In both cases, the slope coefficient is calculated using a linear fit of intensity versus elapsed time. Intensities that are less than or equal to zero are ignored. The count data is normalized to counts per second and the natural log taken. The elapsed time is converted to seconds and the linear (straight line) fit is calculated. Only the slope parameter is saved for the actual volatile correction to allow the correction to be applied to a variety of samples that may contain different absolute intensities at different times (due to beam or standard drift).

Once the slope is calculated it is used in the following expression to adjust the unknown intensity for volatile element loss (or gain):

$$I_C = e^{Log\left(I_R\right) - m \cdot t}$$

where: I_R = Raw (uncorrected) intensity in counts per second
m = Volatile slope coefficient from linear fit of Log intensity versus time
t = Count time for the raw (uncorrected) intensity
Log = Natural logarithim



```
ELEM:     si ka    al ka    fe ka    na
BGD:        OFF      OFF      OFF
MOT:          1        1        2
CRYS:       TAP      TAP      LiF
ORDR:         1        2        1
   1G     299.3   2413.0   2173.0   60
   2G     303.7   2449.9   2176.6   60
   3G     296.1   2442.7   2205.3
   4G     299.1

AVER:     299.5
SDEV:       3.1
1SIG:       5.5
SERR:       1.6
%RSD:       1.0
```

1: 77.6760 2: 134.724

3.00 5.0

16270.2 104

Un 1 unknown sa

Data Rows: 4

17.7189

6.30878

Element: na X-Ray: ka

l for the interference correction must contain a
tion of the interfering element and none of the
ement, nor any other interfering elements.

lect an unknown sample for calibration fit)

Volatile element correction calibration samples should be acquired using the "Volatile" button in the Acquire window.

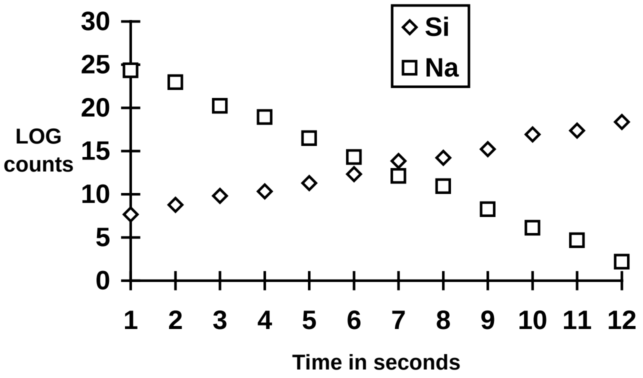
Volatile element calibrations using an assigned calibration sample are specified here. Volatile element self calibrations are assigned to themselves.

Average Slope of Log Counts = -6.172621E-04

Typical Situation for Volatile Correction

For instance, when sodium loss in observed in an alkali glass sample, a corresponding gain in silicon and aluminum x-rays may be noted. The extrapolation correction used in Probe for Windows can be applied to some or all elements in an sample, regardless of whether the x-ray intensities are decreased or increased during the acquisition **(as long as the elements to be corrected are acquired as the first element on each spectrometer, i.e., order number = 1)**.

The correction assumes that the change in counts is linear versus time when the natural LOG of the x-ray counts are plotted (Nielsen and Haraldur, 1981) as shown here :



Depending on the sample, this may or may not be a valid assumption. Under certain conditions, with very volatile hydrous alkali glasses, the change in count rate may actually decrease more quickly than a simple log decay. In this case, it may be necessary to defocus the beam slightly before acquisition.

How to Use the Volatile Correction

To utilize the volatile correction first decide if you require an assigned calibration curve type correction or a self calibration curve type correction. In the self calibration correction the user simply selects the "Self-Calibration" option (see Special Options button in the Acquire! Window) and acquires the data point(s) as one would normally. In the assigned volatile correction the user first selects a sample that is similar in volatile characteristics to the sample to which the assigned calibration curve will be assigned to (it could be the actual sample itself) and then selects the "Assigned Calibration" option (see Special Options button in the Acquire! Window). The next acquisition that is started will create a calibration curve sample that can be assigned to one or more samples for the purpose of correcting volatile loss.

The difference between the two methods is that the assigned calibration curve will apply the same slope correction to all points within the assigned sample. Therefore the variation seen in the sample concentrations do not reflect any statistical variation in the calibration. The self calibration method acquires a calibration curve separately for each data point and therefore the variation from point to point may be partially due to statistical variation between the calibration curves.

Both of the calibration corrections are quantitative in that the correction is based on a calibration curve acquired during the run, on the sample itself or another sample of similar characteristics, and is applied during the ZAF or Phi-Rho-Z iteration phase of the analysis to correct for changes in the matrix correction due to the extrapolation correction. Any number of calibration samples may be acquired and any sample or range of samples can be assigned to use any of the calibration samples for the extrapolation correction. Note that the a previously assigned volatile element correction is automatically loaded for new unknown samples.

Note that the correction uses the actual elapsed time for all calculations to produce the most quantitative results. Nevertheless, the user should be aware that the technique must be applied carefully to obtain the best results.

One possible source of error, with this calculation, is that the correction is not applied to the standard calibration itself. For this reason, it is essential to use standards for all the analyzed elements that are stable under the beam. If it is not possible, for instance, to run your albite standard without affecting the count intensities in the acquisition time used, it would be better to avoid the volatile element extrapolation and use a standard that is similar to the unknown sample itself.

Another concern is with samples that contain more than one element on a given spectrometer. Exactly how the volatile element correction should be applied in such situations is not clear, and therefore Probe for Windows will only apply the volatile element correction for elements that are acquired first on a given spectrometer.

Name/Description

Probe for Windows allows the user to change the sample name and description fields at any time. Use the Insert <cr> button to add a carriage return to the description field for multi-line comments.

The name field is limited to 64 characters and the description field is limited to 255 characters. Any printable characters may be used along with spaces for the name or description of the sample(s).

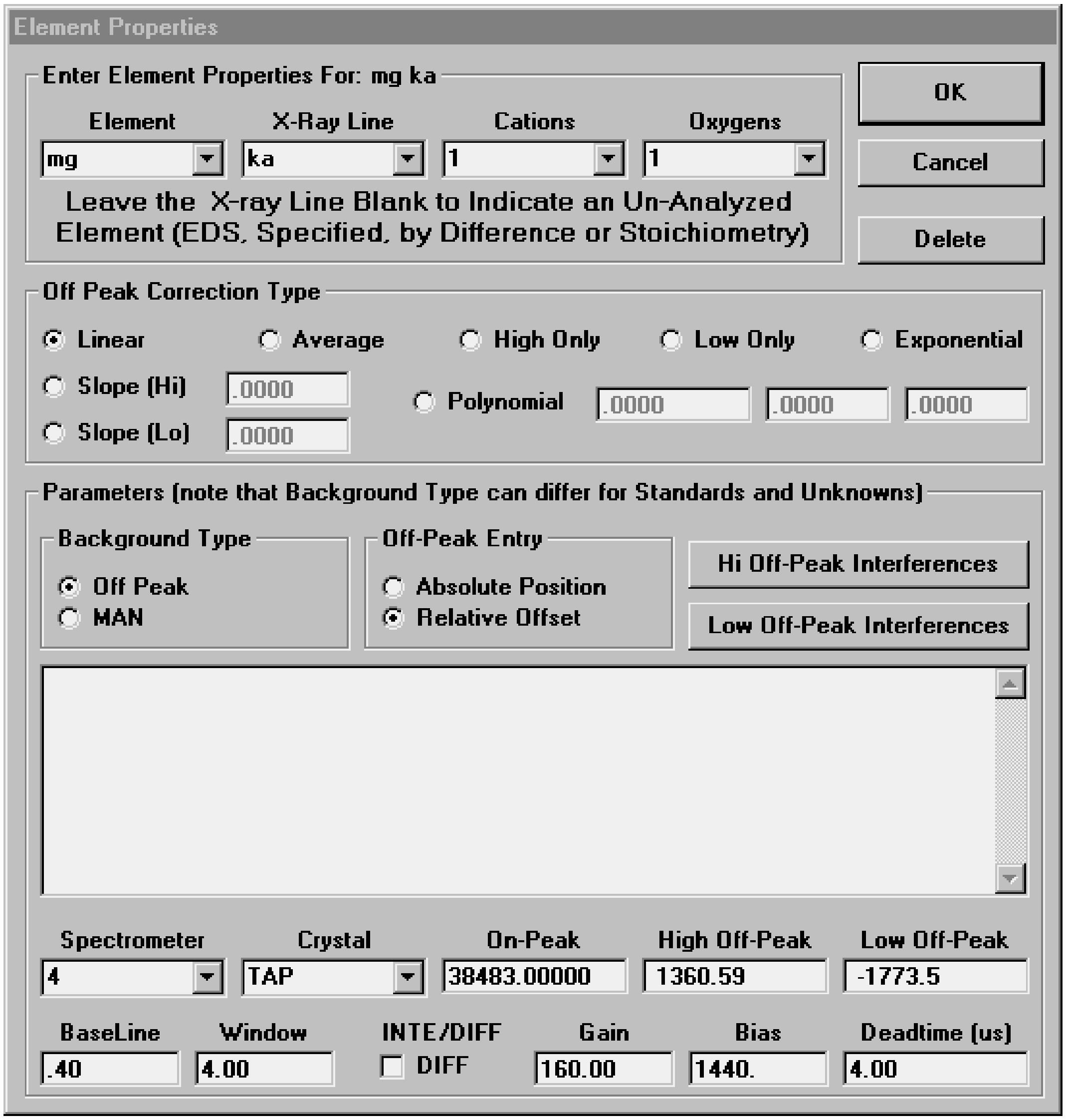
Conditions

The conditions dialog can be used to display the sample conditions. These include the operating voltage in kilovolts, the takeoff angle in degrees, the beam current in nA and the beam size in microns. These fields are used for documentation purposes only, unless the hardware interface is supports controlling these instrument parameters.

For the SX50 interface (InterfaceType=6) and if the BeamSizePresent flag is true in the PROBEWIN.INI file, the beam size parameter will force the instrument into FIX mode and set the beam size using the SIZE command. In this mode the instrument beam scanning is disabled. Therefore if beam scanning is desired, the beam size should be set to zero so that a SIZE OFF command is sent to the instrument which will enable the beam scanning.

Elements/Cations

Clicking the Elements/Cations button opens the analyzed and specified elements dialog box. The analyzed and specified dialog box lists the currently selected elements for analysis and calculations.



Element Properties

Enter Element Properties For: mg ka

Element	X-Ray Line	Cations	Oxygens
mg	ka	1	1

Leave the X-ray Line Blank to Indicate an Un-Analyzed Element (EDS, Specified, by Difference or Stoichiometry)

Off Peak Correction Type

● Linear ○ Average ○ High Only ○ Low Only ○ Exponential

○ Slope (Hi) .0000

○ Slope (Lo) .0000

○ Polynomial .0000 .0000 .0000

Parameters (note that Background Type can differ for Standards and Unknowns)

Background Type: ● Off Peak ○ MAN

Off-Peak Entry: ○ Absolute Position ● Relative Offset

Spectrometer	Crystal	On-Peak	High Off-Peak	Low Off-Peak
4	TAP	38483.00000	1360.59	-1773.5

BaseLine	Window	INTE/DIFF	Gain	Bias	Deadtime (us)
.40	4.00	☐ DIFF	160.00	1440.	4.00

Use this button to add specified elements (elements calculated as fixed concentrations or by difference or stoichiometry) to the selected samples. Also the element cations and oxygens can be changed on a sample by sample basis for the calculation of oxide weight percents.

> **Use the Save Element Setup button to save element setups to the element setup database for use in future runs. Normally one should save the element setup of an element that is assigned as the standard for that element. In this case the standard intensity data is saved to the element setup database for reference purposes.**

Use the Save Sample Setup button to save the currently selected sample as a sample setup for use in creating subsequent samples within the current run. Note that selecting a previous sample setup will update the current element setup with the selected sample setup.

From the Analyze! window, only samples with no data can have their acquisition parameters modified. Parameters for samples that contain data can only be viewed. However, the off-peak type and deadtime parameter can be modified in either case. Off-peak correction types include linear, average, high only, low only, and exponential in addition to slope and polynomial fit backgrounds. Plot a wavescan sample(s) in the Plot! Dialog and click the Model Backgrounds button to see the various background options graphically.

Probe for Windows uses a linear slope calculation as the default off-peak background correction. Because of this, unsymmetrically offset background position measurements can be easily handled. One can even position both of the off-peak measurements on the same side of a peak and extrapolate the background correction.

Off Peak Background Correction Types

Probe for Windows allows the user to select the off-peak type for the background correction. The default slope calculation is the most accurate, except in cases where the user has moved both off-peak positions to the same side of the analytical peak and adjusted the off-peaks excessively close together resulting in a large extrapolation. The off-peak correction types are as follows :

0 = linear slope interpolation or extrapolation
1 = average of both high and low off-peak counts
2 = use only the high off-peak counts
3 = use only the low off-peak counts
4 = exponential off-peak calculation (based on exponent)
5 = slope-high (based on high off-peak and slope coefficient)
6 = slope-low (based on low off-peak and slope coefficient)
7 = polynomial (based on high and low off-peak and three coefficients)

These off-peak background correction types may be assigned on a sample by sample basis either on or off-line. Note that the actual values of the polynomial fit coefficients are dependent on the absolute magnitude of the count intensities. For this reason it is important to avoid changing the value of the nominal beam current once any polynomial fit coefficients have been assigned to samples in the run. The calculations for linear slope and exponential calculation are shown below :

Linear Slope Calculation

$$I_{off} = I_{low} + (I_{high} - I_{low}) \bullet \frac{(P_{on} - P_{low})}{(P_{high} - P_{low})}$$

where :

I_{off} = the off-peak count correction
I_{high} = the x-ray counts for the high off-peak position
I_{low} = the x-ray counts for the low off-peak position
P_{high} = the high off-peak position
P_{low} = the low off-peak position
P_{on} = the on peak position

Exponential Slope Calculation

$$I_{off} = \frac{ce^{-aP}}{P^N}$$

where : I_{off} = the off-peak background intensity
P = the spectrometer position
c = a fit parameter
a = a fit parameter
e = the exponential
N = a user specified exponent

Note that the slope and polynomial off-peak corrections are calculated for each sample line based on the actual measured off-peak intensities for the sample. The coefficients can be modeled from the Plot! Window by displaying a wavescan sample in the Graph dialog and clicking the Model Background button.

Same Side Off-Peaks

Beware of placing same-side off-peaks too close together. A large extrapolation can cause a very large error in the off-peak background correction if the background standard deviation is large. The program will print a warning if it feels that the same-side off-peak extrapolation is excessive. In cases where the same side off-peak extrapolation is excessive, the user may want to select using the average of the off-peak counts. Of course, averaging the off-peak counts is really only applicable in situations where the background slope is zero.

Interferences

The High and Low Off-Peak Interference buttons can be used when both the potential interfering and interfered elements are already present in the sample setup to determine the approximate magnitude of the off-peak interferences. This calculation is based on a gaussian peak shape and various crystal defaults and is intended as a guideline only for off-peak position entry.

The program assumes 100% of the interfering element and 0.1% of the interfered element in each binary pair calculation. For example, if the percent interference is calculated as 10%, then that indicates that instead of measuring 0.1 wt. % of the element, it is likely that a concentration of 10% greater than 0.1 wt. % or 0.11 wt. % will be measured. Note that since this is only a nominal calculation, it should only be used as an indicator of possible problematic analytical situations.

Specified Area Peak Factors (fixed composition APF)

As an alternative to the "calculated" Area Peak Factors, Probe for Windows offers the use of "fixed" composition Area Peak Factors. These are single values that are applied to the element intensity on an emitter basis (as opposed to calculated based on the actual absorber concentrations in the matrix).

These factors may be useful in certain situations where the compositional range is limited and binary Area Peak Factors are not available for some or all elements in the matrix (e.g., Na, K or Ca in an oxygen matrix). This option may be turned off or on in the Analysis Option dialog under the Analytical menu.

Save Element Setup

This button opens the element setup database dialog. Here the user can save element setups to the setup database for re-use in another run or sample setup.

Generally, one usually saves standard sample element setups for elements that are assigned to that standard to the setup database. Later, one can retrieve these calibrated setups for use in creating new sample setups for acquisition in another run.

It is recommended that the user save element setups after selecting the primary standard for that element. In this way the element setup database will preserve a record over time of the peak intensities and peak to background ratios for evaluation purposes.

Save Sample Setup

This button activates the Sample Setup dialog box. Here the operator can save sample setups for starting a new sample based on the previous sample setup within the current run.

Subsequently the user can use the Load Sample Setup button in the New Sample dialog (accessed from the Acquire! window) to create new analytical samples or the Setup dialog from the Automate! window when digitizing position samples for automated acquisition.

Note that the saving of a sample setup actually saves only a pointer to the sample selected. This means that any changes to the sample setup calculation options, elements/cations, standard assignments, etc. will be utilized when a new sample is created based on the modified sample setup.

Delete Selected Line(s)

Use this button to *delete* a single data point or a range of data points. Any data points thus deleted will be flagged "B" for "bad" and will not be used in any analysis or calculations until it is undeleted. Points already deleted are not affected.

Select the lines to be deleted in the Data grid by clicking and dragging the mouse. Only contiguous ranges of lines of data may be deleted using this feature.

When analyzing your standards, look carefully for "bad" points. By this, it is meant points whose total compositions are anomalously low. These could be points where the beam was on epoxy or perhaps the surface was poorly polished. In any case you will see this as a low total. These points should usually be deleted. Note that points whose totals are high, on the other hand, should not be deleted arbitrarily. High totals indicate that something else is wrong. Check for low total points on another standard or for the presence of spectral interferences.

Undelete Selected Line(s)

Use this button to *undelete* a single data point or a range of data points. Any data points thus deleted will be flagged "B" for "bad" and will not be used in any analysis or calculations until it is undeleted. Points already deleted are not affected.

Select the lines to be undeleted in the Data grid by clicking and dragging the mouse. Only contiguous ranges of lines of data may be undeleted using this feature.

List Report

This button will list all standard, background and interference calibration intensities that are associated with the currently selected sample(s) to the log window for inspection. This is useful to determine for example, the drift in count rates from one standardization to another or the magnitude of the interference overlaps.

Because each sample can be assigned different standard and interference assignments, be sure that the desired sample is selected before clicking this button. This output also includes the measured or calculated background count rates on each standard set.

It is possible to use this button on multiple selected samples, even if they contain different element setups and assignments. In this case, the program will print a complete report for each different element setup selected.

Calculation Options

The following calculation options are available from the Analyze! window. Note that these options generally do not need to be explicitly set for use from the Plot! window with the exception of the Formula element and basis.

Do Not Use/Use EDS Element Data

If integrated EDS/WDS acquisition data is available for this sample then this option will allow the user to choose whether to use or not use the EDS data stored in the user data file when the matrix correction is calculated. See the Acquisition Options dialog for this acquisition option.

Do Not Use/Use Integrated Intensities

If integrated WDS intensity data is available for this sample, then this option will allow the user to choose whether to use or not use the stored integrated intensities in the quantitative calculations. See the Elements/cations dialog for this acquisition option.

If this user chooses to use the integrated intensities then the program will calculate the deadtime and beam drift correction for the raw integration data and then calculate the integrated area. Finally the program will then calculate the integrated background based on the currently selected background model and subtract that from the integrated intensities. Note that standards acquired using the integrated intensity option must also be acquired for this option.

If the user chooses not to use the integrated intensities then the program will use the peak intensity from the integrated data. Again, for this mode, the standards must still be acquired using the integrated acquisition intensity option and also have the integrated intensities analysis option turned off.

Display Results As Oxides and Calculate With Stoichiometric Oxygen

Probe for Windows allows the user to display the results of an analysis in oxide weight percents based on the cation ratios defined for each element. In addition the user may select to calculate oxygen by stoichiometry if oxygen is not an analyzed element. If oxygen is measured or calculated by stoichiometry, the program will automatically calculate the actual excess or deficit oxygen in the analysis if the Display As Oxide option is selected.

If the element to be calculated by formula basis, difference or stoichiometry is not already in the sample setup, it must be added as a "not analyzed" element using the Elements/Cations button first, by simply clicking any empty row in the element list and typing in an element symbol with a blank x-ray line.

Note that if the sample being analyzed is a standard and oxygen is not measured, then the program will automatically specify any oxygen from the standard database. However, if the sample is an unknown, then the program will add in any specified oxygen as "excess" oxygen into the matrix correction, if the user has specified any element concentrations.

Formula and Mineral Calculations

The program can compute formulas based on any number of oxygens for oxide runs or any analyzed or specified element in elemental runs. In addition you may select either an olivine, feldspar, garnet or pyroxene end-member calculation if you choose a formula based on oxygen.

It should be noted that the formula calculations are based only on atomic weight and do not consider charge balance and site occupancy for the calculations. For more complicated mineral calculations it is recommended to utilize a 3rd party mineral recalculation software. Please contact Advanced Microbeam for more information.

Calculate Atomic Percents

The program can compute the analyses in atomic percent also. The program will calculate the results based on atomic weight and normalized to 100%. This calculation is sometimes known also as elemental mole percents.

Element by Difference

An element by difference can be included in this way also to account for their affect on the x-ray intensities. The element by difference must be one of the specified elements in the sample setup. Note that the this option should be utilized carefully, because the calculation will always result in a 100% total which could mask the results of a bad acquisition.

Stoichiometry to Oxygen

Another useful feature for the analysis of carbonate or borate samples in an oxide run is the use of the "element by stoichiometry to the stoichiometric element (oxygen)" option. With this feature the analyst can analyze just the cations (Fe, Mg, Ca, etc.) in a sample and have the oxygen calculated by stoichiometry and another specified element (usually C for carbonates or B for borates) calculated relative to oxygen. In the case of carbonates, for example $CaCO_3$, carbon is always in the ratio 1 to 3 to oxygen. Therefore by simply specifying C by stoichiometry relative to the stoichiometric element (oxygen) at 0.333 (1 divided by 3) the correct amount of both oxygen and carbon will be incorporated into the ZAF matrix correction and totals without analyzing for either. This calculation should only be used with compounds where the ratio to oxygen is both known and unchanging.

The following is an analysis example of carbon calculated by stoichiometry to oxygen in a carbonate sample for an oxide run :

```
Analysis of sample  st 135   set  2   calcite (analyzed)
Total Number of Data Points =   5            Number of "G" Data Points =   5
Average Beam Counts =   45527.                  Average Base Time =    10.00
Average Sample Z-bar =   12.572            Average BNA Iterations =   11.000
Element : C   is Calculated Relative to Stoich. Oxygen at   .333 to 1.0 Atom

Results in Weight Percent :

        CO2     Cl      MgO     FeO     MnO     O       SUB

SPEC   43.82     .00     .00     .00     .00     .00   43.82
SDEV     .19     .00     .00     .00     .00     .00

BGD:   MAN     MAN     MAN

        CaO     SO3     P2O5    SUM
  29   55.60     .01    -.01   99.12
  30   56.18     .04    -.01  100.22
  31   56.16     .01     .00  100.10
  32   56.02    -.01    -.02   99.77
  33   56.10    -.01     .01  100.00

AVER   56.01     .01    -.01   99.84
SDEV     .24     .02     .01
SERR     .11     .01     .00
PUBL   56.01    n.a.    n.a.   99.90
%VAR     .00     .00     .00
          *

BFAC  1.0314   .9925  1.0285
KRAW  1.0001   .0001  -.0001
P/B:  277.12    1.02     .87
```

*Note that oxygen **must** be an analyzed or specified element before this calculation can be applied.*

One more point about element by stoichiometry to oxygen. Consider the example of a trace element analysis of several metals in an alumina (Al_2O_3) matrix. If Al and O are not to be analyzed, yet the user desires to have Al_2O_3 added to the matrix correction, how can this be accomplished?

There are two ways this can be achieved. One way would be to simply specify Al by difference and calculate oxygen by stoichiometry. The program will then correctly add in the proper amount of stoichiometric Al_2O_3 to the matrix correction for each analysis line. The other way is to use the element by stoichiometry to oxygen calculation as discussed below.

Adjust the cation ratios of the metals to elemental stoichiometry (one cation and zero anions). Next, select "element by stoichiometry to oxygen" and (in this example) select "al" as the element by stoichiometry. To achieve a 2 to 3 ratio, next enter "0.666" Al atoms per O atom. Run the calculation and note that Al_2O_3 was ***not*** added to the matrix calculation! What happened? In this example, the user had selected a cation ratio for the analyzed elements of all elemental atoms, and since there was zero oxygen to begin the iteration, the program never got to add the Al which then never added the stoichiometric oxygen! How can this be avoided? Simply specify some small concentration of an oxide element (for instance SiO_2) in the specified element concentration, say 0.05 %. This will give the iteration a chance to get started, and allow it to converge on a very close approximation of the Al_2O_3 by difference! The following is an example of how this calculation looks :

```
Analysis of lines:
   23   24   25   26   27   28   29   30
Total Number of Data Points =   8           Number of "G" Data Points =   8
Average Beam Counts =   98504.                 Average Base Time =    10.00
Average Sample Z-bar =   10.816           Average ZAF Iterations =    5.000
Element : Al  is Calculated Relative to Stoich. Oxygen at   .666 to 1.0 Atom
Element : O       is Calculated by Stoichiometry

Results in Weight Percent :

        Se      O       Al      Si      SUB

SPEC     .00   46.68   52.41     .05   99.14
SDEV     .00     .02     .02     .00

BGD:   OFF     OFF     OFF

        Zn      Cu      Pd      SUM
  23     .59     .30     .01  100.02
  24     .56     .30     .01  100.02
  25     .57     .32     .00  100.02
  26     .58     .33     .00  100.02
  27     .53     .33     .02  100.02
  28     .55     .39     .01  100.02
  29     .54     .26     .00  100.02
  30     .56     .31     .01  100.02

AVER     .56     .32     .01  100.02
SDEV     .02     .04     .01
SERR     .01     .01     .00

KRAT   .0047   .0027   .0000
ZCOR  1.2009  1.1976  1.2321
KRAW   .0093   .0027   .0000
P/B:    2.70    2.29    1.02
%INT    -.05     .00     .00


Results in Oxide Weight Percents using Custom Cation Oxide Ratios :

       Se      O       Al2O3   SiO2

AVER    .000    .000  99.029    .107
SDEV    .000    .000    .040    .000

       Zn      Cu      Pd      SUM
  23    .592    .302    .006 100.022
  24    .556    .300    .015 100.022
  25    .574    .323    .000 100.022
  26    .581    .330    .000 100.022
```

```
  27    .528    .332    .018 100.022
  28    .550    .386    .008 100.022
  29    .543    .262    .001 100.022
  30    .561    .308    .013 100.022

AVER    .561    .318    .008 100.022
SDEV    .021    .035    .007
```

Stoichiometry to Another Element

Yet another option for recalculation is the element relative to another element (by stoichiometry). This calculation is similar to the "element by stoichiometry to oxygen" as described below, however the user may select any other analyzed or specified element as the stoichiometric basis element.

This calculation can be used in combination with the other calculations to handle unusual analytical situations. For example, in the following analysis of an silica sample, the results revealed significant contamination of the material with Fe and Ca. Investigation using other techniques, implied that the Fe was in the form of Fe_2O_3 (hematite), and the Ca was present as $CaCO_3$ (calcium carbonate). Aside from the effect of inhomogeneity on the matrix correction, it was desirable to improve the calculation by calculating Si by difference and C by stoichiometry to Ca as follows :

```
Analysis of sample  un   2   seoj #3 random
Total Number of Data Points =   6            Number of "G" Data Points =   3
Average Beam Counts =   79092.                  Average Base Time =    10.00
Average Sample Z-bar =   11.480            Average ZAF Iterations =    2.333
Oxygen Calculated by Cation Stoichiometry and Included in  Matrix Correction
Element : Si  is Calculated by Difference from a Sum of 100.0 Weight Percent
Element : C   is Calculated Relative to Element Ca   at    1.000 to 1.0 Atom

Results in Weight Percent :

       O       Si      C       La      F       SUB

SPEC  51.526  39.743   1.134    .000    .000  92.403
SDEV   2.932  11.897   1.939    .000    .000

BGD:   MAN     MAN     MAN     MAN     MAN     MAN

       Fe      Al      Ca      Mg      Mn      S       SUM
 133    .054    .018    .012    .009    .000    .000 100.000
 134  10.696    .467  11.253    .010    .054    .000 100.000
 135    .119    .013    .083    .004    .000    .000 100.000

AVER   3.623    .166   3.782    .008    .018    .000 100.000
SDEV   6.126    .261   6.470    .003    .031    .000
SERR   3.537    .151   3.735    .002    .018    .000

KRAT   .0306   .0013   .0353   .0001   .0001   .0000
ZCOR  1.1992  1.2245  1.0981  1.3804   .9338   .8713
KRAW   .0451   .0029   .2072   .0001   .0002   .0000
P/B:   14.07    4.78   43.69    1.45    1.07     .92


Results in Elemental Mole Percents :

       O       Si      C       La      F

AVER  65.743  28.728   2.004    .000    .000
SDEV   1.571   7.841   3.430    .000    .000

       Fe      Al      Ca      Mg      Mn      S       SUM
 133    .019    .014    .006    .007    .000    .000 100.000
```

```
 134   4.069    .368   5.965    .009    .021    .000 100.000
 135    .043    .010    .041    .003    .000    .000 100.000

AVER   1.377    .130   2.004    .006    .007    .000 100.000
SDEV   2.332    .206   3.430    .003    .012    .000


Results in Oxide Weight Percents using Custom Cation Oxide Ratios :

       O       SiO2    CO2     La2O3   F

AVER    .000  85.024   4.153    .000    .000
SDEV    .000  25.452   7.104    .000    .000

       Fe2O3   Al2O3   CaO     MgO     MnO     SO3     SUM
 133    .077    .035    .017    .014    .000    .000 100.000
 134  15.293    .883  15.745    .017    .070    .000 100.000
 135    .170    .024    .116    .006    .000    .000 100.000

AVER   5.180    .314   5.292    .013    .023    .000 100.000
```

Note that Fe was recalculated as Fe_2O_3 and C was added relative to Ca by stoichiometry. This calculation can be applied in numerous other ways to a variety of analytical situations. Consider the example below. Here, the user decides to calculate CO_2 on the basis of moles of CaO, rather than by stoichiometry to oxygen.

```
Analysis of sample  st 141   set  1   dolomite (Harvard #105064)
Total Number of Data Points =   3            Number of "G" Data Points =   3
Average Beam Counts =    9102.                  Average Base Time =    10.00
Average Sample Z-bar =   10.875            Average BNA Iterations =    5.000
CO2   is Calculated Relative to Oxide CaO    at    2.000 to 1.0 Mole

Results in Weight Percent :

       CO2     SUB

SPEC  47.791  47.791
SDEV    .071

BGD:   MAN     MAN

       CaO     MgO     SUM
   9  30.501  21.735 100.109
  10  30.424  21.769  99.947
  11  30.420  22.135 100.301

AVER  30.448  21.880 100.119
SDEV    .046    .222
SERR    .026    .128

PUBL  30.560  21.880 100.186
%VAR   -.365   -.001
                  *

UNBE  1.0496  1.2242
KRAW   .5338  1.0000
P/B:     .00     .00


Results in Oxide Mole Percents :

       CO2

AVER  50.002
SDEV    .139
```

```
        CaO     MgO     SUM
   9  25.053  24.841 100.000
  10  25.028  24.917 100.000
  11  24.922  25.233 100.000

AVER  25.001  24.997 100.000
SDEV    .069    .208
```

Calculate Detection Limits and Homogeneity

The user may also select a calculation of the sample detection limits based on the standard counts and the unknown background counts and including the magnitude of the ZAF correction factor. The calculation is adapted from Love and Scott (1974). This detection limit calculation is useful in that it can be used even on inhomogeneous samples and can be quoted as the detection limit in weight percent for a single analysis line with a confidence of 99% (assumes 3 standard deviations above the background).

$$CDL = (ZAF)\frac{3}{I_S}\sqrt{I_B}\cdot 100$$

Where : ZAF is the ZAF correction factor for the sample matrix

I_S is the raw counts on the analytical (pure element) standard

I_B is the raw background counts on the unknown sample

After this, a rigorous calculation of analytical error also for single analysis lines, is performed based on the peak and background count rates also from Love and Scott (Love and Scott, 1974). The results of the calculation are displayed after multiplication by a factor of 100 to give a percent analytical error of the net count rate. This analytical error result can be compared to the percent relative standard deviation (%RSD) displayed in the analytical calculation (both are one standard deviation confidence levels). The analytical error calculation is as follows:

$$\varepsilon_{P-B} = \frac{\sqrt{\dfrac{N_P}{t_P^2} + \dfrac{N_B}{t_B^2}}}{\left| \dfrac{N_P}{t_P} - \dfrac{N_B}{t_B} \right|}$$

Where : N_P is the total peak counts

N_B is the total background counts

t_P is the peak count time

t_B is the background count time

A more comprehensive set of calculations for analytical statistics will also be performed. These statistics are based on equations adapted from "Scanning Electron Microscopy and X-Ray Microanalysis" by Goldstein, et. al. (Plenum Press, 1992 ed., 1981) p. 432 - 436. All calculations are expressed for various confidence intervals from 60 to 99 % confidence.

The calculations are based on the number of data points acquired in the sample and the measured standard deviation for each element. This is important because although x-ray counts theoretically have a standard deviation equal to square root of the mean, the actual standard deviation is usually larger due to variability of instrument drift, x-ray focusing errors, and x-ray production. The statistical calculations include :

1. The range of homogeneity in plus or minus weight percent.

$$W_{1-\alpha} = \pm C \, \frac{t_{n-1}^{1-\alpha}}{n^{1/2}} \, \frac{S_C}{\overline{N}}$$

2. The level of homogeneity in plus or minus percent of the concentration.

$$\pm \frac{W_{1-\alpha}}{C} = \pm \frac{(t_{n-1}^{1-\alpha}) S_C (100)}{n^{1/2} \overline{N}}$$

3. The trace element detection limit in weight percent.

$$C_{DL} = \frac{C_S}{\overline{N}_S - \overline{N}_{SB}} \, \frac{2^{1/2} \, (t_{n-1}^{1-\alpha}) \, S_C}{n^{1/2}}$$

4. The analytical sensitivity in weight percent.

$$\Delta C = C - C' \geq \frac{2^{1/2} \, C \, (t_{n-1}^{1-\alpha}) \, S_C}{n^{1/2} \, (\overline{N} - \overline{N}_B)}$$

Where :	C'	is the concentration to be compared with
	C	is the actual concentration in weight percent of the sample
	C_S	is the actual concentration in weight percent of the standard
	$t_{n-1}^{1-\alpha}$	is the Student t for a 1-α confidence and n-1 degrees of freedom
	n	is the number of data points acquired
	S_C	is the standard deviation of the measured values
	$\overline{N}$	is the average number of counts on the unknown
	$\overline{N}_B$	is the continuum background counts on the unknown
	$\overline{N}_S$	is the average number of counts on the standard
	$\overline{N}_{SB}$	is the continuum background counts on the standard

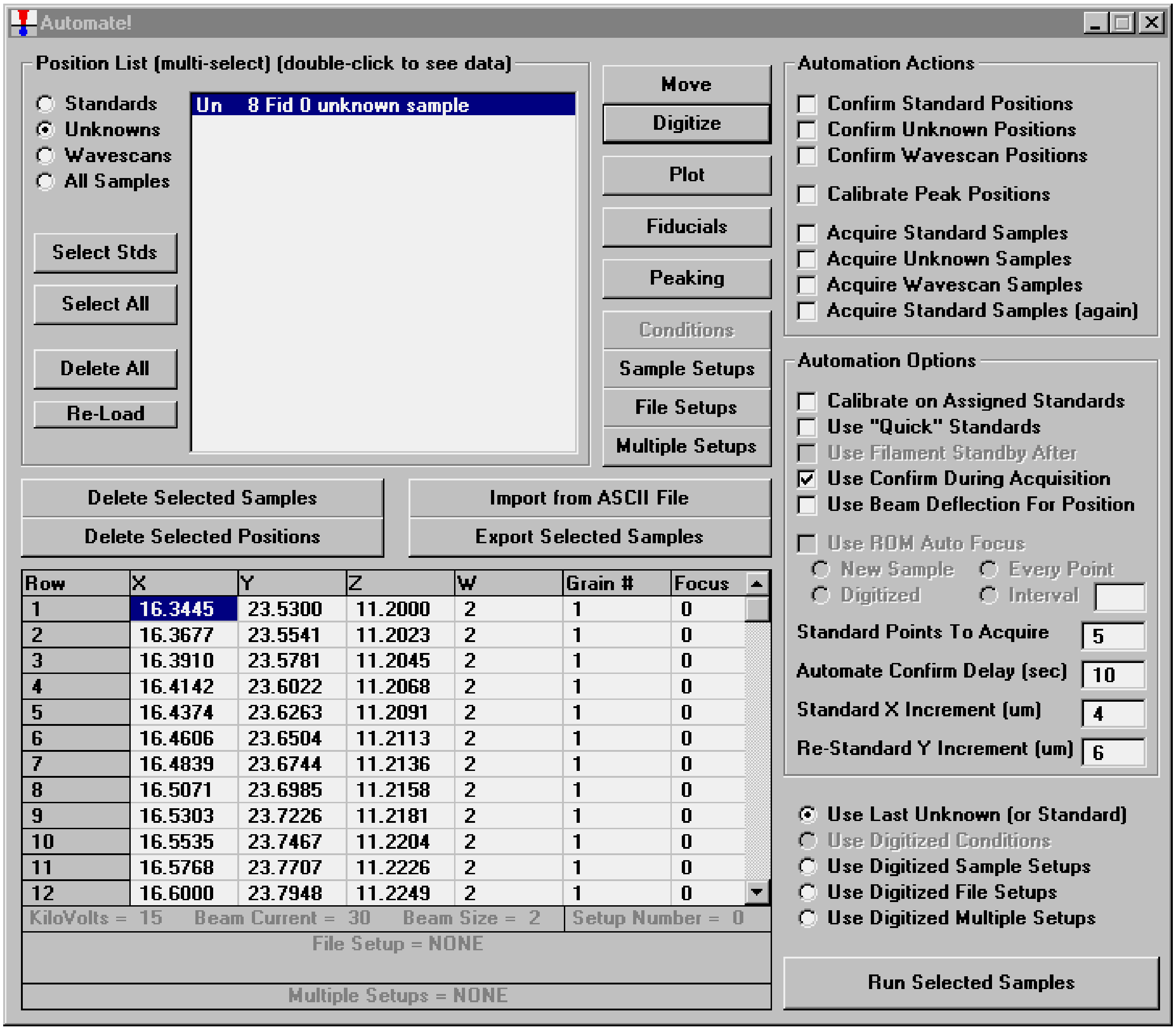
The homogeneity test merely compares the 99 % confidence range of homogeneity value with 1 % of the sample concentration for each element. If the range of homogeneity is less than 1 % of the sample concentration then the sample may be considered to be homogenous within 1 %.

The detection limit calculation here is intended only for use with homogenous samples since the calculation includes the actual standard deviation of the measured counts. This detection limit can, however, be quoted for the sample average and of course will improve as the number of data points acquired increases. Note that the homogenous sample detection limit calculation are ignored for those elements which occur as minor or major concentrations (> 1 %).

Conversely, the analytical sensitivity calculation is ignored for elements whose concentrations are present at less than 1%.

Automate!

The Automate modeless window provides for automation of standard, unknown and wavescan samples, position confirmation and peak centering. Also import, export and digitization of standard, unknown and wavescan coordinates.



Position List (multi-select) (double-click to see data)

○ Standards
● Unknowns
○ Wavescans
○ All Samples

Un 8 Fid 0 unknown sample

Automation Actions

☐ Confirm Standard Positions
☐ Confirm Unknown Positions
☐ Confirm Wavescan Positions
☐ Calibrate Peak Positions
☐ Acquire Standard Samples
☐ Acquire Unknown Samples
☐ Acquire Wavescan Samples
☐ Acquire Standard Samples (again)

Automation Options

☐ Calibrate on Assigned Standards
☐ Use "Quick" Standards
☐ Use Filament Standby After
☑ Use Confirm During Acquisition
☐ Use Beam Deflection For Position
☐ Use ROM Auto Focus
○ New Sample ○ Every Point
○ Digitized ○ Interval

Standard Points To Acquire 5
Automate Confirm Delay (sec) 10
Standard X Increment (um) 4
Re-Standard Y Increment (um) 6

● Use Last Unknown (or Standard)
○ Use Digitized Conditions
○ Use Digitized Sample Setups
○ Use Digitized File Setups
○ Use Digitized Multiple Setups

Row	X	Y	Z	W	Grain #	Focus
1	16.3445	23.5300	11.2000	2	1	0
2	16.3677	23.5541	11.2023	2	1	0
3	16.3910	23.5781	11.2045	2	1	0
4	16.4142	23.6022	11.2068	2	1	0
5	16.4374	23.6263	11.2091	2	1	0
6	16.4606	23.6504	11.2113	2	1	0
7	16.4839	23.6744	11.2136	2	1	0
8	16.5071	23.6985	11.2158	2	1	0
9	16.5303	23.7226	11.2181	2	1	0
10	16.5535	23.7467	11.2204	2	1	0
11	16.5768	23.7707	11.2226	2	1	0
12	16.6000	23.7948	11.2249	2	1	0

KiloVolts = 15 Beam Current = 30 Beam Size = 2 Setup Number = 0

File Setup = NONE

Multiple Setups = NONE

Select Stds

This button will select all standard position samples in the list that have been added to the run using the Standard | Add Standards To Run menu from the main menu.

This is useful for quickly selecting only those standard position samples of interest to the current run. Note that if a standard position sample is selected in the Position List and run as an automated acquisition, the program will automatically add the standard to the run.

Select All

This button will select all position samples shown in the position sample list. Normally this button is used for exporting all listed position samples to an ASCII .POS file.

Delete All

This button will delete all position samples shown in the position sample list. This is especially useful when starting a new run which will require a completely different position sample set from the previous user. New position samples are imported using the Import From ASCII File button described below.

Note that samples not shown in the list (unknowns or wavescans if the Standard option is selected) will **not** be deleted from the position database. To delete all samples in the database, first click the All Samples option and then the Delete All button.

Note that if all position samples that reference a fiducial calibration set are deleted, then the program will ask the user whether to delete the fiducial set that is no longer referenced by any position samples. Normally, the user should allow the program to delete unreferenced fiducial sets unless they plan to digitize additional position samples to the already confirmed fiducial set.

Re-Load

This button will force the position database to be reloaded. This is useful when the position data in the position database has been modified by another application.

Move

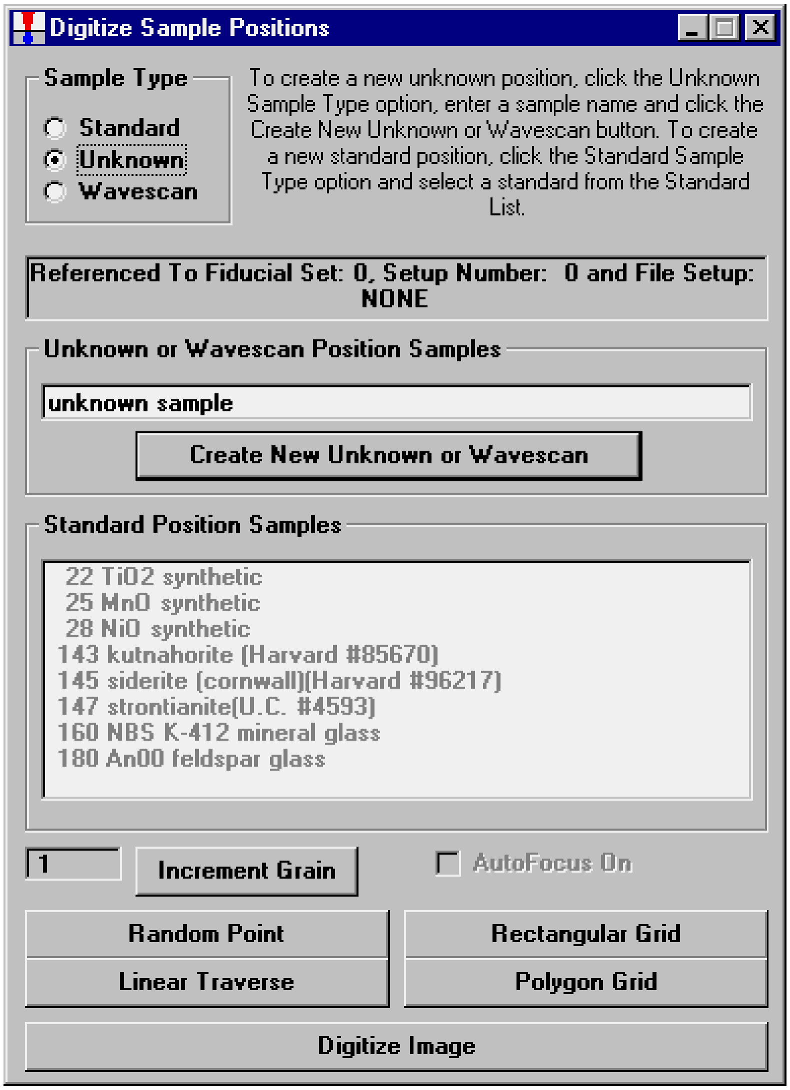
This button will recall the Move dialog for manual stage (and spectrometer and crystal) motion. The Move window is a modeless window which may remain open and accessible during the digitization of position samples.

From the Move dialog one may select to open the Stage Map or the Position Database dialogs, both of which are very useful for digitization of standards and unknown position samples.

Digitize

This button will load the Digitize dialog, where the user can digitize standard, unknown or wavescan positions based on random points, linear traverses or rectangular or polygon gridded areas.

In addition, the user may use the Digitize Image button to open a dialog that can be used to acquire an analog image for the purposes of digitizing sample coordinates by simply clicking on various locations on the image.



Sample Type
○ Standard
● Unknown
○ Wavescan

To create a new unknown position, click the Unknown Sample Type option, enter a sample name and click the Create New Unknown or Wavescan button. To create a new standard position, click the Standard Sample Type option and select a standard from the Standard List.

Referenced To Fiducial Set: 0, Setup Number: 0 and File Setup: NONE

Unknown or Wavescan Position Samples: unknown sample

Standard Position Samples
22 TiO2 synthetic
25 MnO synthetic
28 NiO synthetic
143 kutnahorite (Harvard #85670)
145 siderite (cornwall)(Harvard #96217)
147 strontianite(U.C. #4593)
160 NBS K-412 mineral glass
180 An00 feldspar glass

1

☐ AutoFocus On

To digitize standard positions, the standard to be digitized must first be added to the run using the Standard | Add Standards To Run menu.

To create a new standard position sample, first select the sample type as Standard and then simply click the desired standard from the list box. The standard position sample will automatically be created (in the Automate! position sample list) if it did not already exist. Normally a single random digitized position is sufficient for the digitization of a standard position. The program will automatically increment the X and Y positions for subsequent point acquisitions based on the increment fields in the Automate! window. However, if specific positions are digitized for a standard position sample, the program will use those positions for automated acquisitions.

To create unknown or wavescan position samples, first select the sample type as before and then enter the sample name in the sample name field. Finally, click the Create New Unknown Or Wavescan Position Sample button and the position sample will appear in the Automate! position sample list. Next proceed to digitize points using the random, traverse or grid buttons.

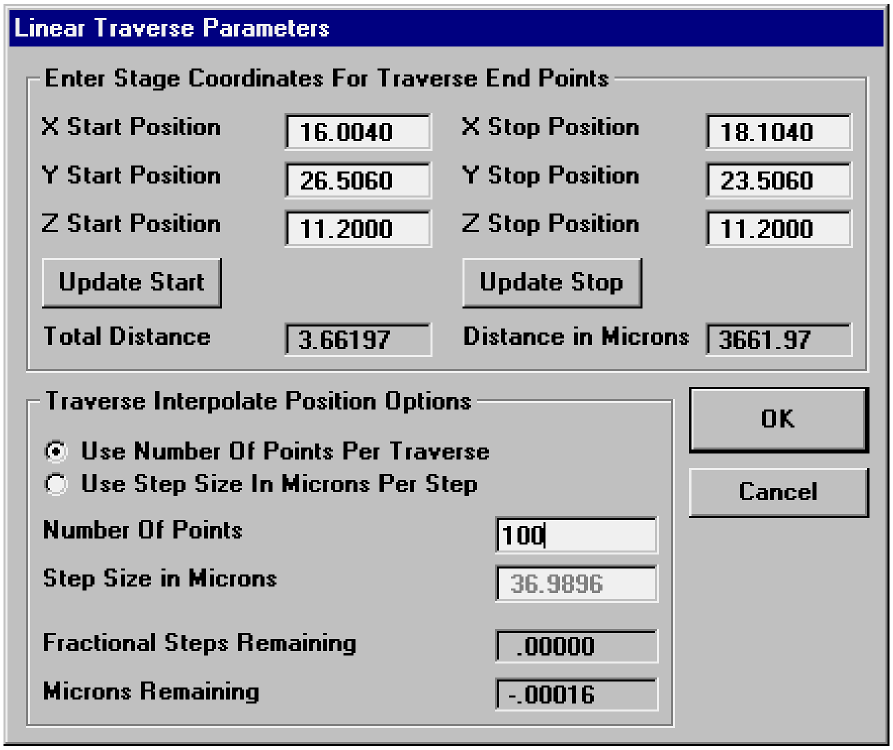
Random Point

To digitize a sample of random points, simply click the Random Points button. each time the button is clicked, it will add a digitized position based on the current position to the currently selected position sample.

Note that any number of random points can be digitized in a single position sample. At the time of acquisition the program will sub-divide them into appropriate sized samples using the keyword sample name "continued" for points in any subsequent samples.

Linear Traverse

The linear traverse dialog allows for digitizing a straight line series of sample positions based on two given end-points. The default start and stop positions are loaded with the current position, therefore it is best to move to the desired start or stop position before clicking the Linear Traverse button.



Linear Traverse Parameters

Enter Stage Coordinates For Traverse End Points

X Start Position 16.0040
X Stop Position 18.1040
Y Start Position 26.5060
Y Stop Position 23.5060
Z Start Position 11.2000
Z Stop Position 11.2000
Total Distance 3.66197
Distance in Microns 3661.97

Traverse Interpolate Position Options

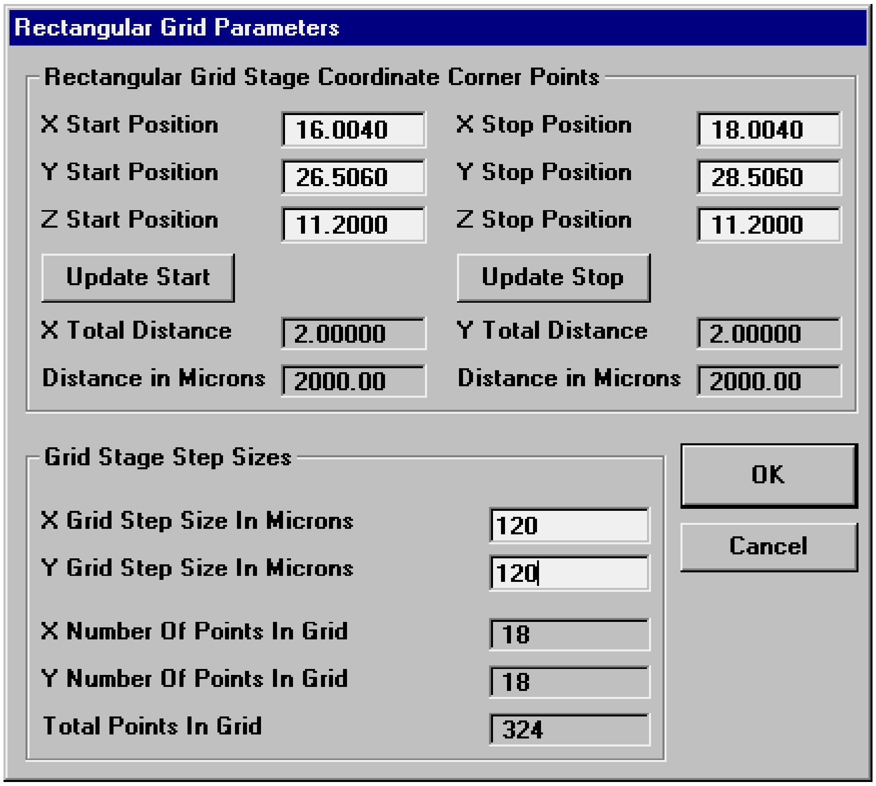
● Use Number Of Points Per Traverse
○ Use Step Size In Microns Per Step

Number Of Points 100
Step Size in Microns 36.9896
Fractional Steps Remaining .00000
Microns Remaining -.00016

Select either a specified number of points per traverse (including the end-points) or a specified distance between points.

Rectangular Grid

The Rectangular Grid dialog allows an offset grid of points to be digitized based on any two opposite corners. The grid spacing is calculated by the X and Y distance in microns specified by the user.



X Start Position 16.0040

Y Start Position 26.5060

Z Start Position 11.2000

X Stop Position 18.0040

Y Stop Position 28.5060

Z Stop Position 11.2000

X Total Distance 2.00000

Distance in Microns 2000.00

Y Total Distance 2.00000

Distance in Microns 2000.00

X Grid Step Size In Microns 120

Y Grid Step Size In Microns 120

X Number Of Points In Grid 18

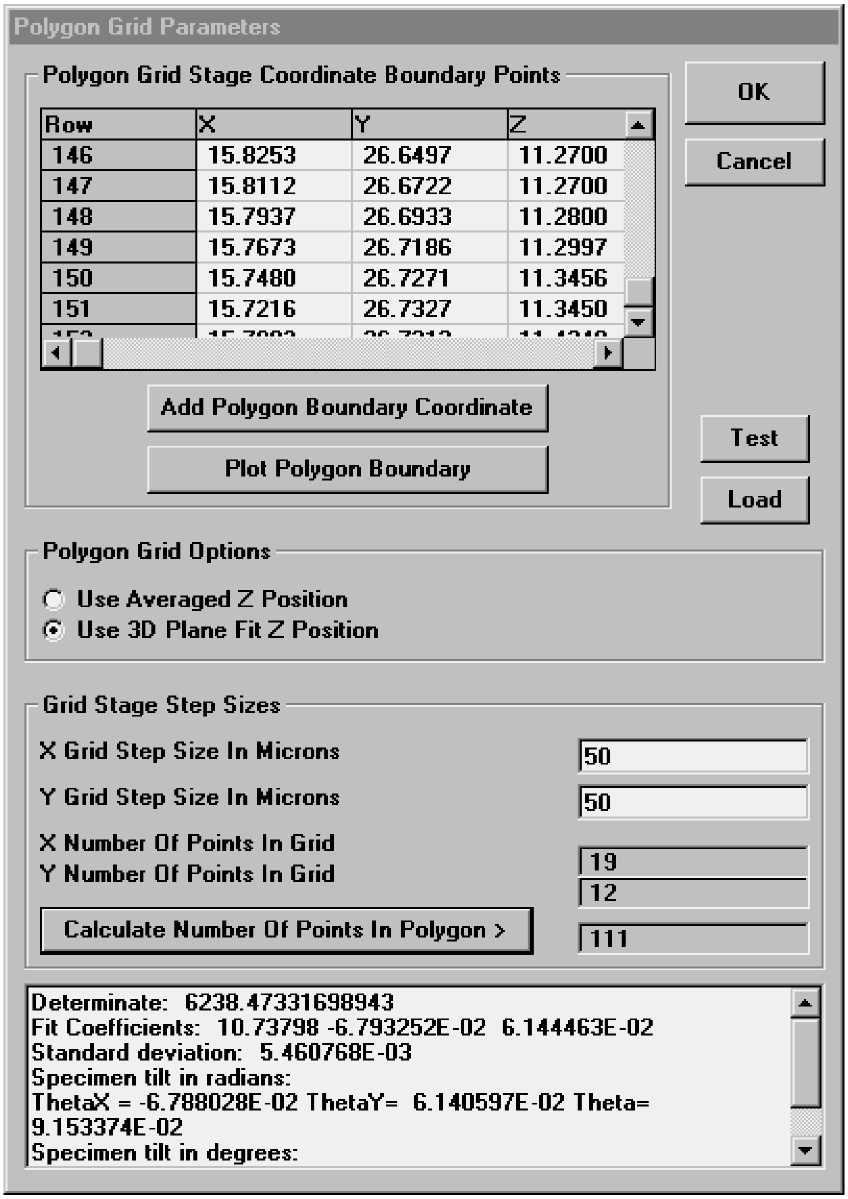
Y Number Of Points In Grid 18

Total Points In Grid 324

As the grid points are digitized, the positions are calculated in a "serpentine" manner to reduce stage motion to a minimum during acquisition.

Polygon Grid

The Polygon Grid dialog allows the user to digitize an offset grid of points which is delimited by an arbitrary boundary digitized by the user. This boundary may contain any number of points and is saved along with the grid points for use in exporting to a .BLN boundary file used to create SURFER 3-D surface and/or contour output..



Row	X	Y	Z
146	15.8253	26.6497	11.2700
147	15.8112	26.6722	11.2700
148	15.7937	26.6933	11.2800
149	15.7673	26.7186	11.2997
150	15.7480	26.7271	11.3456
151	15.7216	26.7327	11.3450

○ Use Averaged Z Position
● Use 3D Plane Fit Z Position

X Grid Step Size In Microns: 50
Y Grid Step Size In Microns: 50
X Number Of Points In Grid: 19
Y Number Of Points In Grid: 12
Calculate Number Of Points In Polygon >: 111

```
Determinate:  6238.47331698943
Fit Coefficients:  10.73798 -6.793252E-02  6.144463E-02
Standard deviation:  5.460768E-03
Specimen tilt in radians:
ThetaX = -6.788028E-02 ThetaY=  6.140597E-02 Theta=
9.153374E-02
Specimen tilt in degrees:
```

To create a boundary for the polygon grid, simply move the stage and click the Add Polygon Boundary Coordinate button for each position to be included in the boundary. Depending on the desired level of detail, any number of coordinates may be used to digitize the boundary feature.

The boundary can be plotted during the digitization process by clicking the Plot Polygon Boundary button and the first and last points are automatically assumed to be closed to complete the polygon boundary. Because the grid algorithm is quite robust, boundaries of any arbitrary shape may be digitized.

The Test button is only visible in demonstration mode and is used to load a default polygon to demonstrate the polygon gridding process.

The Load button is used to load a previously digitized set of polygon grid boundary coordinates. This is useful when the user decides that a different grid spacing is preferred but with the same digitized boundary. By using this feature, the user is not required to re-digitize the boundary coordinates. Typically, one should create a new position sample (with no positions) when loading a previously digitized polygon boundary for the purpose of changing the grid step sizes.

Two methods for determining the Z axis interpolated position are available. The first is based on the average Z position of all boundary positions, the second is a 3-D plane fit to the Z position of all boundary positions.

Adjust the number of points to be gridded during the digitization by entering X and Y grid step sizes in microns. For example, a 100 x 100 micron rectangle (4 boundary points) will produce approximately 100 analysis positions if a 10 micron spacing is used for both X and Y.

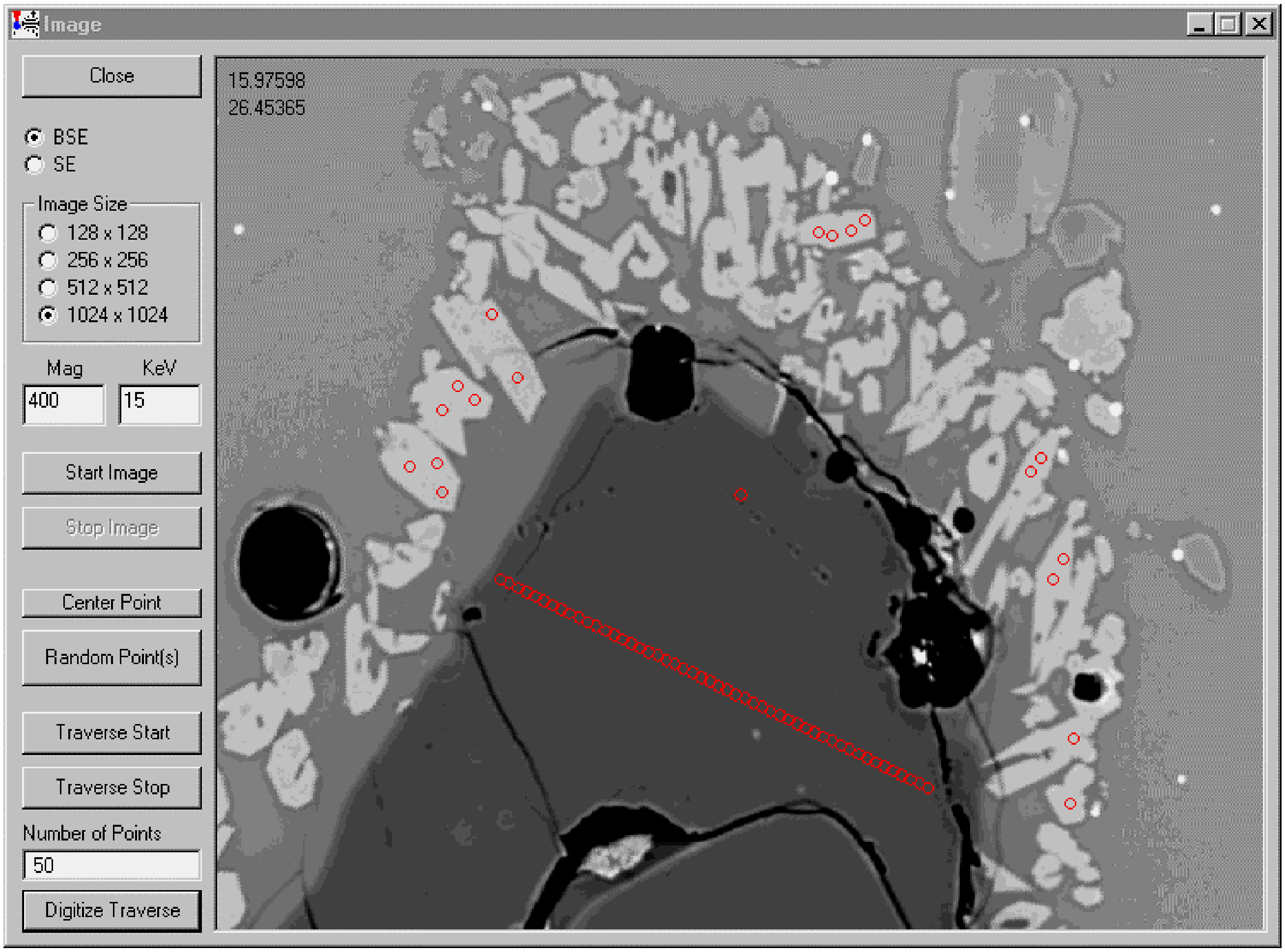
The program will prompt the user if the number of grid positions are excessively large by assuming a "rectangular" boundary. To see the actual number of grid points that will fit inside the digitized boundary, click the Calculate Number of Points in Polygon button to determine the actual number points that will be digitized.

Click OK when the ready to digitize the grid based on the polygon boundary coordinates and the X and Y grid step sizes. Click Cancel to not digitize any polygon grid positions and not save the boundary coordinates.

Digitize Image

This button is used to open a dialog that can be used to digitize stage positions based on an SE or BSE analog image supplied by the system. This feature can acquire images from either the MicroImage-32 software, or the Win30 A/D-D/A board directly. The imaging system requires calibration in order to associate stage coordinates to beam deflection positions before it can be utilized for digitizing. See the parameters defined in the PROBEWIN.INI file [Image] section.

Both random points and traverse coordinates may be saved to the position database by simply clicking the mouse on the displayed image. If the image is supplied by MicroAge, any type of X/Y registered analog image that MicroImage is capable of acquiring may be used for the purpose (beam, stage or mosaic beam/stage scan images included) at any magnification supported by the instrument, in the case of the WIN30 direct interface only beam scan images may be acquired.



Mag: 400

KeV: 15

Number of Points: 50

15.97598
26.45365

Use the Image Size options to select the image resolution, then click the analog source and click the Start Image button. The image acquisition will start. To halt the current image acquisition, click the Stop Image button. An image must be acquired before the user can begin to digitize position coordinates.

The Mag (magnification) and KeV (kilovolts) fields are used by some interfaces (SX50/51/100) to set the actual beam scan conditions for the image acquisition. For other interfaces the actual setting made to the instrument manually must be entered here so that the stage positions are correctly calculated.

The Center Point button can be used to digitize the first point of the position sample in the exact center of the beam scan area if desired.

Once an image is acquired, use the Random Point(s) button to digitize one or more random position coordinates by simply clicking the image at the desired location(s). The stage coordinates of each mouse click is stored in the position database for sample automation purposes.

Use the Traverse Start to indicate the start position of a linear traverse, the Traverse Stop button to indicate the stop position of the linear traverse and the Number of Points field to specify the number of points in the traverse. Click the Digitize Traverse button to actually digitize the traverse and store it in the position database.

Digitize Cluster (of Random Points)

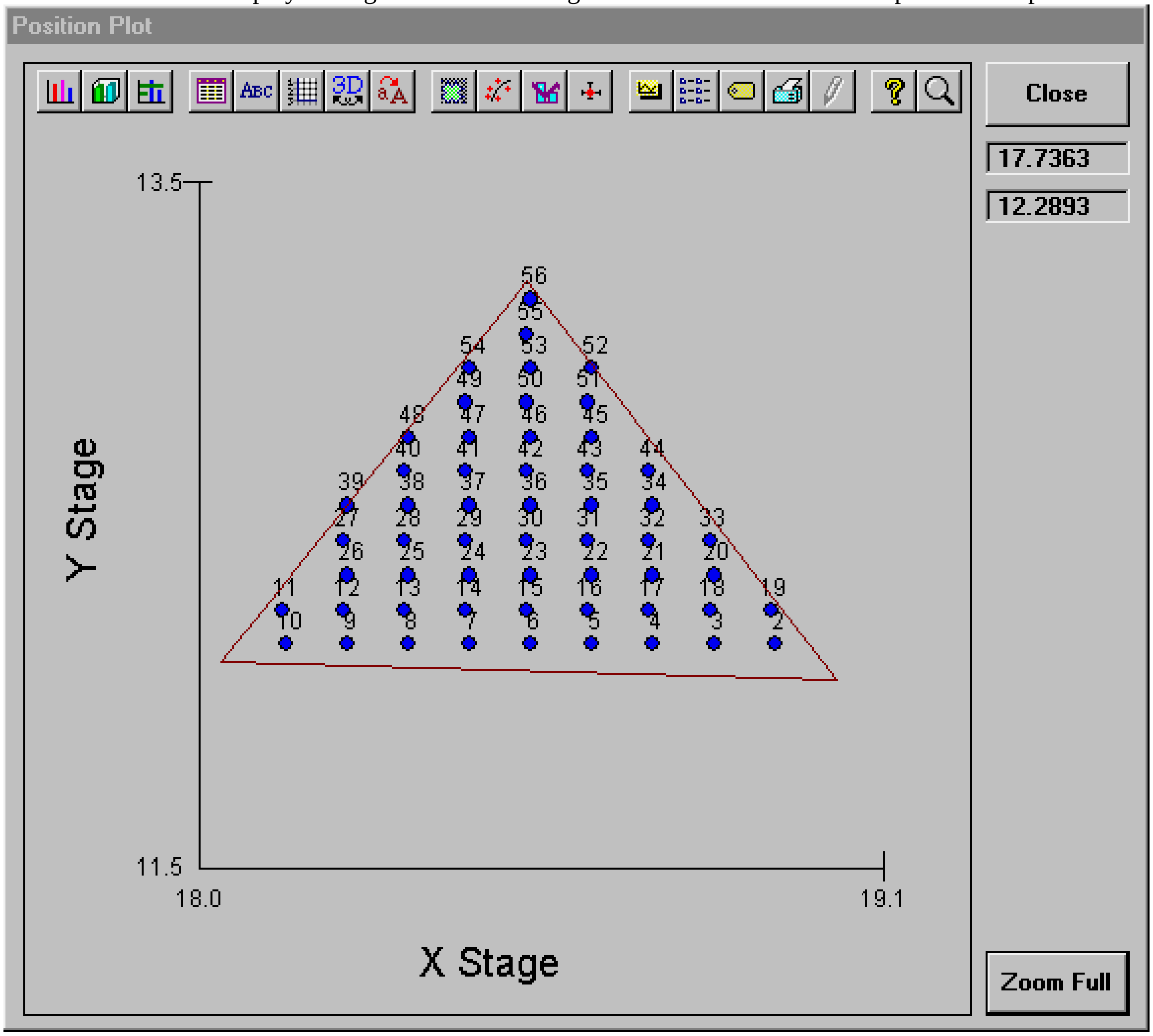
This dialog allows the user to define a circular or square areas of a given diameter or width and specify the number of randomly distributed positions within the area. There is also an option (by default) to save a polygon boundary of the area and the number of points to define the area (round area only).

In addition the user can specify the minimum distance between random points to prevent the beam from analyzing the same spot more than once. Note that if the minimum distance is too large or the cluster diameter or width is too small or the number of points is too large, the program may be unable to find enough evenly spaced coordinates.

This feature is useful when performing careful homogeneity tests when it is important to eliminate instrument drift and grid pattern artifacts. Note that the circular boundary creating when digitizing round areas can be re-used in the polygon gridding dialog using the Load button.

Plot

The Plot button will display the digitized X and Y stage coordinates for the selected position sample.



17.7363

12.2893

The example above shows a plot of a Polygon grid consisting of 55 points digitized using three boundary points.

If the position sample is a polygon gridded position sample then the digitized polygon boundary will also be displayed. The coordinates of the cursor are displayed as the cursor is moved across the plot area.

Fiducials

The Fiducials button will display the current digitized fiducial sets in the position database. To select a different fiducial set to reference subsequent digitized position to, simply select it from the list. In addition, this dialog will allow the user to modify, confirm, create new or delete fiducial sets in the position database.

Although standard positions are normally referenced to fiducial coordinates, it may also be desirable to reference unknown or wavescan coordinates to physical fiducial marks for the purposes of precisely relocating the acquired positions even after they have been removed and later re-loaded into the microprobe.

For this purpose, it is required that the sample mount have been previously scribed or otherwise marked to allow the user to locate 3 points that will be used for the fiducial coordinates. These fiducial coordinates must be permanent and easily re-located in the same order that they were defined to allow the program to calculate a transformation matrix when the sample is re-loaded later on. Once the positions have been referenced and digitized to a selected calibrated fiducial coordinate set, they can be exported to an ASCII file for subsequent use at another time.

See the section Import from ASCII File below for details on using the fiducial coordinates to transform digitized coordinates.

Peaking

Clicking the peaking button opens the peak center window. All currently analyzed elements are listed. The Peak Center method can be specified. A choice of Interval Halving, Parabolic and ROM based (if supported by the hardware) is available. The default peak center method can be specified from the PROBEWIN.INI file. With all methods the program will perform a peak to background measurement to determine if the peak is statistically significant enough to attempt a peak center.

> The **Interval Halving** method is the most accurate, but the slowest. It is based on a peak crawl that changes direction and halves the step size whenever the count intensity drops by more than a standard deviation.
>
> The **Parabolic** method peak center uses a moving three point fit on the peak top that is fit to a 2nd order polynomial. The centroid of the parabola is returned as the new peak center. This method is the fastest, if the spectrometer position is already close to the actual on-peak position.
>
> The **ROM Based** method peak center is available if the hardware interface supports a ROM peak center. The actual method used for the ROM Based peak center depends on the microprobe hardware.

The program will automatically select elements in the element list that were not peaked in the previous peak center when the window is re-opened again. Note that the element list is multi-select so that any combination of elements can be peaked. If more than one element on the same spectrometer was selected, then the program will automatically peak center them one at a time. The Plot Selected Peak Center button may be used to display the last peak center count-position data for the selected element.

An option for performing a spectrometer prescan before the peak center and an option for moving to the selected element on-peaks when closing the dialog are possible.

The move to on-peak option is useful for moving a spectrometer (and setting the PHA conditions) to it's on-peak position for imaging purposes. Remember : the elements to peak center (or move to on-peaks) must be highlighted before closing the dialog.

Spectrometer Peak Center Scan

All spectrometer peak center procedures may be prefaced with a spectrometer peak scan to allow the user to manually specify the nominal peak center position. This spectrometer peak scan is performed using the number of peak scan steps as defined in the Peak/Scan Options dialog. The count time for each peak scan point is based on the peak count time (divided by 4) as defined in the Count Times dialog.

Initial Peak-To-Background Measurement

The program always performs a peak-to-background and peak intensity check before the actual peak center procedure. The peak-to-background is calculated by measuring the count rate at the nominal peak position and the current off-peak positions using the peak count time for each position. The measured count data must meet the required minimum peak-to-background and minimum peak count intensity as defined in the Peak/Scan Options dialog, before performing the actual peak center procedure.

If performing the peak center from the Automate! dialog, the program will not proceed with acquisition of sample data if one or more spectrometer fails to complete a peak center procedure for any reason.

Spectrometer Peak Center Procedures

Three peak center procedures are used by Probe for Windows. They are the Interval Halving, the Parabolic Fit and the ROM based peak center procedures.

Interval Halving

Interval halving is a peak center procedure based on a peak crawl starting at the nominal peak position. The step size of the peak crawl is halved and the direction reversed whenever the count rate drops by more than a single standard deviation (square root of the intensity). The interval halving procedure is slow but extremely precise.

The initial peak step interval is always in a positive direction and the peak interval size is based on the Peaking Start Size as defined in the Peak/Scan Options dialog. Note that the default peaking start and stop sizes from the SCALERS.DAT file are modified by the following expression :

$$S_{actualstart} = S_{start} \bullet \sqrt[3]{\frac{2d_{OnPeak}}{2d_{LiF\,200}}}\,(*2.0)\,\sqrt{\frac{P_{LoLim} + ABS\,(P_{HiLim} - P_{LoLim})}{P_{OnPeak}}}$$

Where :

$S_{actualstart}$	is the actual calculated step size
S_{start}	is the spectrometer default start or stop size
P_{HiLim}	is the spectrometer high limit
P_{LoLim}	is the spectrometer low limit
P_{OnPeak}	is the spectrometer on-peak position
$2\,d_{OnPeak}$	is the 2d spacing of the analyzing crystal
$2\,d_{LiF\,200}$	is the 2d spacing for LiF (4.0267)

From this expression one can see that the actual start and stop sizes are unchanged at the spectrometer high limit when an LiF crystal is used, and are increased for lower spectrometer positions and larger crystal 2d spacings. The calculated start and stop sizes are also doubled when the crystal 2d spacing is larger than 30 angstroms (LDE crystals).

When the interval step size has been halved to a value less than the peaking stop size, the spectrometer is considered to be peak centered and the current position is saved as the new peak position.

If the number of intervals measured exceeds the "Maximum Peaking Cycles" as defined in the Peak/Scan Option dialog, the program terminates the peak center procedure with an error.

Parabolic Fit

The parabolic peak center procedure is based on a moving three point triplet that attempts to fit an inverted parabola. The three positions measured are the middle, high and low positions. The middle position is initially the nominal peak position and the high and low positions are offset from the middle position based on the value of the Peaking Start Size, which is calculated using the process described in the interval peaking procedure above. Ideally the Peaking Start Size calculation should be attempt to yield an offset which places the high and low positions at a position which produces approximately 1/2 the maximum intensity of the peak, for a more precise parabolic fit.

The peak center procedure then determines if the high and low position intensities are at least one standard deviation less than the middle position intensity. If the three intensities meet this criteria, the program calculates the centroid of a parabola based on the count-position data triplet. The results of the centroid peak fit can be viewed by placing the program into "Debug Mode" under the Output menu.

If the data cannot be fit to a parabola, the middle position is shifted by an amount equal to 1/2 of the Peaking Start Size and the middle, high and low position intensities are measured again.

If the number of count-position data triplets measured exceeds the "Maximum Peaking Cycles" as defined in the Peak/Scan Option dialog, the program terminates the peak center procedure with an error.

ROM Peaking

The ROM peaking procedure is available for those interfaces that support an integrated ROM based peaking procedure. These are the Sesame, Tracor, SX100 and SX50/SX51 interface types.

The actual peaking procedure utilized by each interface is somewhat different but usually based on a parabolic fit of some variety. The program passes the "Peaking Start Size" to each interface specific ROM Peaking routine where is it modified if necessary.

JEOL 8900 Direct (InterfaceType=2)- The "Peaking Start Size" is not used. Instead a peak parameter of 1 (fine scan) is always used.

Sesame (InterfaceType=3)- The "Peaking Start Size" is not used by the Sesame interface. The function simply sends the "PEAK" command for the specified motor, at the current position.

Tracor (InterfaceType=4)- The Tracor peak center procedure uses a two step peak center (an initial peak center based on the "Peaking Start Size" and a final peak center based on the "Peaking Stop Size"). The total number of steps is hard-coded since the PAC cannot update this system parameter while another spectrometer is being peaked. The number of TRACOR motor steps per peak scan point is calculated from the number of motor steps per l-unit conversion factor (from the MOTORS.DAT file), the "Peaking Start Size", (or) the "Peaking Stop Size" and the number of peak scan steps (hard-coded to 40). The following expression is used to calculate this value, and assumes a TRACOR peak scan width equal to ten times the Peaking Start Size :

$$sp = M * P * 10 / N$$

where		
	sp	is the Tracor ROM peak center scan motor steps per point
	M	is the motor steps to units conversion factor (from MOTORS.DAT)
	P	is the Peaking Start Size (initial) or Peaking Stop Size (final)
	N	is the number of peak scan steps (hard-coded to 40)

The Tracor step count time is calculated from the Peaking Count Time (divided by four). Note that the PAC Crystal Factor variable must be set to 1 on all spectrometer-crystals for this procedure to perform correctly.

After the initial Tracor peak center has completed, the procedure moves the motor position to the calculated centroid returned from the PAC, then a final Tracor peak center is performed, before the new peak intensity is measured.

SX100 (InterfaceType=5)- The "Peaking Start Size" is modified to produce a number between 0 and 4 by utilizing the following expression :

$$pw = (M_{high} - M_{Low}) * P / 3000000$$

where pw = is the SX100 peak center width
M_{high} = is the motor high limit
M_{low} = is the motor low limit
P = is the Peaking Start Size

SX50 (InterfaceType=6)- The "Peaking Start Size" is modified to produce a number between 0 and 4 by utilizing the following expression.

$$pw = (M_{high} - M_{Low}) * P / 3000000$$

where pw = is the SX50 peak center width
M_{high} = is the motor high limit
M_{low} = is the motor low limit
P = is the Peaking Start Size

If the SX50 peak center width is less than or equal to 2, the SX50 driver sends the "PE" command for the specified motor. Otherwise, if the SX50 peak center width is greater or equal to 3, the SX50 driver sends the "PE WIDE" command instead.

JEOL 8900 Rlogin (InterfaceType=7)- The "Peaking Start Size" is not used. Instead a peak parameter of 1 (fine scan) is always used.

Final Peak-To-Background Measurement

After a successful peak center procedure the peak-to-background is re-calculated by simply measuring the on-peak count rate at the new peak position. This result can be compared with the initial peak-to-background to evaluate the peak center procedure performance.

Conditions

Although Probe for Windows normally acquires automated samples based on the last unknown sample conditions, the program will support the automatic acquisition of samples under various analytical conditions (if the interface hardware supports it).

There are two different options for analytical conditions, the first is using specified values for kilovolts, beam current and beam size, the second is using a specified string to indicate a column condition that is stored internally in the instrument. In this latter case, because the actual value of the operating voltage (kilovolts) in the column condition string is ambiguous, the user should be sure that the single value fields are also correctly specified based on the column condition string specified.

For example, the SX50 supports the use of internally stored column conditions that are indicated by a four character string. Therefore, if the column condition option is selected, and the column condition string is specified as "hv15" (indicating a column condition at 15 keV), then the user should also be sure that the kilovolts field is correctly specified as 15 keV. Failure to properly specify the kilovolts field when using column condition strings will cause the ZAF or Phi-Rho-Z matrix corrections to be inaccurate.

For accurate documentation purposes it is also preferred that the beam current and beam size fields are also properly specified when using column condition strings. If, as in the above example, the "hv15" column condition string also includes a beam current of 10 nA and a beam size of 10 um, then those values should also be specified in the single values fields even though are not actually used in the acquisition procedure. This maintains the accuracy of the documented sample conditions.

By using either the analytical conditions and column condition methods, it is possible to automatically acquire a number of digitized positions using a different kilovolts/beam current/beam size values. This is useful for many purposes, for example, automated thin film analysis, where the acquisition of k-ratios at various operating voltages can improve the analysis considerably.

To utilize this acquisition type, be sure to select the Use Digitized Sample Conditions option before clicking the Run Selected Samples button.

Sample Setups

Although Probe for Windows normally acquires automated samples based on the last unknown sample conditions, the program will support the acquisition of samples based on previously saved sample setups.

This is especially useful when the need to acquire data on samples with a variety of elements are present. For example, there may be a number of quite different phases present that each require a more or less different set of analyzed elements. By previously creating a number of sample setups and saving them, it is possible to have the program automatically acquire a different set of analyzed elements by referencing the various available sample setups during the digitization process.

Sample setups may be created from the Acquire! window by first starting a new sample and then modifying the sample setup to the desired configuration. Then use the Save Sample Setup button from either the Elements/Cations window (accessed from the Analyze! window) to create a pointer to the sample setup for use later on during the creation of new analyzed samples or the digitizing of position samples.

To utilize this automated acquisition method, be sure to select the Use Digitized Sample Setups option before clicking the Run Selected Samples button.

File Setups

Although Probe for Windows normally acquires automated samples based on the last unknown sample conditions, the program will support the acquisition of samples based on previously created file setups. These file setups are simply Probe database files that contain a specific sample setup and may or may not also contain standardization count intensity data.

This is especially useful when the need to acquire data on samples with a variety of elements are present. For example, there may be a number of quite different phases present that each require a more or less different set of analyzed elements. By previously creating a number of file setups, it is possible to have the program automatically acquire a different set of analyzed elements by referencing the various available file setups during the digitization process. Note that the specified file setup must be available to the program when the automation is run or an error will occur.

To utilize this automated acquisition method, be sure to select the Use Digitized File Setups option before clicking the Run Selected Samples button.

Multiple Setups

This button is used to specify multiple sample setups for the digitized position samples. All considerations for Samples Setups apply for Multiple Setups (see Sample Setups above). To create sample setups for digitizing position samples, use the "Elements/Cations" button in the Analyze! Window.

This procedure is affected by the state of the "Combine Multiple Sample Setups" checkbox in the Automation Options frame. If this checkbox is not checked, then a separate sample, based on each multiple setup, will be acquired for each position sample. This is particularly useful for situations where the same elements need to be measured several times with different operating voltages, for example, in thin film analysis, when it is necessary to acquire different voltages on the sample position. Here one would specify two or more sample setups that have the same elements but different kilovolt operating conditions.

If the checkbox is checked, then all specified multiple setups will be combined into a single combined condition sample for the position sample, and the software will adjust the column condition as necessary to acquire the elements. When more than one sample setup is specified by the user, the automation routine will acquire the position sample for each sample setup that is specified. This is useful in several situations, for example, when performing major and trace element acquisition it may be desired to acquire the major elements at one set of sample conditions and the trace elements using a sample setup with different sample conditions (kilovolts, beam current, etc.).

In the former case of major and trace element analysis, each sample setup acquired will create a different analytical sample. To combine the elements into a single sample for quantitative analysis, simply select all the samples that are to be combined and click the "Analyze Selected Samples (Combined)" button in the Analyze! Window.

Delete Selected Samples

This button will delete the currently selected position samples from the position database. If all position samples that reference a fiducial set are deleted, the program will ask whether to delete the fiducial set from the position database also. Normally one should allow the program to delete unreferenced fiducial sets to avoid confusion.

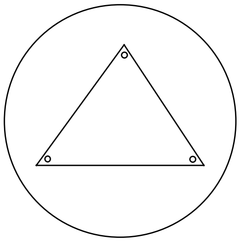
Delete Selected Positions

This button will delete the position coordinates that are currently selected in the position grid display. This option will only delete contiguous position coordinates listed in the position grid.

Import From ASCII File

The Import button allows the user to import an ASCII file of sample positions for automated acquisition. Standard, unknown and wavescan coordinates can be imported separately or in a single file.

The program will ask the user whether the imported coordinates should be transformed using a fiducial based matrix transformation. The fiducial positions on the standard or unknown sample mount should generally be laid out in an easy to re-locate pattern. Generally it is preferred to place the fiducials marks in the corners of a scribed triangle on the mount surface as shown here :



The fiducial marks should be deep enough to withstand repeated re-polishing but small enough to be precise for re-location of the digitized coordinates.

A sample import file of two unknown samples and corresponding positions is shown here. Note that a new standard, unknown or wavescan sample is created during the automation process, if the sample number changes (for standards), or if the sample name changes (for unknowns or wavescans).

Special note: there are two varieties of formats (type = 1 or type = 2) used in position import and export files. The format of the position import and export file is specified by the PositionImportExportFileType parameter in the PROBEWIN.INI file. If the parameter is not specified in the INI file then the default of type = 1 is used.

The first format is the original import/export format (type = 1) and contains the fields shown in the example below without the autofocus flag and analytical setup number fields shown in parentheses at the end of the position lines.

The second format is a newer import/export format (type = 2) which contains two additional integer fields and an additional string field for each position lines. These additional integer fields are used for an auto focus flag and an analytical setup number. The analytical setup number can be used by Probe for Windows to specify which previously saved sample setup is to be used when acquiring data for the position sample. Note that all positions in a single position sample must use the same setup number. If more than one setup number is specified within a position sample, the program will use the last analytical setup specified. If the setup number specified is not available in the current Probe for Windows run, then the program will base the automated acquisition on the last unknown sample. The autofocus flag is used to define a random autofocus (if supported by the hardware interface) for each position. If this autofocus flag is zero, then no autofocus is attempted, if the flag is -1 or 1 then an autofocus will be attempted at that position. The adjustment from the auto-focus will be applied to all positions in that position sample.

The additional string field is used to specify a file setup name that can be used for automated designation of a file setup from an existing Probe for Windows data file. The specified Probe for Windows data file must reside in the Probe for Windows application directory (usually C:\Program Files\Probe for Windows).

The importing positions with a pre-defined file setups, analytical setup numbers (and autofocus flag) are useful when digitizing position samples off-line on an optical microscope using program STAGE. However, it is important when digitizing off-line to ensure that the setup number specified during the digitizing process will eventually correspond to the correct setup number created in the Probe for Windows run in which the digitized position samples will be automatically acquired. Also that the specified file setup data file exists and is updated with valid standardization data.

```
0.0, 0.0, 0.0, 0.0
0.0, 0.0, 0.0, 0.0
0.0, 0.0, 0.0, 0.0
2,     1,     "metallic phase #1", 15.234, 18.12, 10.873, 1.0, 1, (1, 4, "")
2,     1,     "metallic phase #1", 15.547, 18.43, 10.873, 1.0, 1, (0, 4, "")
2,     1,     "metallic phase #1", 15.698, 18.56, 10.873, 1.0, 1, (0, 4, "")
2,     2,     "Si3N4 ceramic matrix",15.747, 18.34, 10.873, 1.0, 1, (1, 7, "")
2,     2,     "Si3N4 ceramic matrix",15.747, 18.34, 10.873, 1.0, 1, (0, 7, "")
2,     2,     "Si3N4 ceramic matrix",15.747, 18.34, 10.873, 1.0, 1, (0, 7, "")
```

The format of these standard position import files is described below. Note that the first three lines are always used to define the coordinates of the 3 physical fiducial marks used for transformation of pre-digitized standard mounts. All parameters are comma, space or tab delimited.

Following the first 3 lines which define the x, y, z, and w coordinates for the three fiducial marks (used for transformation of pre-digitized standard mounts), each position line has the following format :

- the first column contains the sample type (1 = standard, 2 = unknown, 3 = wavescan)

- the second column contains the sample number, which is the standard number for a standard as defined in the STANDARD.MDB database

- the third column is the sample name, which is optional for standard position samples and must be enclosed in double quotes

- the fourth, fifth, sixth and seventh columns contains the x, y, z and w coordinate positions. The w motor position is only used for JEOL 733 microprobes

- the eighth column value is the grain number, which is used for automatically blanking the beam between successive points if different from the preceding point

- the ninth column value (only if PositionImportExportFileType = 2) is the autofocus, which is used for automatically performing an auto focus for each position (1= every sample, 2=every point, 3=digitized, 4=interval).

- the tenth column value (only if PositionImportExportFileType = 2) is the analytical setup number, which is used for automatically loading a previously created sample setup in a Probe for Windows data file. If the specified analytical setup number is not found, the program will load a sample setup based on the last unknown sample in the run.

- the eleventh column value (only if PositionImportExportFileType = 2) is the analytical file setup name. If the specified file setup name is not found, an error is generated.

Note that standard coordinates can be in any order and all positions of a single standard will be appended to a single standard sample automatically. However, if a standard position sample already exists, the program will ask if the user wants to overwrite the existing standard coordinate data.

If no fiducial transformation of the imported positions is required, simply click "No" when asked whether to perform the fiducial transformation.

For unknown and wavescan positions these fiducial positions may used for transformation of sample positions digitized on an optical microscope equipped with a digitizing stage using a different coordinate system than the microprobe stage. All linear transformations in scale, rotation and translation are handled. Although the use of three fiducial coordinates means that the procedure is capable of a three dimensional transformation (using x, y and z), the z coordinate transformation may be ignored, if desired by the user, for unknown or wavescan position transforms. The w coordinate is for reference purposes only.

Export Selected Samples

This button can be used to export selected position samples to an ASCII file. This operation is intended primarily for saving digitized standard positions to the STDPOS*.POS file names defined in the PROBEWIN.INI file, although unknown and wavescan positions can also be exported to ASCII files.

If the selected position samples were referenced to fiducials, then the program will also export the fiducial positions for subsequent fiducial coordinates transformation at another time.

Automation Actions

The automation "actions" desired to be run are to be selected here. The program will execute each automation action for all selected samples in the order listed. Any combination of automation actions may be selected by the user.

Use the Confirm actions to simply confirm the position coordinates of the selected samples and the Acquire actions to actually acquire the data for the selected samples. To confirm positions and/or acquire data for both standards and unknown position samples, select the All samples option and select the standards and unknowns desired. If a confirm action was selected, the program will ignore the setting of the Use Confirm During Acquisition for the confirmation of positions.

Use the "Acquire Standard Samples (again)" if it is desired to run the standards AFTER the unknown or wavescan samples have been run.

If the Peak Center action was selected, the program will cease the automation process if one or more of the elements selected for peaking did not peak properly rather than continue any selected acquisition actions.

Automation Options

The following automation options are supported by Probe for Windows. Some options may not be available depending on the interface type and microprobe hardware.

Calibrate On Assigned Standards

This option forces the program to only attempt a peak center on a standard position sample if the standard is assigned as the primary standard for that element. If the element has no assigned standard, then the program will attempt to assign one automatically based on the highest concentration of the element present among the standards in the run. To add additional standards to the run use the Standard | Add Standards To Run menu.

It is a good idea to check the standard assignments in the Analyze! window before running a peak center with this option selected to insure that the correct standard position samples are selected for peak centering.

Use "Quick" Standards

Although it is generally recommended to acquire a complete analysis (acquire all elements based on the last unknown sample setup) for all selected standards, Probe for Windows includes a feature which allows the acquisition of "quick" standardizations to save time. In this mode the program will, for each standard acquisition, acquire only those elements that are actually assigned to that standard. This includes not only the primary standard assignments and the interference assignments (see the Standard Assignments button in the Analyze! Window), but also any MAN (mean atomic number) standard assignments for background fitting (See the MAN Fits menu under the Analytical menu).

Considerations in the Use of "Quick" Standards

Although the use of "quick" standardizations can save considerable time, it is important to remember, that a complete acquisition (and hence analysis) of a standard enables the analyst to note the accuracy of secondary standard elements and to observe on-peak and off-peak interferences. In addition, the complete acquisition of data on standards used for the quantitative interference corrections is essential for this feature to work correctly.

However, sometimes for well characterized samples, it is feasible to acquire standard data only for those channels that are used as the assigned standard element for that channel. In these "quick" standardizations, Probe for Windows will skip elements that do not contain any assigned elements for that standard.

In a typical run, one would normally use acquire standard samples without the use of the "Quick" standards option. The resulting data can then be checked for interferences and background anomalies. Once these concerns have been addressed, then subsequent standardizations can be acquired using the "quick" standards option to save time.

Use of "Quick" Standards

"Quick" Standards can only be acquired from the Automate! window and are based on the standard assignments of the last unknown (or last standard, if there are no unknowns in the run) as long as the Use Last Unknown (or Standard) option is selected. If another option is selected, for example, the Use Digitized Sample Setups option, then the program will based the "quick" standard on the standard assignments of the digitized sample setup.

The program will check the primary standard, interference, and MAN standard assignments to determine whether an element should be acquired for a quick standard. If the an element in the sample is actually utilized by the "quick" standard being acquired, then the program will retain it for the acquisition of intensities, otherwise the element will be skipped during the acquisition. Note that if no elements in the sample for a standard are assigned for any of these purposes (a true secondary standard), the program will acquire all the elements in the current sample setup for that standard.

Note that the program may warn the user that the acquisition order has been changed to user defined to internally facilitate the acquisition of "quick" standards. However, the program will not change the actual order that the elements are being acquired.

Use Filament Standby After

This option (if supported by the hardware interface) is used to perform a filament shutdown after the automation actions have been completed. This is most useful for extended acquisitions overnight or over the weekend when it is desirable that the filament life be conserved as much as possible.

Use Confirm During Acquisition

This option will allow the user to perform a stage X, Y and Z adjustment just before the sample data is acquired on the first point of each position sample. The default confirm delay time is specified in the PROBEWIN.INI file. This option is ignored for Confirm automation actions.

Use Beam Deflection For Position

This option is available if an imaging interface is supported by the hardware interface (see the ImageInterfacePresent and ImageInterfaceType parameters in PROBEWIN.INI file). If this option is unchecked then normal stage motion is used to position the sample for acquisition. If this option is checked, then the program will utilize beam deflection for positioning the sample (beam position) for acquisition. This option affects only unknown and wavescan position samples (not standards).

This option is primarily intended for situations where very small beam diameters (low overvoltage) are used for precise positioning and acquisition at scales finer than the stage mechanism is capable of. Note that care must be taken to digitize positions that are all within the range of the current beam scan parameters.

Confirm All Positions In Sample

This option forces the program to move to EACH position in the selected position sample and allow the user to confirm the coordinates of that single point. This applies both to the explicit confirm action and to acquisition actions when the Use Confirm During Acquisition option is checked.

Note that even if this option is selected, only the first point in a position sample will be used for a peak calibration procedure. In addition, any coordinates for a standard position sample that are "automatically generated" during the acquisition process will be ignored for the explicit confirm action even if the Confirm All Positions In Sample option is checked.

Combine Multiple Sample Setups

This option allows the user to either run, for each position sample, each specified multiple sample setup as a separate sample or as a single combined sample. This option only affects position samples that have been assigned multiple sample setups.

Specifically, if this checkbox is not checked a separate sample based on each setup will be acquired for each position sample. If it is checked then all specified multiple setups will be combined into a combined condition sample and the software will adjust the column condition as necessary to acquire the elements.

Multiple setups may be sample setups that are single conditions or even multiple conditions sample setups themselves. Certain restrictions apply for combined sample setups, for example, only wavescan samples can have the same element more than once in a single combined sample.

Use ROM Auto Focus

This option (if supported by the hardware interface) is used to perform an automatic ROM based automatic focus on the sample surface. This option supports the following types of auto focus during an automated acquisition:

1 = auto focus on a new position sample only
2 = auto focus on every point in each sample
3 = auto focus using the digitized auto focus flags (or imported from a .POS file)
4 = auto focus using the specified auto focus interval

Standard Points To Acquire

This parameter specifies the number of standard point acquisitions to acquire on each standard. If fewer than this many points are actually digitized in the position database, the program will automatically increment the X stage axis by the amount specified in the Standard X Increment field.

Since Probe for Windows automatically uses the average of the sample count data for the calibration of quantitative intensities, it is recommended to acquire at least 5 to 15 points on each standard for accurate quantitative analysis.

Automate Confirm Delay

This is the time in seconds that the program will utilize for the Confirm action. To change the default confirm delay, edit the parameter in the PROBEWIN.INI file.

Standard X Increment

The is the distance in microns that the program will automatically increment the X stage axis for each standard coordinate acquisition, if the number of points specified in Standard Points To Acquire is less than the number of standard coordinate positions actually contained in the position database. This value can be set to any distance from zero to 100 microns.

Re-Standard Y Increment

This is the distance in microns that the program will automatically increment the Y stage axis for each re-standardization. This parameter is used to avoid analyzing the same sample spot on the standard and should be set to a value somewhat larger than the typical beam size of the microprobe. This value can be set to any distance from zero to 100 microns.

Run Selected Samples

This button starts the selected automation actions based on the selected samples. The program will confirm the number of standard, unknown and wavescan samples to be acquired during the automation.

The automation can acquire sample positions based on three different methods :

Use Last Unknown (Or Standard)

The default method the program uses to acquire automated samples is to base the acquisition on the last unknown sample setup. If no unknown samples exist, the program will base the acquisition on the last standard sample setup.

If no unknown or standard samples exist, the program will not be able to run automated samples. Always create a default sample from the Acquire! window using the New sample button before attempting to run automated samples.

Use Digitized Sample Conditions

If the microprobe interface supports computer control of the kilovolts, beam current and beam size then the user can specify that the program base the acquisition on the last unknown sample setup with these parameters based on the sample conditions specified when the sample positions were digitized. See the Conditions button above for more details on using this feature.

Use Digitized Sample Setups

Another method that can be used to acquire automated samples is to base the acquisition on sample setups that were specified when the sample positions were digitized. In this way, one can automatically data for different sample positions using different sample setups for tremendous flexibility. See the Sample Setups button above for more details on using this feature.

Use Digitized File Setups

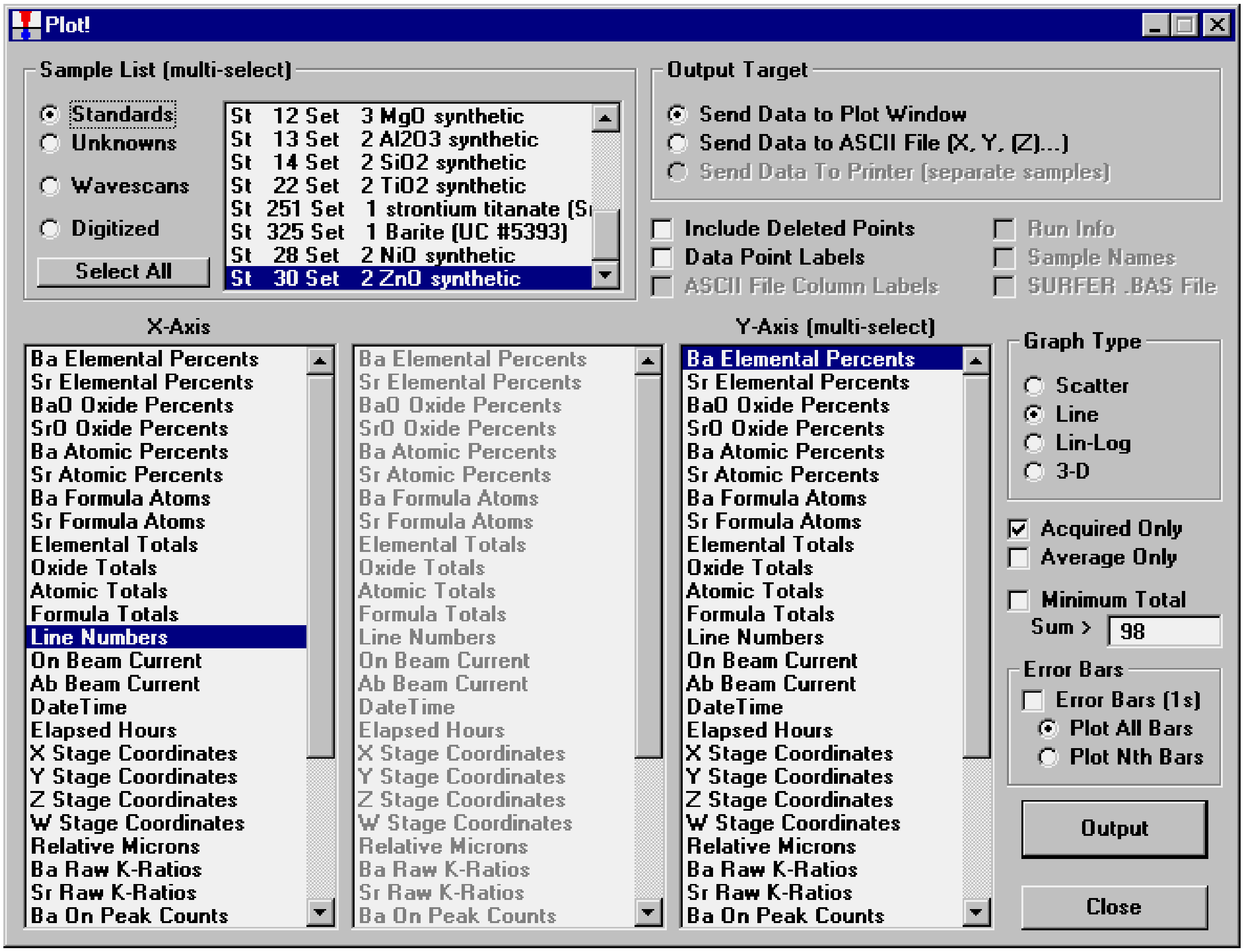
Yet another method that can be used to acquire automated samples is to base the acquisition on file name setups that were specified when the sample positions were digitized. In this way, one can automatically data for different sample positions using different previously created file setups for tremendous flexibility. See the File Setups button above for more details on using this feature. Note that standard intensity data from the file setup will be automatically loaded (if it has not been already) unless the option has been turned off from the Acquisition Options window.

Use Digitized Multiple Setups

Finally as a last option one can acquire automated samples based on multiple sample setups, specified when the samples were digitized. This is of course similar to the sample Setups option above except that one can acquire more than one sample setup for each position sample. See the Multiple Setups button above for more details on using the feature.

Plot!

The Plot! modeless windows provides graphical plotting of numerous data types in Probe for Windows. Standard, unknown and wavescan sample data may be displayed. Digitized data associated with a polygon digitization may also be displayed or output.


Plot!

Sample List (multi-select)

● Standards
○ Unknowns
○ Wavescans
○ Digitized

Select All

St 12 Set 3 MgO synthetic
St 13 Set 2 Al2O3 synthetic
St 14 Set 2 SiO2 synthetic
St 22 Set 2 TiO2 synthetic
St 251 Set 1 strontium titanate (S
St 325 Set 1 Barite (UC #5393)
St 28 Set 2 NiO synthetic
St 30 Set 2 ZnO synthetic

Output Target

● Send Data to Plot Window
○ Send Data to ASCII File (X, Y, (Z)...)
○ Send Data To Printer (separate samples)

☐ Include Deleted Points
☐ Data Point Labels
☐ ASCII File Column Labels
☐ Run Info
☐ Sample Names
☐ SURFER .BAS File

X-Axis

Ba Elemental Percents
Sr Elemental Percents
BaO Oxide Percents
SrO Oxide Percents
Ba Atomic Percents
Sr Atomic Percents
Ba Formula Atoms
Sr Formula Atoms
Elemental Totals
Oxide Totals
Atomic Totals
Formula Totals
Line Numbers
On Beam Current
Ab Beam Current
DateTime
Elapsed Hours
X Stage Coordinates
Y Stage Coordinates
Z Stage Coordinates
W Stage Coordinates
Relative Microns
Ba Raw K-Ratios
Sr Raw K-Ratios
Ba On Peak Counts

Ba Elemental Percents
Sr Elemental Percents
BaO Oxide Percents
SrO Oxide Percents
Ba Atomic Percents
Sr Atomic Percents
Ba Formula Atoms
Sr Formula Atoms
Elemental Totals
Oxide Totals
Atomic Totals
Formula Totals
Line Numbers
On Beam Current
Ab Beam Current
DateTime
Elapsed Hours
X Stage Coordinates
Y Stage Coordinates
Z Stage Coordinates
W Stage Coordinates
Relative Microns
Ba Raw K-Ratios
Sr Raw K-Ratios
Ba On Peak Counts

Y-Axis (multi-select)

Ba Elemental Percents
Sr Elemental Percents
BaO Oxide Percents
SrO Oxide Percents
Ba Atomic Percents
Sr Atomic Percents
Ba Formula Atoms
Sr Formula Atoms
Elemental Totals
Oxide Totals
Atomic Totals
Formula Totals
Line Numbers
On Beam Current
Ab Beam Current
DateTime
Elapsed Hours
X Stage Coordinates
Y Stage Coordinates
Z Stage Coordinates
W Stage Coordinates
Relative Microns
Ba Raw K-Ratios
Sr Raw K-Ratios
Ba On Peak Counts

Graph Type

○ Scatter
● Line
○ Lin-Log
○ 3-D

☑ Acquired Only
☐ Average Only

☐ Minimum Total Sum > 98

Error Bars

☐ Error Bars (1s)
● Plot All Bars
○ Plot Nth Bars

Wavescan samples can display KLM markers from the NIST database based on the analyzed elements in the range, all elements in the range or a specific element in the range.

Select All

This button will select all samples displayed in the Plot! sample list. Note that because each sample may contain a different set of acquired elements, the axis list boxes are updated each time a different sample is selected.

Output Target

The user may select either output to the Graph window or to a tab delimited ASCII file or to have each sample plot data automatically output to the default printer. If ASCII file output is selected, the program will ask for the output filename

(*.DAT extension) after calculation of all data has been performed. The default is to send data to the graph window for viewing.

Include Deleted Points

This option, if selected, will output data from samples even if the data has been previously flagged as deleted. This option has no effect on quantitative sample output.

Data Point labels

This option will display a line number for each value plotted in the graphical display mode. This option has no effect if ASCII file output is selected.

ASCII File Column Labels

This option will output a line of data column labels for each column of data in the ASCII file. The labels will be enclosed in double quotes. If the elements being output change during the output process, the program will automatically write a new column label line. This option has no effect if Graph output is selected.

Run Info

This option will output a header to the ASCII file including the user, title and description of the current run. In addition the current time and date will be displayed also. This option has no effect if Graph output is selected.

Sample Names

This option will output a line containing the sample name in double quotes before the data for that sample is output to the ASCII file. This option has no effect on data sent to the graph window.

Force Black and White Print

This option will force a color printer to print graphs black and white instead of color. This option applies to both the Print button in the Graph window as well as the Send To Printer option in the Plot! Window. In color mode monochrome printers (laser printers) will print grayscale and color printers will print color. In the Force Black and White mode, both types of printers will print black and white only.

Off Peak Labels

This option will add off-peak labels to the off-peak markers. Note that both actual and current off-peak markers and labels are shown. Actual off-peak markers are the positions of the off peaks when the sample was acquired while the current off-peak markers are the off-peak positions that are currently specified.

SURFER .BAS File

If the SURFER .BAS File option is selected, Probe for Windows, will, in addition to the actual data file, create a script file for use with SURFER for Windows (from Golden Software) for automatically generating contour and surface plots of your data. This assumes that the sample data is applicable to this type of processing.

Be sure that the SurferOutputVersionNumber parameter is correctly specified in the PROBWIN.INI file when using this option. Probe for Windows assumes Surfer v. 6.0, unless the parameter is specifically indicated. Due to changes in the file formats expected by Surfer v. 6 and Surfer v. 7, the correct script format must be output or the script will not run properly.

If the sample was a digitized polygon area grid, then the program will also automatically output a .BLN file for use with the SURFER for Windows surface and contour plot boundary feature. Notice that the digitized polygon grid coordinates are saved automatically and can be viewed, once the data is acquired, using the "plot" window to display digitized samples.

> *Be aware that SURFER for Windows expects X and Y coordinate data in the first two columns of the .DAT file, followed by multiple columns of Z data for plotting contours or surfaces. To accomplish this, be sure to select the 3-*

D graph type and select the X coordinate data in the x-axis list box, the Y coordinate data in the y-axis list box and the Z data (weight percents, etc.) in the z-axis list box using multiple selection.

The creation of the SURFER .BAS file requires that the GRIDBB.BAS to be present in the default directory (usually C:\Program Files\Probe for Windows).

SURFER for Windows Instructions

Probe for Windows can create a .BAS basic script file containing OLE code for creating contour and surface plots, or just screen display for testing purposes. This .BAS basic file is only to be used with the GS Scripter utility that comes with SURFER for Windows 95/NT v. 6.0 or higher.

To run the .BAS script, double click the GS Scripter icon, select File, Open and double click the correct .BAS file. Then click Run, Start to begin the automated plotting.

IMPORTANT! Note that the default output mode of the script file is "TEST", which will only output the plots to the screen and will produce a log of the plotting operations. To produce output to the default printer, comment out the line : OutputType$ = "TEST" by inserting a single quote in front of the line like this : 'OutputType$ = "TEST", and UNcomment the line : OutputType$ = "SURF" by removing the single quote in front of it.

Remember, the last uncommented "OutputType$" line will select the output. If necessary, the "GridMethodType%" can also be changed in the same fashion to change the gridding method used by the grid module. Note also, that any element column plot can be skipped by simply commenting out the appropriate "ZLabel$" lines, by inserting a single quote in front of the line.

Graph Type

The various graph types are selected here. The default graph type may be set from the PROBEWIN.INI file. Currently the following graph types are supported :

- Scatter (x and y data as points)

- Line (x and y data as points connected by lines, suitable for wavescan samples)

- Lin-Log (x data on a linear axis and y data on a log axis

- 3-D (x, y and z data as points)

Analyzed Elements Only

This option will re-load the axis lists to display either the analyzed elements only, or both the analyzed and specified elements. This option is used to allow the user to graph or output those elements calculated by difference or stoichiometry.

This option can also be utilized in order to output all elements in a run, even if the last sample does not contains elements analyzed and/or specified in other samples. Consider that because the elements listed in the axis lists are based on the last sample selected, if a selected sample prior to the last selected sample contains additional elements not in the last selected sample, they will not be output simply because they cannot be selected. Therefore, if it is desirable that these additional elements be output, follow these directions:

- From the Analyze! window sample list, select the last sample that will be output from the Plot! window.
- Click the Elements/Cations button.
- Add, as specified elements (no x-ray symbol), all additional elements (found in previous samples) that are desired to be output, that are not already in the last sample.

- Click OK and return to the Plot! window and uncheck the Analyzed Only button. This will allow the program to display all elements in the last selected sample, including the specified elements just added.
- Select all elements to be output. The program will output the selected elements whether they are analyzed or specified.

Average Only

This option will output only the data average of each sample to the plot window or the output ASCII data file. Note, if data point labels are selected for the plot window, instead of line numbers the program will use sample numbers.

Minimum Total

This option (if selected) will output only those data lines whose totals are at least the value specified. This option must be used with at least one column of quantitative data selected for graph or file output.

Error Bars

This option will allow the program to calculate one sigma error bars for certain data types, specifically the Elemental, Oxide Percents and On Peak Counts data types only.

Plot All Bars

This option will allow the program to plot one sigma error bars for each data point in each data set. The bars will be centered on the data point.

Plot Nth Bars

This option will allow the program to plot one sigma error bars for each data point in each data set as above, however the frequency of the plotted errors bars will depend on the number of data points displayed according to the following table:

Less than or equal to 50 points	Plot error bars for all points
Less than or equal to 100 points	Plot error bars for every other data point
Less than or equal to 150 points	Plot error bars for every 3rd data point
And so on…	

Error Bar Calculations

The formula for these error calculations are as specified:

On Peak Counts Errors:

$$\Delta I = \sqrt{I_P}$$

Where: I_P = On-peak counts (including background, de-normalized for deadtime, beam current and time)

Note that the result is normalized to deadtime, beam current and count time after the square root is calculated and before it is displayed.

K-Ratio Errors:

$$\Delta K = \frac{\sqrt{I_U}}{\left(I_S * ZAF_S\right)}$$

Where: I_U = Unknown on peak counts (background corrected, de-normalized for deadtime, beam and time)

I_S = Standard on peak counts (background corrected, de-normalized for deadtime, beam and time)
ZAF_S = ZAF correction for standard counts (to obtain pure element counts)

Note that the result is normalized to deadtime, beam current and count time after the square root is calculated and before it is displayed.

Elemental (or Oxide) Weight Percent Errors:

$$\Delta C = \Delta K * ZAF$$

Where: ΔK = K-ratio error (from above)
ZAF = unknown ZAF correction factors

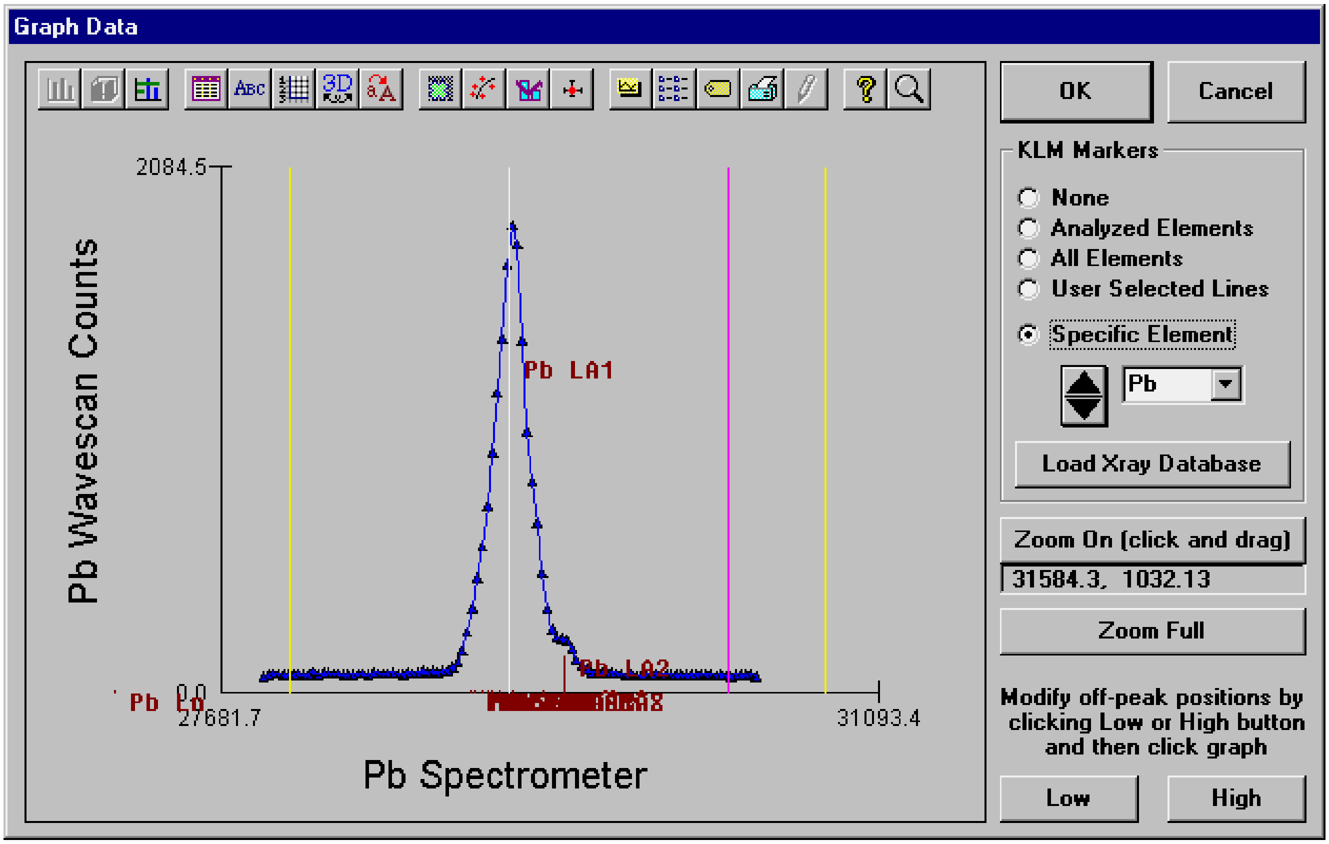
Output

This button will start the output process selected based on the Output Target type (graph window or ASCII file) selected by the user.

Graph Data

The Graph Data window is used in two modes. The first to plot x-ray intensity or compositional data from standard and unknown samples, and the second to plot x-ray intensity and spectrometer position data for wavescan samples. Multiple Y data is automatically plotted and listed in a color coded legend if specified in the Plot! window.

Digitized x and y position data from polygon boundary acquisitions may also be plotted in this window if this data exists in the current probe database file.



○ None
○ Analyzed Elements
○ All Elements
○ User Selected Lines
● Specific Element

Pb

31584.3, 1032.13

Modify off-peak positions by clicking Low or High button and then click graph

Numerous plot properties may be modified using the tool bar buttons at the top of the graph. Of particular interest is the System tool bar button which will allow the user to print a monochrome or color hardcopy of the display graph. Note that if a GDI layer is display it will not be output to the printer. In that case it is best to save the graph using the meta-file option and paste it to another application where a hard copy can be printed.

ASCII File Output

If ASCII file output was selected, the program will calculate data for the specified samples and then prompt the user for a path and filename to save the data to. All data files will be <tab> delimited and will normally have a .DAT extension.

Plotting Standard and Unknown Data

Both x-ray and compositional data may be plotted in the Graph Data window. Click and drag any portion of the graph for zooming into a region of the graph.

Click the Zoom On button to use "Hot Hit" mode which allows the user to click near a specific symbol to get the exact x and y data values. In this mode the click and drag zoom feature is disabled. Click the "Hot Hit" button to restore click and drag zooming.

Plotting Wavescan Data

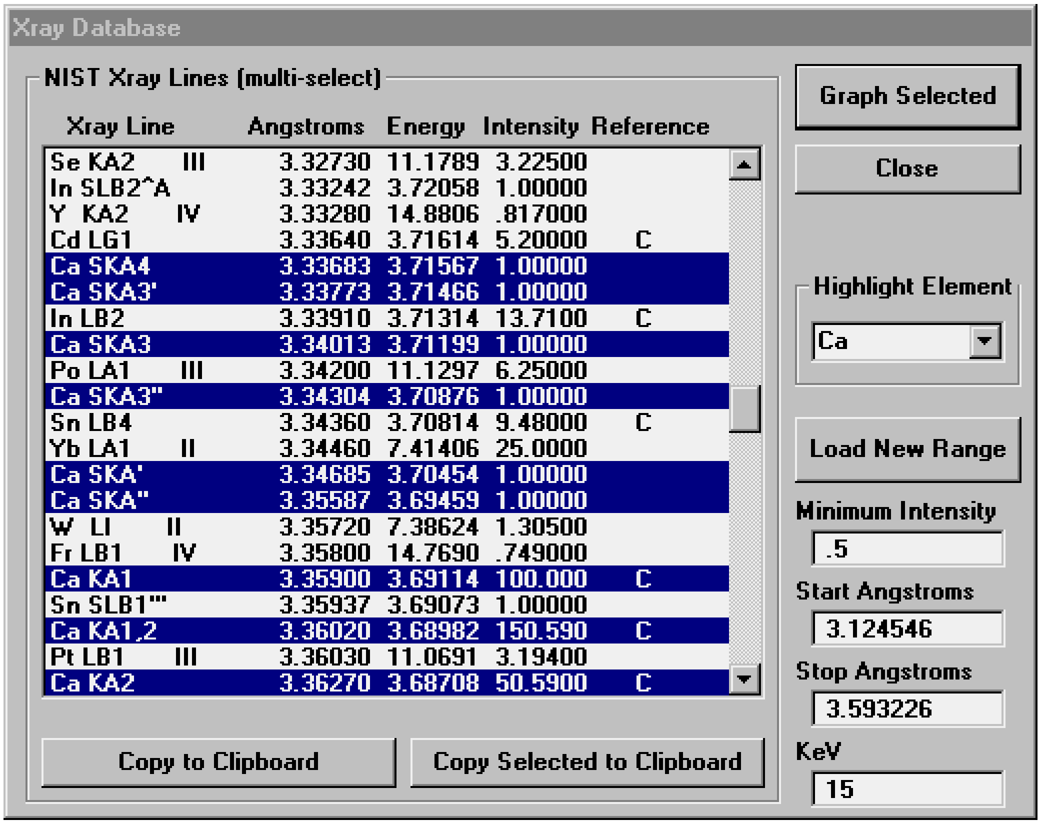
When wavescan data is plotted in the Graph Data window additional options for wavelength dispersive data are enabled.

KLM Markers

- None:	No KLM markers (default).
- Analyzed only:	All KLM markers for all elements in the wavescan sample (analyzed and specified).
- All Elements:	All KLM markers for all elements in range.
- User Selected Lines:	Only lines selected by the user in the Load Xray Database window.
- Specific Element	Only lines for the element selected in the list.

Load X-ray Database

Allow the user to see a list of all files in the graph range. If the User Selected Lines option is clicked, the users can select specific lines for graphing.



Xray Line	Angstroms	Energy	Intensity	Reference
Se KA2 III	3.32730	11.1789	3.22500	
In SLB2^A	3.33242	3.72058	1.00000	
Y KA2 IV	3.33280	14.8806	.817000	
Cd LG1	3.33640	3.71614	5.20000	C
Ca SKA4	3.33683	3.71567	1.00000	
Ca SKA3'	3.33773	3.71466	1.00000	
In LB2	3.33910	3.71314	13.7100	C
Ca SKA3	3.34013	3.71199	1.00000	
Po LA1 III	3.34200	11.1297	6.25000	
Ca SKA3"	3.34304	3.70876	1.00000	
Sn LB4	3.34360	3.70814	9.48000	C
Yb LA1 II	3.34460	7.41406	25.0000	
Ca SKA'	3.34685	3.70454	1.00000	
Ca SKA"	3.35587	3.69459	1.00000	
W LI II	3.35720	7.38624	1.30500	
Fr LB1 IV	3.35800	14.7690	.749000	
Ca KA1	3.35900	3.69114	100.000	C
Sn SLB1'"	3.35937	3.69073	1.00000	
Ca KA1,2	3.36020	3.68982	150.590	C
Pt LB1 III	3.36030	11.0691	3.19400	
Ca KA2	3.36270	3.68708	50.5900	C

Highlight Element: Ca

Minimum Intensity: .5

Start Angstroms: 3.124546

Stop Angstroms: 3.593226

KeV: 15

To change the range enter the start and stop (ending) angstroms limits and click the Load New Range button. The KeV parameter will limit the appearance of higher order lines that cannot be excited below the specified voltage. The Graph Selected button is only active if the User Selected Lines option is selected in the Graph dialog.

Low and High Off Peak Positions

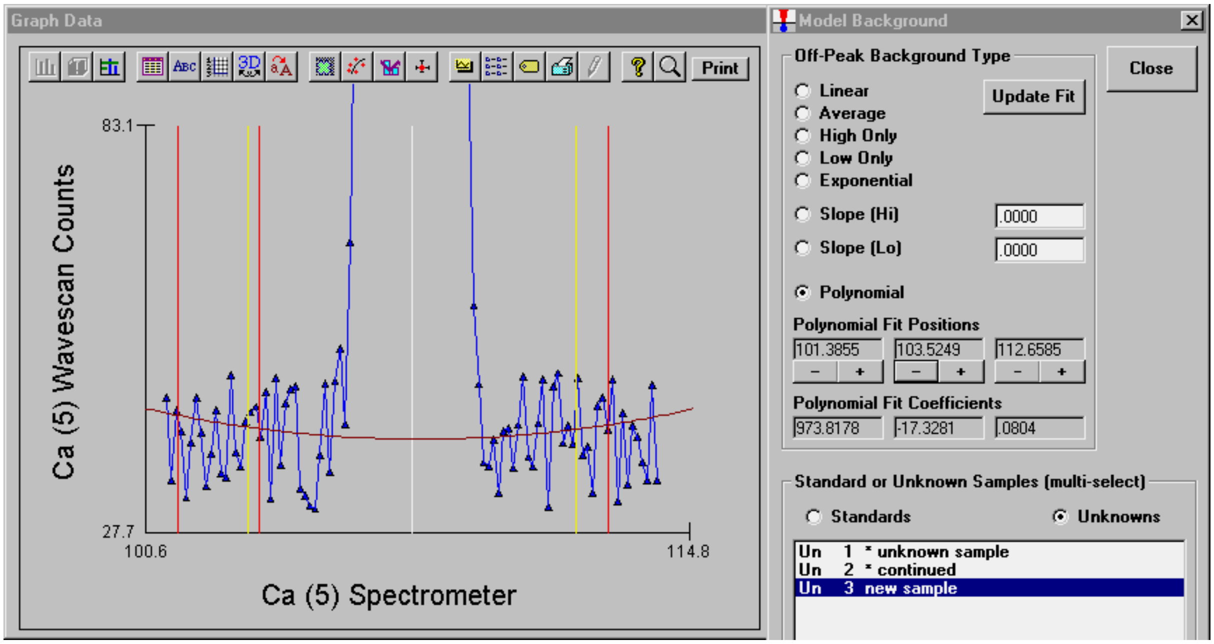
To move an off-peak position for the next standard or unknown acquisition, click either the Low or High off-peak button. Position the cursor over the graph (note that the cursor changes to a cross-hair) and click where it is desired that the new off-peak position be located.

If the current sample contains no data, the off-peak position is updated for that sample, otherwise a warning is given that the off-peak position selected will be applied to the next new sample that is created.

The new off-peak position is shown in yellow, while the original off-peak position for the displayed sample is shown in purple.

Model Background

To model the various off-peak background correction types, click the Model Background button. This option is only available when a wavescan sample is displayed.



Off-Peak Background Type

○ Linear
○ Average
○ High Only
○ Low Only
○ Exponential
○ Slope (Hi) .0000
○ Slope (Lo) .0000
● Polynomial

Polynomial Fit Positions: 101.3855 103.5249 112.6585

Polynomial Fit Coefficients: 973.8178 -17.3281 .0804

Standard or Unknown Samples (multi-select)

○ Standards ● Unknowns

Un 1 * unknown sample
Un 2 * continued
Un 3 new sample

To see the background model, click the specific background type and observe the plotted background curve in the Graph window.

For the exponential, slope and polynomial off-peak background types coefficients are required. The exponential, slope-high and slope-low coefficients are entered by hand.

The exponential exponent may be –6 to +6. A slope of zero will produce exactly the same result as the high-only or low-only off-peak background type.

A non-zero slope will produce a sloped background curve from either the high or low off-peak position depending on the selection.
The polynomial background fit coefficients are calculated automatically as the three different fit positions are moved by clicking the minus (-) or plus (+) buttons.

The slope or polynomial coefficients can be applied to selected standard or unknown samples for use with a sample specific slope or polynomial off-peak background correction. That is, the actual slope or polynomial background correction will be calculated for each sample based on the actual off-peak data for that sample using the fit coefficients from the fit model.

To make the assignment to the selected unknown samples, be sure to click Ok in the Model Background dialog AND ALSO in the Graph Data dialog.

Area Peak Factor Calculations
The dialog will also calculate the background corrected peak and integrated areas. These are useful for Area Peak Factor (APF) calculations. The program will calculate both the P/I (Peak/Integrated) and the I/P (Integrated/Peak) values. The one that is ultimately utilized for an APF calculation by the user depends on whether the wavescan represents the primary standard or the secondary standard. See the section of APF calculations in the Analytical menu section for details on how these values are used for the calculation of APFs.

The APF calculations are in units of counts per second for the peak intensities and counts per second * spectrometer range (between the low off-peak and high off-peak positions) for the integrated intensities.

Close

This button will close the Plot! Window, although since it is a modeless dialog it can be left open if enough screen space is available.

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Probe Menu Details

File

The File menu can be used to create new Probe for Windows runs, open an existing run, save the current run to another name, close the current run, display the current probe database file information, print the current log window contents or log window selection or exit the program.

New

The New menu opens a dialog to select a **new** Probe for Windows database file. If the file already exists, the program will prompt the user whether to overwrite the existing file. After the new file has been created the program will display a file information dialog to allow the user the personalize the run title and description.

The program will automatically append a .MDB extension to the file name if one is not supplied by the user. The .MDB file name indicates Microsoft Database and is a valid Microsoft Access v. 1.1 database file.

Open

The Open menu allows the user to select an **existing** Probe for Windows database file to process previously acquired data or to append additional acquisition data. The program will check that the specified file is of the correct format and warn the user if not. Probe for Windows generally uses a file extension of .MDB for it's database file extension.

Probe for Windows performs a general initialization of the ZAF correction factors when it opens a Probe for Windows database. If a x-ray initialization error occurs during this procedure, the program will allow the user to continue to open the file anyway or not. If an error does occur, for example an x-ray overvoltage condition, the user should take whatever steps are necessary to correct the anomalous condition.

Save As

The Save As menu allows the user to save the currently open Probe for Windows database file to another file name.

This option allows the user considerable flexibility in how new runs are created and utilized. For example, one possibility is to create a number of generic Probe for Windows runs that contains only a standardization for certain elements (FELDSPAR.MDB for example). Then, at a later time, the user could open that run and use the Save As option to save the run to another file name. Doing so, will create a new run with only the previously acquired standardization, to which unknown can then be acquired without altering the original run containing only the standardization.

Of course, whether this procedure is applicable or not depends greatly on both the reproducibility of the spectrometers and the accuracy required by the user.

Compact
The Compact menu allows the user to compact the current Probe database file. This allows the database to be sorted for maximum efficiency and the indexes updated. If the database has become very fragmented this process can also save a significant amount of disk space for large database files.

Close
The Close menu allows the user to close the currently open Probe for Windows database file. This can be used to close the current file before opening a different Probe for Windows database file without re-starting the program. The program will ask the user to confirm the close operation and all parameters are automatically saved.

Find File
This menu allows the user to open an existing run by browsing the available runs on disk. This is useful when the filename itself is unknown to the user, but the run itself, might be recognized on another basis such as user name or title.

This dialog has a directory browser that can be used to load Probe run files from various sub-directories if desired. For example, each user could utilize a separate sub-directory or their own or the different types of analysis could be used to define a sub-directory structure to hold files.

File Information
This menu displays and allows the user to change the run title and/or description fields for the currently open run. The title and user fields are limited to 64 characters and the description field is limited to 255 characters.

Normally, the user should completely fill out these fields since the text is saved not only to the user's run database, but also the user log database (see USERWIN.EXE). It is often helpful to use the description field to list the sample numbers or designations. Click the Insert <cr> button to make a new line in the description field.

Print Log
This menu allows the user to print the entire contents of the log window or selected portions of the log window. Be sure to click the Selection field if only a text selection is to be printed, otherwise the program will print the entire log window text.

Print Setup
This menu allows the user to change the default printer or printer setup options for the currently selected printer.

Exit
This menu exits the program. The program will ask the user to confirm the exit procedure if an analytical run is open.

Edit

Cut
This menu cuts the selected text from the log window to the Windows Clipboard.

Copy
This menu copies the selected text from the log window to the Windows Clipboard.

Paste

This menu pastes text from the Windows Clipboard to the log window.

Select All

This menu selects all text in the log window.

Clear All

This menu clears all text from the log window.

Standard

Standard Database

This menu invokes a shell process to launch the Standard for Windows database application (STANDARD.EXE) as an asynchronous process. The standard database is used to maintain the database of standard compositions and also includes a number of features for exploring analytical conditions based on various standard compositions.

Since the shell process runs independently, it can be accessed during an automation or analysis procedure.

Add Standards To Run

This menu allows the user to specify additional standards to be used in the currently open Probe for Windows run.

Note that if the run is closed before any data is acquired for these newly added standards, then they will have to be added again, if the run is re-opened later to acquire data for them unless they are saved as the probe database is closed by clicking Yes when asked whether to save them for the next session. This is because Probe for Windows updates the list of standards in the run each time a database file is opened based on the standard samples that have been actually created.

All standards that need to be digitized, acquired or assigned, must be first added to the run using this window dialog.

Standards may also be removed from the standard list with some reservations. First, a standard cannot be removed from the standard list if it references a standard sample already created in the current probe database. Second, even if a standard does not reference a standard sample, it may still itself be referenced by a standard, interference standard or MAN (mean atomic number) background standard assignment, especially if a sample setup was loaded from another probe database using the Load File Setup option. It is strongly suggested that the user check that all assignments do not reference the standard number that is to be removed.

In the case of MAN standard assignments, it may be useful to use the Clear All MAN Assignments menu item under the Analytical menu to remove all standard references. These assignments can be easily reloaded by default the next time the MAN Fits menu is selected.

X-Ray

X-Ray Database

This menu allows the user the view specified ranges of the NIST x-ray wavelength database. This database covers the wavelength range from approximately 0.5 to 100.0 angstroms and includes higher order reflections as well. Higher order reflections are reduced in intensity by 25% for each subsequent order to simulate the effect of PHA analysis.

The x-ray list range (Start Angstroms and Stop Angstroms) and minimum search intensity (Minimum Intensity) of the x-ray list can be specified by the user. Note that since the display is limited to about 1000 items, the program will automatically increase the minimum intensity until the returned list of x-ray lines is less than 1000. To view x-ray lines of lesser intensity, simply reduce the x-ray range and click the Re-Load button.

Description of the X-ray Database (adapted from NIST documentation by C. Fiori)

The NIST x-ray database is based on 4985 (1st order) entries and includes all the measurable x-ray lines, satellites and absorption edges from under 100 eV to over 120 keV. Additionally, most of the x-ray lines and satellites are assigned a relative intensity (relative to the alpha-1 line in each family). The data base was assembled primarily from four sources:

1.) B.L. Doyle, W.F. Chambers, T.M. Christensen, J.M. Hall and G.H. Pepper "SINE THETA SETTINGS FOR X-RAY SPECTROMETERS", Atomic Data and Nuclear Data Tables Vol. 24, No 5, 1979.

2.) E.W. White, G.V. Gibbs, G.G. Johnson Jr. and G.R. Zechman "X-RAY WAVELENGTHS AND CRYSTAL INTERCHANGE SETTINGS FOR WAVELENGTH GEARED CURVED CRYSTAL SPECTROMETERS" Report of the Pennsylvania State Univ., 1964.

3.) J.A. Bearden "X-RAY WAVELENGTHS AND X-RAY ATOMIC ENERGY LEVELS" Rev. Mod. Phys., Vol. 39, No. 78, 1967.

4.) J.A Bearden and A.F. Burr,"REEVALUATION OF X-RAY ATOMIC ENERGY LEVELS", Rev. Mod. Phys., Vol. 31, No. 1, 1967.

Each x-ray line or edge series as a function of atomic number was fit to a fourth degree polynomial. The fit was subtracted from the appropriate data and the residuals plotted and examined. In this way rogue entries could be identified and corrected. The resulting data base is considered to be sufficiently accurate for any application involving the Si (Li) x-ray detector and single crystal wavelength spectrometers.

Note that the last entry in the x-ray database window gives a code for the source of the entry. If the column is blank the source is reference 2. If the column contains the letter "C" the source is reference 1. If the letters "BB" appear, the source is reference 4. The letters "W,F" mean that reference 2 was used but the relative transition probability has been adjusted by Fiori. Reference 3 was used as a check since it is the source of many of the entries of reference 1.

In column 3 the notation KA1,2 means the entry is the weighted sum of the KA1 and KA2 in the ratio 2 to 1. For low atomic number the entries are not self consistent since the data is from different sources. If the column begins with the capital letter S then the entry is a satellite line due to doubly ionized atoms. The relative transition values for these entries are only valid for electron excited specimens, and are, at best, estimates.

Th following are Siegbahn to shell-transition notation conversions:

```
KA   = KA1+KA2+KA3
KA1,2 = (2*KA1+KA2)/3
KA1  = K-L3
KA2  = K-L2
KA3  = K-L1
KB   = SUM(KBn)
KBX  = Metal
KB1  = K-M3
KB1' = KB1+KB3+KB5
KB2  = (K-N3)+(K-N2)
KB2' = K-N3
KB2'' = K-N2
KB3  = K-M2
KB4  = K-N4)+(K-N5)
KB5  = K-M4)+(K-M5)
```

```
KB5' = K-M5
KB5'' = K-M4
Kd1  = K-O3
Kd2  = K-O2
LA   = LA1+LA2
LA1  = L3-M5
LA2  = L3-M4
LB1  = L2-M4
LB10 = L1-M4
LB15 = L3-N4
LB17 = L2-M3
LB2  = L3-N5
LB3  = L1-M3
LB4  = L1-M2
LB5  = L3-O4)+(L3-O5)
LB6  = L3-N1
LB7  = L3-O1
LB9  = L1-M5
LG1  = L2-N4
LG11 = L1-N5
LG2  = L1-N2
LG3  = L1-N3
LG4  = L1-O3
LG4' = L1-O2
LG6  = L2-O4
LG8  = L2-O1
Ll   = L3-M1
Ln   = L2-M1
Ls   = L3-M3
Lt   = L3-M2
Lu   = L3-N6)+(L3-N7)
Lv   = L2-N6
MA1  = M5-N7
MA2  = M5-N6
MB   = M4-N6
MG   = M3-N5
MG2  = M3-N4
MZ1  = M5-N3
MZ2  = M4-N2
Md   = M2-N4
Me   = M3-O5
```

Spectrometer Table

This menu item outputs a table of spectrometer positions for all elements based on the crystals configured for the current microprobe setup in the SCALERS.DAT file in the Probe for Windows sub directory.

Both the alpha and beta series lines are listed. This table is especially useful when printed out and used as a reference when setting up an analytical setup to determine which spectrometers and crystal combinations can be used for the elements of interest.

Emission Table

This menu lists a table of emission energies (or angstroms) for all elements and their major analytical x-ray lines.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries use the CalcZAF program.

Edge Table

This menu lists a table of absorption edge energies (or angstroms) for all elements and their major analytical x-ray lines.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries use the CalcZAF program.

Fluorescent Yield Table

This menu lists a table of fluorescent yield fractions for all elements and their major analytical x-ray lines.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries use the CalcZAF program.

MAC Table

This menu lists a table of MACs (mass absorption coefficients) for the specified element and it's major analytical x-ray lines.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries use the CalcZAF program.

The program will automatically load the values based on the selected default MAC table. See the Analytical | ZAF Selection section for a description of the MAC table choices.

Analytical

Analysis Options

The Analysis Options dialog allows the user to turn on or off a number of analytical options. This allows for greater flexibility in the quantitative analysis of unusual samples or to pinpoint problems in the analysis.

Use Deadtime Correction

The deadtime correction can be disabled for use in certain unusual analytical situations. The program will type a warning to the log window during a quantitative analysis if the deadtime correction is disabled. Two deadtime correction options are available, the normal expression shown here :

Expression # 1 :

$$I = \frac{i}{1.0 - i\tau}$$

Where :	i	is the raw measured counts in cps
	τ	is the deadtime constant in seconds
	I	is the deadtime corrected counts in cps

and a high precision expression (Willis, 1993) for use with very high count rates (> 50K cps) shown here :

Expression #2 :

$$I = \frac{i}{1.0 - \left| \, i\tau + i^2 \frac{\tau^2}{2} \, \right|}$$

This high precision expression differs from the normal expression only at very high count rates. The following table shows the difference between the two expressions at various count rates :

CPS	#1 @ 1 us	#2 @ 1 us	#1 @ 2 us	#2 @ 2 us	#1 @ 4 us	#2 @ 4 us
5K	.995	.9950125	.99	.99005	.98	.9802
10K	.99	.99005	.98	.9802	.96	.9608
50K	.95	.95125	.9	.905	.8	.82
100K	.9	.905	.8	.82	.6	.68
200K	.8	.82	.6	.68	.2	.52
400K	.6	.68	.2	.52	--	--

Use Beam Drift Correction

The beam drift correction can be disabled for use in certain unusual analytical situations. The program will type a warning to the log window if the beam drift correction is disabled.

The beam drift correction calculation is shown here (where beam integration time is taken into account) :

$$I_C = I_U \frac{B_N \frac{T_U}{T_N}}{B_U}$$

Where : I_C is the beam drift corrected unknown intensity
I_U is the uncorrected unknown intensity
B_N is the nominal beam current
T_U is the count time for the unknown intensity
T_N is the count time for the nominal beam current measurement
B_U is the beam current for the unknown intensity

However, since Probe for Windows stores and utilizes x-ray intensities in counts per second (already normalized to time), the actual beam drift correction, for direct reading faraday cup microprobes, is quite a bit more simple than the rigorous example above, and is instead shown here:

$$I_C = I_U \frac{B_N}{B_U}$$

Where : I_C is the beam drift corrected unknown intensity
I_U is the uncorrected unknown intensity
B_N is the nominal beam current
B_U is the beam current for the unknown intensity

This means that if the actual measured beam current for your unknown decreases over time, the value of the fraction $\frac{B_N}{B_U}$, increases and therefore the corrected x-ray intensity is adjusted upwards. If on the other hand, the actual measured beam current for your unknown increases over time, then the value of the fraction $\frac{B_N}{B_U}$, decreases and therefore the corrected x-ray intensity is adjusted downwards.

The beam drift correction essentially normalizes the x-ray intensities to the x-ray intensity that would be expected at the nominal beam current. For example, if most of the x-ray measurements are made at a beam current of 20 nA, then setting the nominal beam current to 20 nA will ensure that the x-ray intensities will appear scaled to readings taken at 20 nA (even if the measurement is taken at a different beam current). In an alternative usage, the x-ray intensities may be expressed in counts per second per nA by setting the nominal beam current to 1.0. See the Count Times dialog in the Acquire! Window to manually change the nominal beam current.

Use Automatic Drift Correction on Standard Intensities

Probe for Windows uses a sophisticated automatic drift correction to correct the counts on the standard, interference and MAN standard intensities. This standard drift correction should not be confused with the beam drift correction described previously. With the automatic standard drift correction, when Probe for Windows loads the unknown count data to analyze a sample data point, the program will automatically check each element to see if the standard for that channel was measured both before and after the data on the unknown. If there exist sufficient standard sets, the program will automatically compute a linear interpolated drift corrected analysis.

If there are only a single standard data set, the program will simply use the set of counts on that standard which occurred closest in time prior to the unknown. Thus it is possible to perform analyses of unknowns as they are acquired, although they will not be corrected for drift. Later, when the standards have been run again, the program can recalculate all the analyses, and they will be corrected for standard count drift in real time.

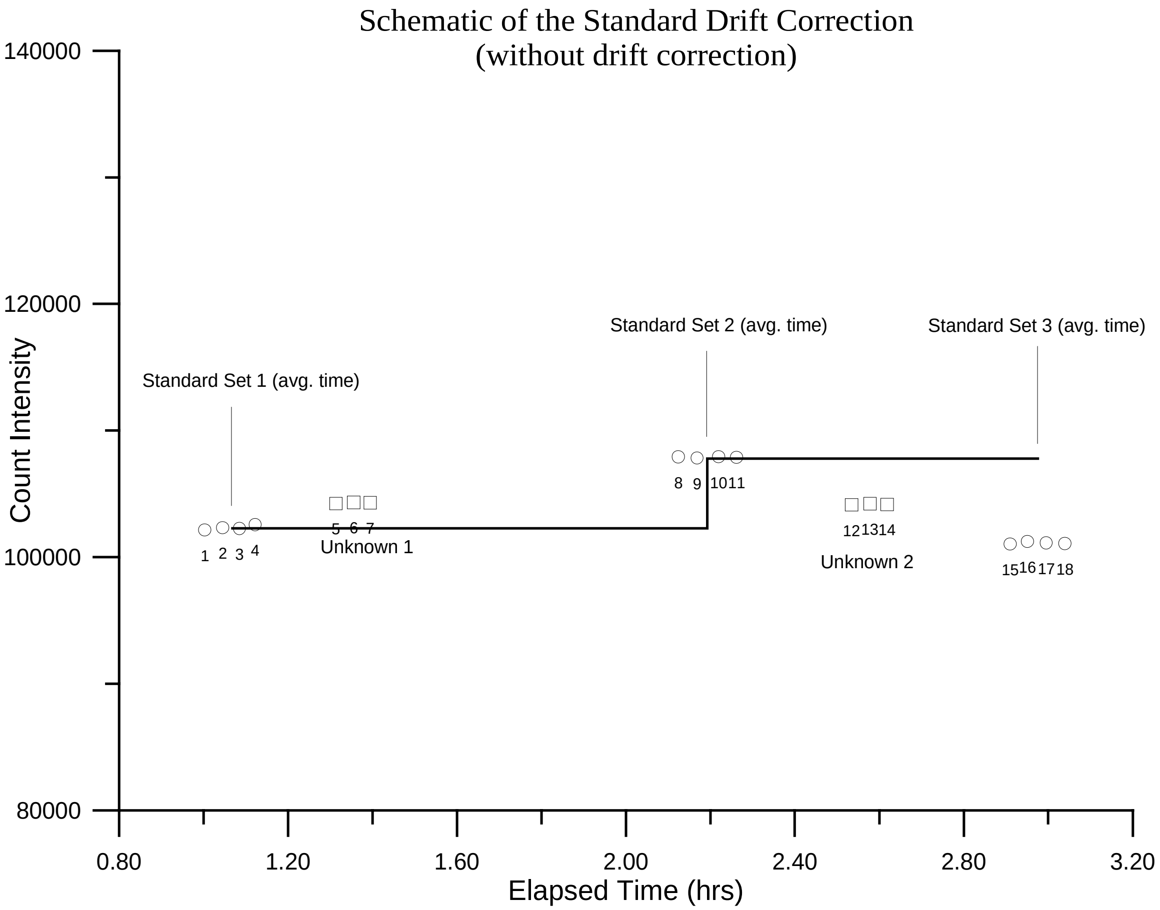
The user may go back and acquire each standard up to 20 times during a run (20 "sets" of counts on the standard) and the program will determine which two sets, if any, were made closest, before and after, the counts on the unknown. The form of the standard drift correction is shown below :

$$I_S = I'_S + (I''_S - I'_S)\frac{(T_U - T'_S)}{(T''_S - T'_S)}$$

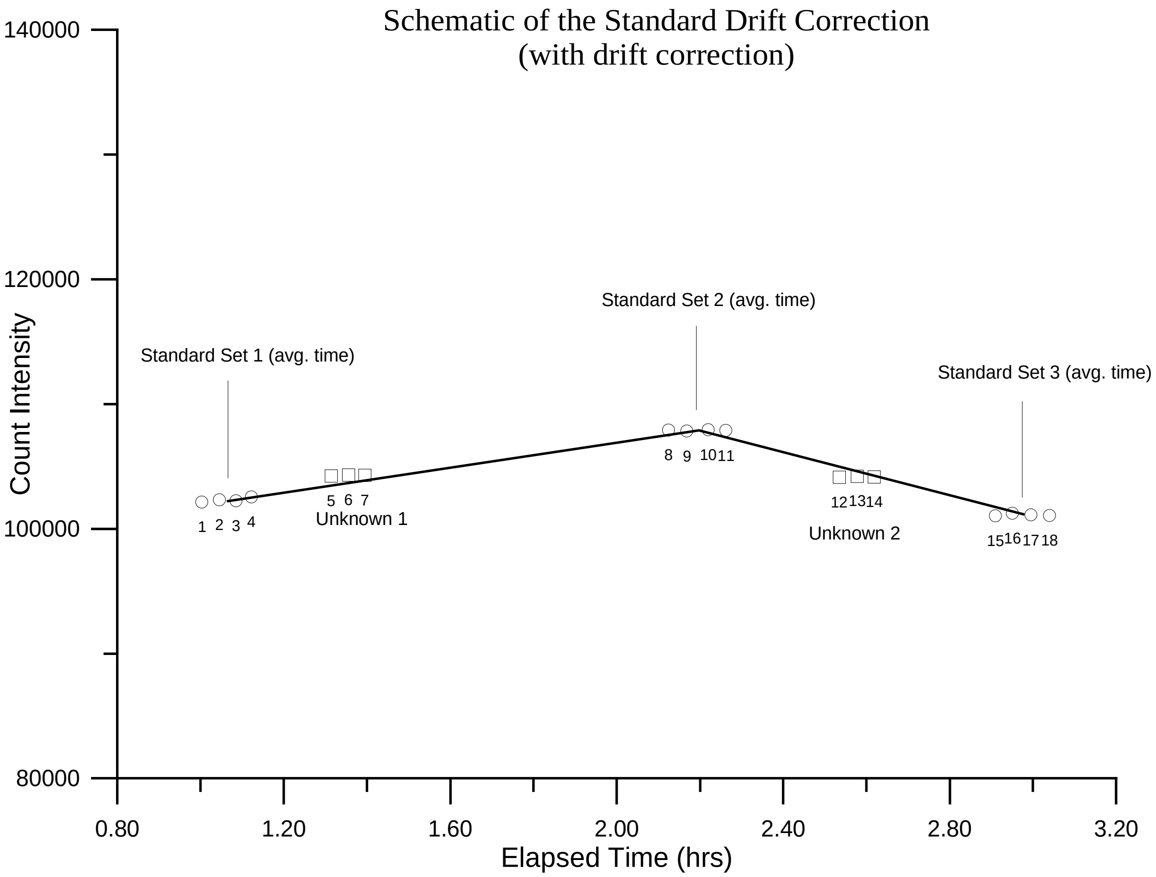
Where : I_S is the drift corrected standard intensity
I'_S is the standard intensity from the preceding standardization
I''_S is the standard intensity from the following standardization
T_U is the real time of the unknown analysis
T'_S is the real time of the preceding standardization
T''_S is the real time of the following standardization

The following explanation will demonstrate how the standard drift correction is actually implemented in Probe for Windows. The example data shown here consists of four standard data points (set 1), followed by three unknown data points (points 5, 6 and 7), followed by four more standard data points (set 2), followed by three more unknown data points (points 12, 13, and 14), followed by a final four standard data points (set 3).

The first graph shows the standard intensities and how they are applied to unknown data ***without*** the use of a standard drift correction. The stepped horizontal lines drawn between the standard sets represents the standardization intensities. Note that each unknown data point uses the average intensity of the previous standardization. In a situation where drift has occurred, significant errors will result for the unknown analyses.



The second graph shows the standard intensities and how they are applied to unknown data ***with*** the use of a standard drift correction. The sloped horizontal lines drawn between the standard sets represents the standardization intensities. Note that each unknown data point uses the ***interpolated*** average intensity of the previous ***and*** the subsequent standardizations. In a situation where linear drift has occurred, the standard drift correction allows for the adjustment of standard intensities for intervening unknown analyses.



Note that because all quantitative samples (standard and unknowns) are treated as unknowns for the purposes of quantitative analysis, the standard drift correction is also applied to the analysis of standard samples. However, it is useful to note that for a given primary (assigned) standard sample, standard data points acquired ***just before*** the average time of acquisition of that standard are interpolated between the previous standard and the standard itself, while standard data points acquired ***just after*** the average time of acquisition of that standard are interpolated between the standard itself and the subsequent standard. Therefore, for primary standards, the data points within that standard are normally analyzed using two different standard intensity data sets. Furthermore, even in the case of no standard drift correction, the program will still utilize two different standard intensities when analyzing those data points just before or just after the average time of acquisition of the standard.

Use Assigned Interference Corrections on Standards and Unknowns

This option will either enable or disable the interference corrections for subsequent quantitative analysis. This option is automatically loaded if interference corrections are specified by the user. Use this option to easily determine the total effect of all of the interference corrections specified, on a quantitative analysis.

Use Assigned Volatile Corrections on Unknowns

This option will either enable or disable the volatile element corrections for subsequent quantitative analysis. This option is automatically loaded if volatile element corrections are specified by the user. Use this option to easily determine the total effect of all of the volatile element corrections specified, on a quantitative analysis.

Use Absorption Corrected MAN Continuum Intensities

This option will either enable or disable the use of absorption corrected continuum intensities for calculation of the MAN background correction in subsequent quantitative analysis. For details on this procedure see Donovan and Tingle, 1996.

Use Empirical MAC Values

This option will either enable or disable the use of empirical MACs (mass absorption coefficients) for subsequent quantitative analysis. This option is automatically loaded if empirical MACs are specified by the user. Use this option to easily determine the total effect of all of the empirical MACs specified by the user, on a quantitative analysis.

Use Empirical APF Values

This option will either enable or disable the use of empirical APFs (Area Peak Factors) for subsequent quantitative analysis. This option is automatically loaded if empirical APFs are specified by the user. Use this option to easily determine the total effect of all of the empirical APFs specified by the user, on a quantitative analysis.

Use Detailed Printout for Data and Analytical Results

This option will disable the output of some of the printed output to the log window for a quantitative analysis. The output options not seen if this option is enabled include the standard and unknown k-factors and ZAF correction factors.

Use Automatic Format For Results

This option will allow the program to use maximum precision or only the statistically significant digits when formatting numbers for output rather then fixed format (F8.3). This option is useful when printing out trace element concentrations when more significant digits to the right of the decimal point are desired or when it is desirable to easily ascertain exactly how sensitive the quantitative analysis is, based on the counting statistics.

If the statistically significant automatic format output option is selected, then the program will calculate the percent error (precision) and the detection limit for each element and use this information to first format the number so that only statistically significant digits are displayed and finally it will check the value against the calculated detection limit and set the value to "n.d." (not detected) if the value is below the detection limit. This is scaled for each data type output (elemental, oxide, atomic percent and formula).

The calculation for significant digits is based on the percent analytical error as follows:

Percent Analytical Error	Number of Significant Digits
> 10	1
> 1	2
> 0.1	3
> 0.01	4
> 0.001	5
> 0.0001	6

Print Analyzed and Specified On Same Line

This option will allow both analyzed (measured) and specified elements to be printed out on the same line in the log window. Normally the default is to print specified elements on a separate line from the analyzed elements. This causes the log window and Analyze! grid results to be more comparable in appearance.

Calculate Electron and X-ray Ranges

This option will cause the program to calculate both Kanaya-Okayama incident electron distance radii and effective x-ray transmission for a range of densities and thicknesses based on the actual sample composition at the current sample conditions (KeV).

The expression:

$$electron\,radius = \frac{0.0276 \cdot A \cdot E^{1.67}}{\rho \cdot Z^{0.89}}$$

is used for the Kanaya-Okayama calculation and assumes, for compound specimens, that the average atomic number and average atomic weight are calculated based on the weight fractions of the elements in the specimen.

The expression:

$$transmission\ fraction = e^{-\mu \rho t}$$

is used to calculate the effective x-ray transmission and assumes, for compound specimens, that the average mass absorption coefficient is calculated based on the weight fractions of the elements in the specimen.

Use Oxygen From Halogens (F, Cl, Br and I) Correction

This option will allow the program to calculate the equivalent oxygen from the measured or specified halogen concentrations (F, Cl, Br and I) and subtract that amount from the amount of calculated stoichiometric oxygen.

During the calculation, since it requires two halogen atoms to replace one oxygen atom, one-half (by atom) of the halogens present are converted to equivalent oxygen and that amount is subtracted during the compositional iteration procedure. The adjustment is iterated along with the modified matrix correction factors, due to the change in overall composition. This option is applicable for ZAF/pr(z), Bence-Albee and calibration curve matrix correction calculations. This option applies only to samples where oxygen is calculated by stoichiometry and have measured or specified F, Cl, Br or I.

If this option is NOT used for samples where oxygen is calculated by stoichiometry and halogens are present, the software will simply report the oxygen equivalent of the halogens without subtracting the calculated amount. In this case, the user may then manually subtract the oxygen equivalent from the stoichiometric calculated oxygen, however, since the matrix correction is not adjusted for the change in oxygen concentration, the calculation of the other elements (especially F, due to it's large correction factor in the presence of oxygen) will be slightly in error.

Finally it should be noted that to be internally consistent in the matrix calculations, all oxide standard compositions used in halogen analyses should reflect the same adjustment for equivalent oxygen in the standard database as is used for the unknown (or standard) analysis in Probe for Windows.

For example, the following standard composition is entered with the assumption that all cations have a full complement of stoichiometric oxygen:

```
St  112 biotite #3
TakeOff = 40  KiloVolts = 15
Oxide and Elemental Composition

Average Total Oxygen:       40.474     Average Total Weight%:  101.488
Average Calculated Oxygen:  40.474     Average Atomic Number:   13.413
Average Excess Oxygen:        .000     Average Atomic Weight:   21.358
Oxygen Equiv. from Halogen:  1.697

ELEM:     SiO2   Al2O3     FeO     MgO     CaO    Na2O     K2O    TiO2
XRAY:       ka      ka      ka      ka      ka      ka      ka      ka
OXWT:   38.622  10.721  18.131  14.011    .020    .690   9.210   2.290
```

```
ELWT:   18.053   5.674  14.093   8.449    .014    .512   7.646   1.373
KFAC:    .1368   .0382   .1205   .0528   .0001   .0024   .0689   .0119
ZCOR:   1.3201  1.4841  1.1692  1.6008  1.0860  2.1594  1.1093  1.1506
ATWT:   13.527   4.426   5.311   7.316    .007    .469   4.115    .603

ELEM:      MnO     BaO    Rb2O      Cl       F     H2O       O
XRAY:      ka      la      la      ka      ka              ka
OXWT:     .950    .111    .030    .020   4.020   2.663    .000
ELWT:     .736    .099    .027    .020   4.020    .298  40.474
KFAC:    .0062   .0008   .0002   .0002   .0112   .0030   .1826
ZCOR:   1.1891  1.3161  1.4008  1.2064  3.6053   .0000  2.2167
ATWT:     .282    .015    .007    .012   4.453   6.222  53.236
```

Note that the total for the above composition is actually greater than 100% due to the fact that in reality the fluorine and chlorine actually replace some of the cation oxygen in this mineral. Note also, the oxygen equivalent from all halogens (F, Cl, Br and I) is reported, but not subtracted from the stoichiometric oxygen (oxygen from cations).

Calculations using this standard composition and an adjustment for equivalent oxygen from halogens in the matrix correction procedure will be slightly in error due to the fact that the standard k-factor calculation will not reflect the proper reduction of stoichiometric oxygen due to the presence of halogens.

Since the default mode of this analysis option (unchecked) is to only display the equivalent oxygen and not actually utilize it in the matrix corrections, then results calculated using typical standard compositions will at least be internally consistent.

However, if it is desired to use this analysis option by reducing the calculated stoichiometric oxygen in the matrix correction then for internally consistent results, the user should make an adjustment (reduction) in the amount of stoichiometric oxygen in the standard composition. This is easily done by noting the actual amount of stoichiometric oxygen (adjusted) in the Standard Composition dialog (see menu Standard | Modify) and entering that value for the concentration of oxygen as seen here:

```
St  112 biotite #3
TakeOff = 40  KiloVolts = 15
Oxide and Elemental Composition

Average Total Oxygen:       38.777     Average Total Weight%:   99.791
Average Calculated Oxygen:  40.474     Average Atomic Number:   13.505
Average Excess Oxygen:      -1.697     Average Atomic Weight:   21.480
Oxygen Equiv. from Halogen:  1.697

ELEM:     SiO2   Al2O3     FeO     MgO     CaO    Na2O     K2O    TiO2
XRAY:      ka      ka      ka      ka      ka      ka      ka      ka
OXWT:   38.622  10.721  18.131  14.011    .020    .690   9.210   2.290
ELWT:   18.053   5.674  14.093   8.449    .014    .512   7.646   1.373
KFAC:    .1367   .0382   .1207   .0529   .0001   .0024   .0689   .0119
ZCOR:   1.3208  1.4841  1.1681  1.5982  1.0858  2.1546  1.1091  1.1499
ATWT:   13.836   4.527   5.432   7.483    .008    .479   4.209    .617

ELEM:      MnO     BaO    Rb2O      Cl       F     H2O       O
XRAY:      ka      la      la      ka      ka              ka
OXWT:     .950    .111    .030    .020   4.020   2.663  -1.697
ELWT:     .736    .099    .027    .020   4.020    .298  38.777
KFAC:    .0062   .0008   .0002   .0002   .0113   .0030   .1731
ZCOR:   1.1881  1.3153  1.4016  1.2073  3.5626   .0000  2.2398
ATWT:     .288    .016    .007    .012   4.555   6.364  52.168
```

Note that if excess oxygen from Fe is also present and reported, then that concentration needs to be added to the actual oxygen concentration after subtraction of the oxygen equivalent from the halogens.

Use Zero Point For Calibration Curve (Off-Peak Elements Only)

This analysis option is only applicable when using the Calibration Curve (multi-standard) option in the ZAF Selections dialog.

If this option is checked then the program will add an additional data point to the calibration curve fit of value 0,0. That is, zero counts and zero concentration. This option only applies to elements that have been acquired using off-peak backgrounds.

This option may be useful if some of the analyzed elements in the run have not been entered in the standard database with a concentration of zero and the user wishes to have the zero concentration data point included in the calibration curve fit.

Assign MAN Fits

This menu allows the user to display or modify the MAN (mean atomic number) background assignments and fits used for the background correction of elements in the currently open Probe for Windows database. The user has the option of using absorption corrected intensities and constant, linear or polynomial fits. An interactive graphical display is provided to facilitate to assignment process.

These MAN assignments are based on the element, x-ray, spectrometer number and crystal (and kilovolts and take-off angle) conditions and are applied globally to all quantitative samples in the run. If an element is analyzed on more then one set of x-ray, spectrometer number or crystal conditions, then the user is required to assign MAN fits for each additional set of conditions.

Be sure to click the Update button if any changes are made to the MAN assignments. This must be done after the changes to the MAN assignments to each element are made.

MAN background corrections can be utilized when the P/B is sufficiently high or when the use of fixed spectrometers are required. The MAN Fits menu allows the user to enter what MAN (mean atomic number) background samples will be used to calculate the background fit for the polynomial MAN iteration of an analyzed sample. The MAN background correction scheme is valid, because the primary cause of background (aside from trace contamination and peak over-lap interferences) is the average atomic number of the sample. If a number of pure samples of known atomic number are measured, then a function can be fit to the data and applied to the unknown sample. However, since the atomic number depends on the composition and that is unknown, then the MAN background must be applied during the ZAF iteration calculation.

Again, remember that MAN backgrounds can be used whenever the ratio of x-ray peak to background is high (>10:1), and this is usually the case with WDS microanalysis. Note that MAN backgrounds must be used for any fixed spectrometers. When measuring trace quantities be sure to use the off-peak measurements instead of the MAN correction.

MAN Backgrounds

There are two common methods for performing a background correction on WDS X-ray fluorescence data. The method most familiar to many probe users is the Off-Peak background correction method. PROBE uses this method and another called the MAN (for Mean Atomic Number) background correction.

This latter method is based on the fact that most of the background production in a sample is directly proportional to the average atomic number of the sample. If the average atomic number of the sample is low (for example, silicates) and the peak to background ratio is high, then the MAN background correction method works quite well down to minor element concentrations, since the total background is often less than one standard deviation of the x-ray signal.

The advantage of the MAN method is that it requires only a simple calibration of the analyzing channel over a range of atomic number that includes the atomic number range of the standards and/or samples. If many samples are to be measured for their major and even minor element concentrations, then substantial time may be saved using the MAN method. In addition, the use of fixed monochromators, requires MAN backgrounds for those channels, since they can not be adjusted off-peak.

However, if measuring high atomic number samples and/or trace concentrations, one will need to use the off-peak background correction method. See below for more on off-peak background corrections. Note that if no fixed spectrometers are used and only off-peak standards or unknown samples are acquired, then it is unnecessary to acquire or assign MAN background samples at all.

The Mean Atomic Number (MAN) background correction is an empirical calibration curve correction for background. Probe for Windows uses an iterated linear or polynomial fit of up to 10 background measurements to provide quantitative correction over a wide range of sample atomic numbers.

The technique is based on the fact that background is essentially a function of the average atomic number of a sample often referred to as "Z-bar". You can determine the Z-bar of any composition by using the "data" command in program Standard for Windows to print out a standard composition. You can of course also create a dummy composition for reference purposes and delete it later.

The program uses a graphical display of the measured counts on the background standards versus their Z-bars for each analyzed element to allow the user to select individual points for the calibration curve. Once the curves are fit the program can then calculate the background of any sample during the ZAF iteration based on the composition of the sample for a quantitative background correction.

Selecting Suitable Standards for the MAN Calibration

To start with you should have some idea of the range of Z-bar of your unknown samples. For silicates this will normally be in the 10.5 to 14 or 15 range, for oxides 10 or 12 to 20 or 22, for other samples use the Standard program to calculate the ranges or just look at a periodic table and estimate. You then select standards (usually simple synthetic oxides or metals of known purity) that will cover the range of Z-bar for both the standards and unknowns that you will be analyzing.

For example, in a silicate analysis of olivine the range of Z-bar will be from forsterite (10.577) to fayalite (18.69). Therefore you would select several standards in that range to use as MAN backgrounds including for instance MgO (Z-bar=10.4), Al_2O_3 (Z-bar=10.6), SiO_2 (Z-bar=10.8), TiO_2 (Z-bar=16.39) and NiO (Z-bar=21.17). These standards adequately cover the range of Z-bar for olivine analysis. Other pure oxides of similar Z-bar could, of course, be used also.

To begin manually collecting data on a MAN background (let's say MgO, which was given a standard number of 12 in the standard database), create a new sample from the Acquire! window. Click OK and the sample will display in the log window.

Note that "set 1" means the first time you have collected data for this standard sample in this run. You can now manually start the counters, and collect a series of lines or points of data. Each data line or point of data represents a single unit of counting time. These lines or points are grouped together into samples each containing 50 "lines" or less. All lines are numbered sequentially, and these line numbers are used frequently for reference purposes.

The line number, "status" of each line, beam counts and the x-ray counts on each channel are typed on the screen as the data is collected. The status of every line is initially "G" for good. If you see a bad line (epoxy, etc.) you can delete it at any time from the Analyze! window. This will change the status of that line to "B" for bad, and it will be ignored in all calculations. It may later be undeleted if you change your mind.

When you have collected a sufficient number (5-10) of good lines for this background sample, proceed to the next one. Note that as each point is acquired, the program will print out a summary of the current sample. The summary includes average counts, the standard deviation and the one sigma (predicted standard deviation) of the average counts on successive lines. If the scatter in the counts is due totally to counting randomness, the standard deviation of the number of counts should equal to or less than the one sigma values. If the standard deviation is much larger it indicates scatter due to other causes, such as sample inhomogeneity etc.

Continue collecting data on the MAN background samples until you are done. Although the background due to the continuum usually drifts very little, the program will automatically correct the MAN background correction for drift, if more than one set of MAN backgrounds are acquired. If it is suspected that the background is drifting, possibly due to a change in the operating vacuum for instance, simply acquire an additional set of MAN background samples and the program will automatically calculate a linear drift correction before each quantitative analysis. For a description on the form of the drift correction see the section above titled "Automatic Standard Drift Correction".

MAN Background Assignments

Once you have collected all the MAN (Mean Atomic Number) background samples you must decide which MAN backgrounds to use on each channel. MAN background corrections are made by assuming that background counts are a linear function of the mean atomic number of the sample. For each channel you may give the program up to 10 different backgrounds to fit. It will compute a second order polynomial (default) or force a straight line fit between background counts and MAN for these samples. These coefficients are then used to determine background counts on your unknowns, based on iterative approximations of the composition of the unknowns.

Be sure to click the Update button if any changes are made to the MAN assignments. This must be done after the changes to the MAN assignments to each element are made.

You will find that the MAN background corrections in PROBE work quite well down to approximately 200-300 ppm (0.02-0.03 weight percent) levels in silicate samples (z-bar ~10 - 15) using 10 to 20 second count times. Higher atomic number samples will of course be more difficult and may require longer count times or the use of off-peak background measurements.

Correction for Absorption of the Continuum

Probe for Windows will also automatically perform an absorption correction for the continuum background counts if it is specified. This correction is made using the ZAF or Phi-Rho-Z absorption correction selected by the user. Although it is not absolutely correct to use the absorption correction for characteristic lines to correct absorption of the continuum, it is a good approximation and since the background correction itself is a second order effect the difference is negligible (Donovan, 1996).

The correction for absorption is made by simply multiplying the absorption correction for each analyzed element in each MAN background standard times the measured counts before fitting to the calibration curve. This results in a MAN background calibration curve corrected for absorption. Then, before subtracting the background correction for the standards or unknowns, the program uncorrects the calculated background counts obtained from the calibration curve based on the absorption correction for that specific standard or unknown. This effect is usually very small, on the order of 100 ppm, however if a large absorption edge is present it could improve the MAN background correction somewhat.

Note that when comparing the background fit with the actual calculated background counts, be aware that because the MAN background fit is based on counts corrected for absorption, the calculated background for a particular sample is automatically uncorrected for the absorption in the sample, by dividing the calculated background from the fit by the absorption correction term, before subtracting the background counts from the measured raw intensity. This of course only applies when using the correction for absorption on the MAN fits.

Graphical Plot of Background vs. Z-bar

To tell the program which MAN backgrounds you want to use on which channels, click the MAN Fits under the Analytical menu. The program will load the standard count data and graphically plot the acquired MAN background samples using a default assignment. After selecting or deselecting (hold down the <ctrl> key while clicking) standards in the standard list box click the Update Fit button to save the changes. The program will then calculate and display the updated fit. You should avoid using samples which contain the channel element or produce an interference. These samples will show up as high points on the graph.

Note that the default selection of MAN standards by Probe for Windows is somewhat intelligent; if the standard database composition for the standard contains more than 0.01 weight percent of the element in question, then the program will not assign that standard in the initial plot. Of course, the user can still specify the standard explicitly by clicking it in, but remember, if the element is present then you are not actually measuring the background for that element. The "force linear fit" option can be used when you feel that the program is over-fitting the data.

You can assign the MAN fits as soon as you finish collecting data for the backgrounds, or just before you begin your first analysis. You can change the MAN assignments you have made at any time. If additional MAN backgrounds are necessary, add them to the run and acquire the additional data.

Clear All MAN Assignments

This menu will clear the current probe database of all MAN (mean atomic number) background assignments. This is generally only necessary, when after loading in a setup from another run, the user significantly modifies the element/spectrometer setup resulting in "orphaned" MAN assignments. In this case it is sometimes easier to simply clear the MAN assignments loaded with the file setup and re-assign them using the MAN Fits menu.

After clearing the existing MAN assignments, simply use the MAN Fits menu to load the default MAN assignments for the current element set in the run based on the loaded standards and then modify as necessary.

Use Off Peak Elements For MAN Fit

This menu will allow the program to utilize intensity data from standards that were acquired using off-peak backgrounds, for the MAN fit calibration. This means that elements in unknown or standard samples that were acquired using the MAN background acquisition option (no off-peaks), can utilize intensity data from standards that were acquired using the off-peak background acquisition option.

This is possible because an off peak element acquisition has the on-peak intensity acquired along with the off-peak intensities. When this option is utilized the off peak data is NOT subtracted from the on peak intensities for the purpose of the MAN assignments. Normally, only standards that have been acquired using the MAN background acquisition option are utilized for the MAN assignments.

To enable this option, one must click the Use Off Peak Elements For MAN Fit menu, then one must clear the MAN fits using the Clear All MAN Assignments menu, and then re-assign the MAN fits using the Assign MAN Fits menu.

Use MAN Correction For Off Peak Elements

This menu will allow the user to perform MAN background corrections on element data that was originally acquired using off-peak background methods (assuming that the MAN calibration curves are properly assigned). This option may require the acquisition of MAN standards for the MAN calibration and for user to clear the MAN fits and re-assign the MAN assignments to make sure that the off-peak elements are assigned MAN calibration curves.

This option is very powerful in that the user can switch between MAN and off-peak background corrections on the same sample dataset to see exactly what the difference between the two methods are. Once the off-peak elements have been properly assigned, there is no need to re-clear and re-assign the MAN assignments when subsequently using this menu option.

Empirical MACs

This menu allows the user to selectively load empirical MACs (mass absorption coefficients) from a supplied ASCII file or data entry.

Empirical MAC (Mass Absorption Coefficients) are stored in a file called EMPMAC.DAT in the Probe for Windows directory (usually C:\Program Files\Probe for Windows). If the user obtains any new empirical MAC data the file can be edited using any text editor such as NotePad or TextPad. Be careful to avoid adding any <tab> characters when editing the file (use space or comma delimited values only). The values can then be accessed using from the Analytical | Empirical MACs menu for runs that contains a matching emitter and absorber binary. Any changes to the default MAC values will be stored in your .MDB database file. Note that the elements may be in any order although they have been sorted to facilitate editing.

The format of the EMPMAC.DAT file is shown below :

```
  "b"     "ka"    "b"        3471         "Pouchou (1992)"
  "b"     "ka"    "c"        6750         "Pouchou (1992)"
  "b"     "ka"    "n"        11000        "Pouchou (1992)"
  "b"     "ka"    "o"        16500        "Pouchou (1992)"
  "b"     "ka"    "al"       64000        "Pouchou (1992)"
  "b"     "ka"    "si"       80000        "Pouchou (1992)"
  "b"     "ka"    "ti"       15000        "Pouchou (1992)"
  "b"     "ka"    "v"        18000        "Pouchou (1992)"
  "b"     "ka"    "cr"       20700        "Pouchou (1992)"
  "b"     "ka"    "fe"       27800        "Pouchou (1992)"
  "b"     "ka"    "co"       32000        "Pouchou (1992)"
  "b"     "ka"    "ni"       37000        "Pouchou (1992)"
  "b"     "ka"    "zr"       4400         "Pouchou (1992)"
  "b"     "ka"    "nb"       4500         "Pouchou (1992)"
  "b"     "ka"    "mo"       4600         "Pouchou (1992)"
  "b"     "ka"    "la"       2500         "Pouchou (1992)"
  "b"     "ka"    "ta"       23000        "Pouchou (1992)"
  "b"     "ka"    "w"        21000        "Pouchou (1992)"
  "b"     "ka"    "u"        7400         "Pouchou (1992)"
  "c"     "ka"    "b"        39000        "Pouchou (1992)"
  "c"     "ka"    "c"        2170         "Pouchou (1992)"
  "c"     "ka"    "si"       35000        "Pouchou (1992)"
  "c"     "ka"    "ti"       8097         "Pouchou (1992)"
  "c"     "ka"    "v "       8850         "Pouchou (1992)"
  "c"     "ka"    "cr"       10700        "Pouchou (1992)"
  "c"     "ka"    "fe"       13150        "Pouchou (1992)"
  "c"     "ka"    "zr"       25000        "Pouchou (1992)"
  "c"     "ka"    "nb"       24000        "Pouchou (1992)"
  "c"     "ka"    "mo"       20500        "Pouchou (1992)"
  "c"     "ka"    "hf"       18000        "Pouchou (1992)"
  "c"     "ka"    "ta"       17000        "Pouchou (1992)"
  "c"     "ka"    "w"        18000        "Pouchou (1992)"
  "n"     "ka"    "b"        15800        "Pouchou (1992)"
  "n"     "ka"    "n"        1640         "Pouchou (1992)"
  "n"     "ka"    "al"       13800        "Pouchou (1992)"
  "n"     "ka"    "si"       17000        "Pouchou (1992)"
  "n"     "ka"    "ti"       4270         "Pouchou (1992)"
  "n"     "ka"    "v "       4950         "Pouchou (1992)"
  "n"     "ka"    "cr"       5650         "Pouchou (1992)"
  "n"     "ka"    "fe"       7190         "Pouchou (1992)"
  "n"     "ka"    "zr"       24000        "Pouchou (1992)"
  "n"     "ka"    "nb"       25000        "Pouchou (1992)"
  "n"     "ka"    "mo"       25800        "Pouchou (1992)"
  "n"     "ka"    "hf"       14000        "Pouchou (1992)"
  "n"     "ka"    "ta"       15500        "Pouchou (1992)"
  "o"     "ka"    "li"       1600         "Bastin  (1992)"
  "o"     "ka"    "b"        8550         "Bastin  (1992)"
  "o"     "ka"    "b"        1200         "Bastin  (1992)"
  "o"     "ka"    "f"        1850         "Love et al. (1974)"
  "o"     "ka"    "ne"       2750         "Love et al. (1974)"
  "o"     "ka"    "na"       3630         "Love et al. (1974)"
  "o"     "ka"    "mg"       5170         "Bastin  (1992)"
  "o"     "ka"    "al"       6720         "Bastin  (1992)"
  "o"     "ka"    "si"       8790         "Bastin  (1992)"
  "o"     "ka"    "p"        9820         "Love et al. (1974)"
  "o"     "ka"    "s"        12400        "Love et al. (1974)"
```

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"o"     "ka"    "cl"       14300        "Love et al. (1974)"
"o"     "ka"    "ar"       16100        "Love et al. (1974)"
"o"     "ka"    "k"        20500        "Love et al. (1974)"
"o"     "ka"    "ca"       24600        "Love et al. (1974)"
"o"     "ka"    "sc"       26800        "Love et al. (1974)"
"o"     "ka"    "ti"       19900        "Bastin  (1992)"
"o"     "ka"    "cr"       2900         "Bastin  (1992)"
"o"     "ka"    "mn"       3470         "Bastin  (1992)"
"o"     "ka"    "fe"       4000         "Bastin  (1992)"
"o"     "ka"    "co"       4500         "Bastin  (1992)"
"o"     "ka"    "ni"       5120         "Bastin  (1992)"
"o"     "ka"    "cu"       5920         "Bastin  (1992)"
"o"     "ka"    "zn"       6350         "Bastin  (1992)"
"o"     "ka"    "ga"       7090         "Bastin  (1992)"
"o"     "ka"    "y"        15100        "Bastin  (1992)"
"o"     "ka"    "zr"       16200        "Bastin  (1992)"
"o"     "ka"    "nb"       17100        "Bastin  (1992)"
"o"     "ka"    "mo"       18000        "Bastin  (1992)"
"o"     "ka"    "ru"       19700        "Bastin  (1992)"
"o"     "ka"    "sn"       15050        "Bastin  (1992)"
"o"     "ka"    "ba"       4560         "Bastin  (1992)"
"o"     "ka"    "la"       3600         "Bastin  (1992)"
"o"     "ka"    "ta"       10600        "Bastin  (1992)"
"o"     "ka"    "w "       11300        "Bastin  (1992)"
"o"     "ka"    "pb"       11000        "Bastin  (1992)"
"o"     "ka"    "bi"       12100        "Bastin  (1992)"
"f"     "ka"    "xe"       27300        "Henke et al. (1982)"
"na"    "ka"    "zn"       10500        "Henke et al. (1982)"
"na"    "ka"    "pm"       7920         "Henke et al. (1982)"
"mg"    "ka"    "tb"       8240         "Henke et al. (1982)"
"al"    "ka"    "tm"       4730         "Henke et al. (1982)"
"cr"    "la"    "te"       39500        "Henke et al. (1982)"
"co"    "la"    "co"       1920         "Henke et al. (1982)"
"ge"    "ka"    "ta"       265          "Henke et al. (1982)"
"ge"    "la"    "gd"       8500         "Henke et al. (1982)"
"mo"    "la"    "hg"       951          "Henke et al. (1982)"
```

The first column ("b" in the example equals Boron) is the atomic symbol of the emitting element. The second column ("ka" in the example equals Kα) is the x-ray line of the emitting element. The third column is the atomic symbol of the absorber element. The element and x-ray symbols must be enclosed in double quotes. The fourth column is the actual mass absorption coefficient (MAC) that has been experimentally measured. The last parameter is a comment string that may contain a comment, usually the experimenter or attribution. The comment string must be in double quotes.

The first and third columns (emitting atomic number and absorber atomic number) must be integer values from 1 to 94. The second column (x-ray line type) must be an integer value from 1 to 3. The fourth column (MAC value) must be a real number greater than zero.

See Appendix C below for a large set of empirical mass absorption coefficients from a number of sources that indicates some of the variation among MACs from various sources.

Empirical APFs

This menu allows the user to selectively load empirical APFs (Area Peak Factors) from a supplied ASCII file or data entry.

A powerful feature in Probe for Windows allows the user to select an Area-Peak-Factor (APF) correction for use in correcting x-ray intensities for peak shift and shape changes between the standard and the unknown. This can occur especially with Kα lines of the light elements such as oxygen, nitrogen, carbon and boron. With these elements, differences in the bond energies between say TiC and SiC will produce significantly different peak shapes resulting in systematic errors in the analysis of these materials when only the peak x-ray intensities are used.

For example, when analyzing oxygen and using MgO or Fe2O3 as an oxygen standard, analyses of SiO2 will produce errors up to about 4.5%. This effect is independent of the matrix correction and can be corrected for only by the use of the appropriate APFs.

The APF concept was proposed by Bastin and Heijligers (Bastin and Heijligers, 1986) to provide a simple correction scheme for this effect. It basically requires measuring the ratio of the peak intensity to the integrated intensity between a primary standard to a number of secondary standards on the light element spectrometer used. The wavelength scan feature in Probe for Windows can be used to acquire the peak shape profiles. After correcting for background and removal of extraneous peaks from interfering lines, the APF can be calculated. The calculation of the APF factor is shown below :

$$\mathrm{APF} = \frac{I_U^I \cdot I_S^P}{I_U^P \cdot I_S^I}$$

Where : I_U^I is the integrated intensity of the secondary standard

I_S^P is the peak intensity of the primary standard

I_U^P is the peak intensity of the secondary standard

I_S^I is the integrated intensity of the primary standard

The peak shape and shift of light element peaks can be easily demonstrated using MgO and SiO_2 standard samples. First select a suitable layered dispersive element (LDE) analyzing crystal for oxygen Kα analysis. Tune the spectrometer using the MgO standard and acquire a standard sample for MgO. Assign MgO as the calibration standard for oxygen. Then move to the SiO_2 standard and acquire another quantitative standard sample on the SiO_2 sample. Note that the analysis of SiO_2 using MgO as a calibration standard will result in a low total for the SiO_2, somewhere around 96-97 %. This is not an error in the mass absorption coefficient (MAC), and no attempt should be made to correct this problem by entering an arbitrary MAC value.

Instead, the proper APF correction will need to be applied as shown below. Although the values below are relative to Fe_2O_3, you will note that the APF for MgO is 1.000, which means that these correction factors apply equally well relative to MgO. In the Oxygen Area Peak Factor table below you will note that oxygen in the presence of SiO_2 needs to be corrected by a factor of 1.0444 when MgO or Fe_2O_3 is used as a calibration standard. After entering this APF correction factor for the oxygen channel and re-analyzing, note that the total is now close to 100 %.

It is very important to note that the APF values selected or entered are always measured relative to some standard sample. For example, if measuring C Kα and using Fe_3C as the primary standard for carbon, then any C Kα APF values used must be those measured relative to Fe_3C. For the same reason, if using APF values for a particular (light) element and one decides to re-assign the standard for that element, the APF values for that element must also be changed to reflect the standard re-assignment. See the section above for information on re-calculating the APF values relative to another standard.

For the above reason it is usually most efficient to simply always use the same standard for each light element analyzed. Typically (in order to utilize the APF values in the supplied EMPAPF.DAT file) these will be :

- Oxygen : MgO or Fe2O3
- Nitrogen : AlN
- Carbon : Fe3C
- Boron : B metal

The APF correction in Probe for Windows NT will allow the user to enter empirical APF values in each run. The user may enter one or more APF factors for each emitting element although they are generally applied to soft x-ray lines. Note however that even S Kα exhibits peak shift and shape changes when comparing sulfide and sulfate peaks. The APF for each absorber will be summed according to it's weight fraction in the composition and applied to the emitting element counts during the ZAF or Phi-Rho-Z iteration.

If it is desired to correct a complete unknown for peak shape changes, simply perform a wavelength scan on the standard being used for the light element emitter (oxygen, carbon, etc.) and also on the unknown. Calculate the APF using the expression above and from the Analytical | Empirical APFs menu, enter the same APF for all absorbers in the unknown. The program will then sum the APFs which will be the same as the single measured APF. Note that this APF may only apply to a single composition. One can perform additional wavelength scans on other samples to confirm this however.

The APF correction values are defined in the EMPAPF.DAT file in the Probe for Windows directory (usually C:\Program Files\Probe for Windows). The file contains some 50 or 60 values that may or may not be applicable, depending on the analyzing crystals and standards available. The user may edit the file using any ASCII text editor such as NotePad or TextPad to insert their own measurements. Be careful to avoid adding any <tab> characters when editing the file (use space or comma delimited values only). Note that the elements may be in any order although they have been sorted to facilitate editing.

The format of the EMPAPF.DAT file is shown below :

```
"b"     "ka"    "c"       1.02         "B4C/B/STE"
"b"     "ka"    "n"       1.2          "BN/B/STE"
"b"     "ka"    "al"       1.12         "AlB2/B/STE"
"b"     "ka"    "al"       1.01         "AlB12/B/STE"
"b"     "ka"    "si"       1            "SiB3/B/STE"
"b"     "ka"    "si"       .92          "SiB6/B/STE"
"b"     "ka"    "ti"       .75          "TiB/B/STE"
"b"     "ka"    "ti"       .88          "TiB2/B/STE"
"b"     "ka"    "v"       1            "VB2/B/STE"
"b"     "ka"    "cr"       .9           "CrB/B/STE"
"b"     "ka"    "cr"       1.1          "CrB2/B/STE"
"b"     "ka"    "fe"       1.1          "FeB/B/STE"
"b"     "ka"    "fe"       1.25         "Fe2B/B/STE"
"b"     "ka"    "co"       1.2          "CoB/B/STE"
"b"     "ka"    "co"       1.02         "Co2B/B/STE"
"b"     "ka"    "ni"       1.2          "NiB/B/STE"
"b"     "ka"    "ni"       1.06         "Ni2B/B/STE"
"b"     "ka"    "ni"       .98          "Ni3B/B/STE"
"b"     "ka"    "zr"       .8           "ZrB2/B/STE"
"b"     "ka"    "nb"       .8           "NbB/B/STE"
"b"     "ka"    "nb"       .9           "NbB2/B/STE"
"b"     "ka"    "mo"       .94          "MoB/B/STE"
"b"     "ka"    "la"       .9           "LaB6/B/STE"
"b"     "ka"    "ta"       .88          "TaB/B/STE"
"b"     "ka"    "ta"       1.1          "TaB2/B/STE"
"b"     "ka"    "w"       .98          "WB/B/STE"
"b"     "ka"    "u"       1.04         "UB4/B/STE"
"c"     "ka"    "b"       1.01         "B4C/Fe3C/WSi/59.8"
"c"     "ka"    "si"       .933         "SiC/Fe3C/WSi/59.8"
"c"     "ka"    "ti"       .868         "TiC/Fe3C/WSi/59.8"
"c"     "ka"    "v"       .873         "V2C/Fe3C/WSi/59.8"
"c"     "ka"    "v"       .873         "VC/Fe3C/WSi/59.8"
"c"     "ka"    "cr"       .8           "Cr7C3/Fe3C/STE"
"c"     "ka"    "cr"       .83          "Cr3C2/Fe3C/STE"
"c"     "ka"    "cr"       .8           "Cr23C6/Fe3C/STE"
"c"     "ka"    "zr"       .88          "ZrC/Fe3C/WSi/59.8"
"c"     "ka"    "nb"       .79          "NbC/Fe3C/STE"
"c"     "ka"    "mo"       .82          "Mo2C/Fe3C/STE"
"c"     "ka"    "hf"       .83          "HfC/Fe3C/STE"
"c"     "ka"    "ta"       .96          "TaC/Fe3C/STE"
"c"     "ka"    "w"       .97          "WC/Fe3C/STE"
"c"     "ka"    "w"       1.02         "W2C/Fe3C/STE"
"n"     "ka"    "si"      1.103          "Si3N4/AlN/WSi/59.8"
"n"     "ka"    "ti"       .997          "TiN/AlN/WSi/59.8"
"n"     "ka"    "v"       1.0226         "VN/AlN/WSi/59.8"
"n"     "ka"    "cr"      1.018          "Cr2N/AlN/WSi/59.8"
"n"     "ka"    "fe"      1.012          "Fe2N/AlN/WSi/59.8"
"n"     "ka"    "zr"       .9952         "ZrN/AlN/WSi/59.8"
"n"     "ka"    "hf"      1.002         "HfN/AlN/WSi/59.8"
"o"     "ka"    "b"       1.0628       "B6O/Fe2O3/WSi/59.8"
"o"     "ka"    "mg"       1            "MgO/Fe2O3/WSi/59.8"
"o"     "ka"    "al"       1.0213       "Al2O3/Fe2O3/WSi/59.8"
"o"     "ka"    "si"       1.0444       "SiO2/Fe2O3/WSi/59.8"
"o"     "ka"    "ti"       .9796        "TiO2/Fe2O3/WSi/59.8"
"o"     "ka"    "cr"       .993         "Cr2O3/Fe2O3/WSi/59.8"
```

```
"o"     "ka"    "mn"       1.0121       "MnO/Fe2O3/WSi/59.8"
"o"     "ka"    "fe"       .9962        "Fe3O4/Fe2O3/WSi/59.8"
"o"     "ka"    "co"       1.0133       "CoO/Fe2O3/WSi/59.8"
"o"     "ka"    "ni"       1.0153       "NiO/Fe2O3/WSi/59.8"
"o"     "ka"    "cu"       .9946        "Cu2O/Fe2O3/WSi/59.8"
"o"     "ka"    "cu"       .9943        "CuO/Fe2O3/WSi/59.8"
"o"     "ka"    "zn"       .9837        "ZnO/Fe2O3/WSi/59.8"
"o"     "ka"    "ga"       1            "Ga2O3/Fe2O3/WSi/59.8"
"o"     "ka"    "zr"       .9823        "Y3Fe5O12/Fe2O3/WSi/59.8"
```

The first column (in the example is "b" for Boron) is the atomic symbol of the emitting element. The second column ("ka" in the example equals Kα) is the x-ray line of the emitter. The third column is the atomic symbol of the absorber element. The fourth column is the actual area peak factor (APF) that has been experimentally measured. The last parameter is a string that contains the conditions under which the APF was measured. For example, the comment "B4C/B/STE" indicates that the peak and integrated intensities for the APF were measured on a B_4C (boron carbide) sample, relative to a B (elemental Boron) primary standard, using a Pb Stearate analyzer. The reason that this information is needed is because the APF is a relative measurement (to a standard) and the value is dependent on the resolution (crystal type) used. If a LDE (layered dispersive element) analyzer is used, the 2d of the analyzer should be indicated also as seen in the last two lines of the example.

The first and third columns (emitting atomic symbol and absorber atomic symbol) must be valid element symbols. The second column (x-ray line) must be "ka", "la" or "ma". All element and x-ray symbols must be enclosed in double quotes. The fourth column (APF value) must be a real number greater than 0.0. The comment string must be enclosed in double quotes.

These values can then be loaded from the Analytical | Empirical APFs menu item in Probe for Windows. Note that the comments listed after each APF value can be used to indicate the experimental conditions under which the APF was measured. For consistency sake, the formula of the compound measured is listed first, then the primary reference standard used and finally the analyzing crystal type (and 2d spacing if desired) used. Edit the EMPAPF.DAT file in the Probe for Windows directory to add additional APFs from your own measurements.

The following is a short listing of some typical APFs taken from Bastin and Heijligers (1986). Because the factors are spectrometer and crystal dependent these should be used as a guide only.

Oxygen Area Peak Factors (APF)

Relative to Fe_2O_3 or MgO using W/Si LDE (2d = 59.8)

Oxide	Area-Peak Factor (APF)
B_6O	**1.0628**
MgO	**1.0000**
Al_2O_3	**1.0213**
SiO_2	**1.0444**
TiO_2	**0.9796**
Cr_2O_3	**0.9930**
Mn_3O_4	**1.0121**
Fe_3O_4	**0.9962**
CoO	**1.0133**
NiO	**1.0153**
Cu_2O	**0.9946**
CuO	**0.9943**
ZnO	**0.9837**

Ga_2O_3	**1.0000**
$Y_3Fe_5O_{12}$	**0.9823**

Carbon Area-Peak Factors

Relative to Fe_3C using W/Si LDE (2d = 59.8):

Carbide	Area-Peak-Factor (APF)
B_4C	**1.010**
SiC	**0.933**
TiC	**0.868**
V_2C	**0.873**
VC	**0.873**
ZrC	**0.880**

Nitrogen Area-Peak Factors

Relative to AlN using W/Si LDE (2d = 59.8):

Carbide	Area-Peak-Factor (APF)
AlN	**1.000**
Si3N4	**1.103**
TiN	**.997**
VN	**1.0226**
Cr2N	**1.018**
Fe2N	**1.012**
ZrN	**.9952**
HfN	**1.002**

ZAF Selections

This menu allows the user to modify or display the ZAF or Phi-Rho-Z quantitative matrix correction options for all samples. The default selection is the Phi-Rho-Z correction optimized by John Armstrong for silicates. It is often the best choice for most compositions. The user will almost always find the default set of corrections satisfactory. Any changes to the default ZAF or Phi-Rho-Z selections will be saved along with any empirical MAC or APF data for the next time the run file is re-opened.

Alpha-Factors

Alpha-Factor corrections based on ZAF or Phi-Rho-Z calculated k-ratio intensities and empirical a-factors from an ASCII disk file are also supported. There are two main reasons for this : first, alpha-factor corrections are very simple (once calculated for a run condition) and are therefore extremely fast. This is particularly useful when recalculating large (> 100-1000) data analyses.

Second, because it has been noted (Armstrong, 1988) that almost all reasonable ZAF or Phi-Rho-Z calculations will produce 2nd order polynomial fit alpha-factors with very similar slope and curvature terms (differing mainly in the intercept term), the use of a single binary standard empirically determined k-ratio allows the analyst to accurately construct an exact calibration curve for especially difficult matrices. This is easily accomplished by editing a single ASCII file (EMPFAC.DAT) for the empirically determined alpha-factor terms measured and fit by the user. The alpha-factor correction is summarized by the following expression :

$$C_A^u = \frac{C_A^s}{\mathrm{Beta}_{\lambda_A}^s} \frac{I_A^u(\lambda_A)}{I_A^s(\lambda_A)} \mathrm{Beta}_{\lambda_A}^u$$

where : $\mathrm{Beta}_{\lambda_A}^u$ is the unknown beta-factor

$\mathrm{Beta}_{\lambda_A}^s$ is the standard beta-factor

Use of Empirical Alpha-Factors

To utilize empirically determined Alpha-Factor terms, simply edit the EMPFAC.DAT file in the Probe for Windows directory (usually C:\Program Files\Probe for Windows). If an Alpha-factor entry in the file matches the run conditions of the current run will can be used to overwrite the previously calculated alpha-factor terms. The format of the EMPFAC.DAT file is described below :

```
 52.5  20.0  1  "au"  "la"  "ag"  1.142   0.000   0.000 "NBS Spec. Pub. 260-28"
 52.5  20.0  1  "ag"  "la"  "au"  1.483   0.000   0.000 "NBS Spec. Pub. 260-28"
 52.5  20.0  1  "au"  "la"  "cu"  1.347   0.000   0.000 "NBS Spec. Pub. 260-28"
 52.5  20.0  1  "cu"  "ka"  "au"  0.758   0.000   0.000 "NBS Spec. Pub. 260-28"
 52.5  20.0  2  "au"  "la"  "ag"  1.159  -0.056   0.000 "NBS Spec. Pub. 260-28"
 52.5  20.0  2  "ag"  "la"  "au"  1.505  -0.071   0.000 "NBS Spec. Pub. 260-28"
 52.5  20.0  2  "au"  "la"  "cu"  1.347  -0.014   0.000 "NBS Spec. Pub. 260-28"
 52.5  20.0  2  "cu"  "ka"  "au"  0.759  -0.007   0.000 "NBS Spec. Pub. 260-28"
 52.5  20.0  3  "au"  "la"  "ag"  1.121   0.127  -0.179 "NBS Spec. Pub. 260-28"
 52.5  20.0  3  "ag"  "la"  "au"  1.458   0.170  -0.247 "NBS Spec. Pub. 260-28"
 52.5  20.0  3  "au"  "la"  "cu"  1.332   0.061  -0.075 "NBS Spec. Pub. 260-28"
 52.5  20.0  3  "cu"  "ka"  "au"  0.740   0.089  -0.096 "NBS Spec. Pub. 260-28"
```

Each line contains a single empirical alpha-factors fit for half of a binary pair (one emitter-absorber system per line) for various take-off angles, operating voltage, elements, x-ray lines, etc.

Columns 1 and 2 contain the takeoff angle (degrees) and operating voltage (KeV) that the alpha-factor was measured at. Column 3 is the fit flag (1 = constant expression, 2 = linear fit and 3 = polynomial fit). Columns 4 and 5 are the emitter element symbols and x-ray lines. Column 6 is the absorber element symbols. Note that Probe for Windows only supports the use of elemental alpha-factors. That is, referenced to elemental end-members as opposed to oxide end-members.

The three remaining columns are the alpha-factor terms. Note that constant alpha-factors have only one term, linear alpha-factors have two terms and non-linear or polynomial fit alpha-factors have three terms. For the non-linear alpha-factors, the first term is the intercept, the second term is the slope and the third term is the curvature term.

Empirical alpha-factors are calculated from experimentally measured k-ratios using a set of known binary (two element) standards covering a range of composition, by fitting the concentrations and intensity data to a plot of α vs. C, where C represents the standard concentrations and α is defined by the following expression :

$$\frac{\left(\frac{C}{K}\right)-C}{(1-C)}=\alpha$$

Each binary data set can be fit to any or all of the alpha-factor fits, that is, constant (single term at 50/50 composition), linear (two terms) or polynomial (three terms).

Calibration Curve

In addition, Probe for Windows offers a sophisticated calibration curve based on a 2nd order polynomial fit to multiple standard intensities. This option is best employed in special situations such as the analysis of trace carbon or nitrogen in metal alloys and when a suitable set of standards are available. Probe for Windows will calculate a separate calibration curve for off-peak and MAN acquired data (MAN acquired data is not corrected for background with the Calibration Curve). If a standard is available that contains a zero concentration of the element, it will also be included in the Calibration Curve fit.

Generally, for best results, it is preferred to have a set of 4 or more standards with a similar matrix to the unknown and a range of the element of interest that brackets the unknown concentration. Probe for Windows will construct a separate set of calibration curves for both off-peak and MAN element data and fit a 2nd order polynomial expression that is used in the iteration procedure to determine the concentration of the element.

Elements calculated by difference or stoichiometry can be calculated also along with the calibration curve corrected elements. The program will print a warning if this correction method is selected.

ZAF Matrix Corrections

ZAF matrix corrections (included here are also the newer Phi-Rho-Z corrections) are a fundamental calculation of the x-ray intensities based on physical models of the atomic and electron interaction. It is the most accurate way available to correct for matrix effects without using an empirical calibration curve. It is also very calculation intensive and for that reason it helps to have a fast CPU when analyzing a large number of elements or samples. The following expression summarizes the ZAF correction :

$$C_A^u = \frac{C_A^s}{[ZAF]_{\lambda_A}^s}\frac{I_A^u(\lambda_A)}{I_A^s(\lambda_A)}[ZAF]_{\lambda_A}^u$$

where : $\frac{C_A^s}{[ZAF]_{\lambda_A}^s}\frac{I_A^u(\lambda_A)}{I_A^s(\lambda_A)}$ is the unknown k-ratio and

$[ZAF]_{\lambda_A}^u$ is the ZAF correction factor of the unknown

and : $I_A^u(\lambda_A)$ is the unknown intensity for element A at λ

$I_A^s(\lambda_A)$ is the standard intensity for element A at λ

C_A^s	is the concentration of the element in the standard
$[\,ZAF\,]_{\lambda_A}^s$	is the ZAF correction for the element in the standard

The following is taken from John Armstrong's CITZAF documentation on how to select among the various matrix correction options :

1. Choice of absorption corrections (ABSCOR's):

1 ... ABSORPTION CORRECTION OF PHILIBERT
2 ... ABSORPTION CORRECTION OF HEINRICH/AN. CHEM.
3 ... ABSORPTION CORRECTION OF HEINRICH/1985 MAS [Heinrich, Microbeam Analysis--1985, 79]
4 ... ABSORPTION CORRECTION OF LOVE/SCOTT -- 1983 [e.g., Love & Scott, J. Phys. D. 11, 1369 (1978)]
5 ... ABSORPTION CORRECTION OF LOVE/SCOTT I -- 1985 [Sewell, Love & Scott, J.Phys.D. 18, 1233 (1985)]
6 ... ABSORPTION CORRECTION OF LOVE/SCOTT II -- 1985 [Sewell, Love & Scott, J.Phys.D. 18, 1233 (1985)]
7 ... PHI(PZ) ABS. CORRECTION OF PACKWOOD/BROWN 1982/XRS [Brown & Packwood, X-ray Spec. 11, 187 (1982)]
8 ... PHI(PZ) ABSORPTION CORRECTION OF BASTIN 1984/XRS [e.g., Bastin et al., X-ray Spec. 13, 91 (1984)]
9 ... PHI(PZ) ABS. CORRECTION OF ARMSTRONG (P/B) 1981/MAS [Armstrong, Microbeam Analysis--1984, 208;--1982, 175]

The conventional ZAF correction is #1 which is also used in the FRAME program developed by Kurt Heinrich at NIST (NBS). The best results for silicates and other oxide samples are obtained with the Phi(Rho-Z) correction (#9) and with the original Love/Scott correction (#4). Correction #3 should not be used until revisions are made. The default selection is #9.

2. Choice of atomic number corrections:

1 ... ATOMIC NUMBER CORRECTION OF DUNCUMB/REED(FRAME)
2 ... ATOMIC NUMBER CORRECTION OF LOVE/SCOTT
3 ... ATOMIC NUMBER CORRECTION OF BROWN/BASTIN/JTA PHI(PZ)
4 ... ATOMIC NUMBER CORRECTION OF BROWN/BASTIN/JJD (Z^.85)

The conventional correction is #1. Corrections #1 and #2 give similar results. When using #2, also use the Love/Scott expressions for backscatter coefficients and Phi(0). When using #1, use the Heinrich backscatter coefficients and Reuter Phi(0). Correction #3 may only be employed when one of the Phi(Rho-Z) absorption corrections are being used. Even then, it does not give as good results as #1 or #2 for silicates and oxides and it is seldom used. A new option is #4 which is the standard Duncumb/Reed correction with a modified backscatter loss calculation based on modified electron fractions as opposed to mass fractions. See Donovan and Westphal (2000). This modification is using a Z^0.85 power scaling to compensate for nuclear screening effects. This modified calculation works especially well when the standard and unknown are very different in average Z. The default selection is #2.

3. Choice of backscatter coefficients:

1 ... BACKSCATTER CORRECTION OF HEINRICH
2 ... BACKSCATTER CORRECTION OF LOVE/SCOTT

The default selection for backscatter is #2.

4. Choice of mean ionization potentials:

1 ... MEAN IONIZATION POTENTIALS OF BERGER/SELTZER
2 ... MEAN IONIZATION POTENTIALS OF DUNCUMB/DA CASA

Calculation #2 should only be used if the Philibert (#1) absorption correction and Duncumb-Reed atomic number (#1) correction are employed. Selection #1 is the default.

5. Choice of Phi(0) expressions: (Not applicable if one of the ZAF corrections (#1-6) was selected for the absorption correction.)

1 ... PHI(0) EQUATION OF REUTER
2 ... PHI(0) EQUATION OF LOVE/SCOTT
3 ... NONE

Both #1 and #2 give similar results. The default selection is #2.

Fluorescence Correction

Probe for Windows uses as a default, a modified form of the REED/ARMSTRONG fluorescence from John Armstrong's CITZAF program. The two modifications are the addition of a fluorescence by M line correction, calibrated using data from Heinrich on Nb and U binary alloys and a modification of the fluorescence intensity (Armstrong, Microbeam Analysis, 1988). The analyst may also select the exact fluorescence correction from Armstrong's CITZAF code without the M line fluorescence or Armstrong ionization term. Also provided is a fluorescence correction with the fluorescence by M lines but without the Armstrong ionization term and a fluorescence correction with the Reed intensity expression.

Mass Absorption Coefficients

Probe for Windows provides four automatic lookup tables of mass absorption coefficients for use in the analytical calculations.

LINEMU.DAT is a compilation of Henke (unpublished, 1985) coefficients for energies less than 10 KeV and Heinrich (1966) for energies greater than 10 KeV.

CITZMU.DAT is the complete set of coefficients from Heinrich used by John Armstrong at CalTech/NIST which is from Heinrich (1966) and Henke and Ebisu for the light elements.

MCMASTER.DAT is a calculated set derived from equations (fits) from McMaster at Livermore National Laboratory (modified by Rivers).

MAC30.DAT is from Heinrich (1987), which is a fit to the Goldstein, et. al. MAC table.

MACJTA.DAT is from Armstrong's MACCALC program.

The default mass absorption coefficient file is LINEMU.DAT. This default MAC file may be specified in the PROBEWIN.INI file. If one or more of the mass absorption coefficients in these tables are not acceptable, the user can select the use of other empirically derived values from the Analytical | Empirical MACs menu.

Create Virtual Standard Intensity

A new option is the use of "virtual" standard intensities. These are calculated intensities based on interpolation and matrix corrected similar (atomic numbers) elements measured on the same crystal, spectrometer and voltage as the unknowns. This may be useful in cases where a physical standard is not available (e.g., Ar).

To calculate a virtual standard intensity, first decide on the element and create a "virtual" standard composition using the Standard database application. This will be a composition that does not physically exist in the microprobe laboratory (e.g., pure iodine). Then decide what x-ray line, spectrometer, crystal and kilovolts to use for the unknown measurement. These will be actual measurements on the unknown (usually trace concentrations). It may not be possible to peak the spectrometer properly without a major concentration of the element in a standard, so the use of a multiple peaks calibration procedure is suggested (see program StartWin for details) for getting the spectrometer position calibrated. Set the PHA parameters as for x-ray lines of similar energy.

Enter these parameters into the Virtual Element/Spectrometer Parameters frame. Then using the same x-ray line, spectrometer, crystal, and kilovolts, measure an available element on either side (in atomic number space) of the element that is used for the virtual standard intensity. For example if creating a virtual Ar intensity, it might useful to measure K and Ca since they are close to Ar.

Enter the chosen fit elements, intensities and standards in the Fit Elements, Intensities and Standards dialog and click the Add button. The program will calculate the virtual intensity and store it in a table in the database file. To utilize this intensity for standardizing an unknown, go to the Analyze window and click the Standard Assignments button, click the virtual element and select the virtual standard from the standard list and check the Use Virtual Standard for Virtual Intensity Calculation checkbox.

Student's "t" Table

This menu item provides a listing for typical confidence levels of Student's "t" values. These values are used in many statistical calculations. See Calculate Detection Limits and Homogeneity above for an example of this parameter in use.

Run

This menu will output a variety of run information in summary format to the log window. This information can then be cut and pasted to another application or simply selected and printed using the File | Print Log menu.

List Run Summary

This menu will output a summary of all major run and sample parameters (based on the current sample) to the log window. This summary may be worth printing out for record keeping purposes.

List Sample Names

This menu will output a summary of all standard, unknown and wavescan samples to the log window. Note that the first column of numbers listed here are the sample row numbers of the samples, which is also used as a pointer for saving and loading sample setups within the run.

List Standard Counts

This menu will output a summary of all standard counts in the order acquired to the log window. This includes all standards, whether or not they are actually assigned as a standard for primary, interference or MAN calibrations. The average time of acquisition is also listed in the output.

To see a listing of the standard intensities that are applicable to a specific quantitative sample, see the List Report in the Analyze! window.

List Standard Compositions

This menu item will list the compositions of all standards in the current run based on the current values in the standard composition database.

List Current MACs

This menu item will list the currently loaded MACs (mass absorption coefficients) from the last quantitative analysis. Note that any empirical MACs listed from this menu will be used to overwrite table based MACs automatically during calculations.

List Current APFs

This menu item will list the currently loaded APFs (area peak factors) from the last quantitative analysis.

Output

Log Window Font

This menu allows the user to modify the current log window font type, size and appearance using the font Common dialog. It is strongly recommended to use a non-proportional font, such a New Courier, so that numeric column data is properly aligned in the log window.

Note that the default font and size of the log window may be permanently defined in the PROBEWIN.INI file.

Debug Mode

This menu toggles the program in and out of Debug mode. In Debug mode, the program generates an abundance of additional output to the log window for the purposes of debugging the program or viewing the analytical calculations in greater detail.

Debug mode is also useful to check the low level commands sent to the hardware interface. Note that in Debug mode the Form level events are not protected against unanticipated errors.

Extended Format

This menu toggles the output option for printout to the log window of element data. If unchecked, the program automatically wraps element data output to 8 elements per line. Note that wavescan data is wrapped 4 elements per line. If this option is checked, the program does not wrap element data to the log window or disk file (if Save To Disk option is enabled), instead the data is written out as far as necessary to the right.

Save To Disk Log

This menu toggles the output option to mirror all log window output to a user specified text file. This may be useful for importing text or data from the log window to another non-Windows program. The default extension is .OUT but another extension may be entered by the user.

If a large amount of output is to be generated, this option will capture all output to the log window so that it can be printed out afterwards. If the output file selected by the user already exists, the program offers the option to overwrite or append subsequent output to the already existing file.

View Disk Log

This menu invokes the file viewer specified in the PROBEWIN.INI file (default file viewer is NotePad) to view the previously or currently opened disk log file.

If the file is currently open, the program will first close the disk log file before viewing it. Note that because the default file viewer (NotePad) is somewhat limited, it may be necessary to specify a more robust file viewer such as TextPad or Word for Windows. See the section on editing the PROBEWIN.INI file above to change the default file viewer.

Load Custom Position Format #1

This menu will load a custom position file format (#1). This format is a fixed length field, comma delimited ASCII file that contains off-line digitized data from an automated light microscope. This format is intended for use with Canadian Geological Survey laboratory .LEP position files only.

See additional information on the "Import From ASCII File" button in the Automate! window and also the PositionImportExportFileType parameter in the PROBEWIN.INI file.

Save Custom Analysis Format #1

This menu will automatically create a custom file format. This format is a fixed length field, comma delimited ASCII file that contains unknown weight percent data only. The data will be output to a different file based on the analytical setup used to create the sample (if one was used). The file naming convention is based on the file setup name used to create the sample. For example, samples created using a setup file called FELDSPAR.MDB, will be output to FELDSPAR.DAT, and so on.

The first column contains an eight character wide field containing the sample name for each data point. The final column contains the totals. All elements, analyzed and specified will be output. The column labels are not enclosed in double quotes but the sample names are. The oxide or elemental basis for calculating the data is based on the first sample of a particular setup file for ensure that the data type is consistent within a single output file.

This format is intended for use with the Canadian Geological Survey MREPORT mineral recalculation program set which has been published as a open file report. An short example is shown here:

```
  SAMPLE   ,      BI,      SB,   TOTAL
 "mabisb06", 99.7657, .207527, 99.9733
 "mabisb06", 99.4798, .185703, 99.6655
 "mabisb06", 99.1180, .298709, 99.4167
 "mabisb75", 99.4394, .978249, 100.418
 "mabisb75", 98.2293, 1.69337, 99.9227
 "mkbisb06", 98.0328, 1.69929, 99.7321
 "mkbisb06", 97.5753, 1.45358, 99.0289
 "mkbisb06", 98.8127, 1.78520, 100.598
 "msbisb25", 89.4619, 10.3681, 99.8300
 "msbisb25", 99.6894, 1.03415, 100.724
 "msbisb25", 82.0337, 18.5264, 100.560
 "msbisb25", 80.8562, 17.6338, 98.4900
 "msbisb25", 85.7570, 13.9368, 99.6939
 "mabisb06", 99.4645, .463434, 99.9279
 "mabisb06", 99.2842, .682973, 99.9671
 "mabisb06", 99.4594, .567973, 100.027
 "mabisb25", 26.0476, 73.0580, 99.1056
 "msbisb25", 98.9069, .576286, 99.4832
 "msbisb25", 98.9150, .624202, 99.5392
 "msbisb25", 99.4395, .368290, 99.8078
 "msbisb25", 94.9391, 4.22091, 99.1600
 "mabisb75", 98.6824, .741002, 99.4234
 "mabisb75", 100.201, .230280, 100.431
```

```
 "mabisb75", 99.5045, .514928, 100.019
 "mabisb75", 99.3453, .749748, 100.095
 "mabisb75", 98.2343, .853120, 99.0874
```

Save Custom Analysis Format #2

This menu will automatically create a custom file format. This format is a tab delimited ASCII file and contains extensive information including elemental, oxide, atomic weight percents, raw, background and net counts, k-ratios and one sigma calculations for several data types, stage positions, count times, and ZAF correction factors.

A separate output file is created for each unknown based on each position sample. In addition, an option is available to have each output file automatically imported into an excel spreadsheet. This file format was specified by Haldor Topsoe Ltd. and cannot be modified for other applications.

A short explanation of the various column labels used for this custom output format is given here, but note that in reality, the symbol (El) will be replaced by the actual element symbol:

LINE	Line number
El AT%	Atomic weight percent
El WT%	Elemental weight percent
El (O2)	Oxide weight percent
TOTAL	Total weight percent
El OX/CA	Oxide to cation ratio
El ONTIM	On-peak counting time
El HITIM	High off-peak counting time
El LOTIM	Low off-peak counting time
El DETIM	Deadtime correction factor
X-POS	X stage position
Y-POS	Y stage position
Z-POS	Z stage position
RELDIST	Relative distance (in microns)
BEAMCUR	Beam current
El ONCNT	On-peak count intensity (cps/nominal beam)
El OFCNT	Off-peak count intensity (cps/nominal beam)
El NECNT	Net (off-peak corrected) count intensity (cps/nominal beam)
El +-CNT	One sigma standard deviation count intensity (cps/nominal beam)
El KRAT	Raw k-ratio
El +-RAT	One sigma standard deviation raw k-ratio
El Z-COR	Atomic number correction factor
El A-COR	Absorption correction factor
El F-COR	Fluorescence correction factor
El ZAF	Z*A*F correction factor

Save Custom Analysis Format #3

This menu will automatically create a custom file format. This format is a tab delimited ASCII file and contains extensive information including elemental, oxide, atomic weight percents, detection limits, analytical error (percent error), raw k-ratios, raw and corrected x-ray intensities, background and net counts, count times, deadtimes, beam current, stage positions, relative distance (in microns) and standard counts.

This file format was specified by John Hanchar and cannot be modified for other applications. A short explanation of the various column labels used for this custom output format is given here, but note that in reality, the symbol (El) will be replaced by the actual element symbol:

LINE	Line number
El AT%	Atomic weight percent
El WT%	Elemental weight percent
El (O2)	Oxide weight percent
TOTAL	Total weight percent
El CDL99	Detection limit in elemental weight percent at 99% confidence
El %ERR	Analytical error in percent
El K-RAW	Elemental raw k-ratio (unk counts/standard counts)
El ONCNT	On-peak count intensity (cps/nominal beam)
El OFCNT	Off-peak count intensity (cps/nominal beam)

El NECNT	Net (off-peak corrected) count intensity (cps/nominal beam)
El ONTIM	On-peak counting time
El HITIM	High off-peak counting time
El LOTIM	Low off-peak counting time
El DETIM	Deadtime correction factor (in secs)
BEAMCUR	Beam current (in nA)
X-POS	X stage position
Y-POS	Y stage position
Z-POS	Z stage position
RELDIST	Relative distance (in microns)
El STDCTS	Standard counts (cps/nominal beam)

Save Custom Analysis Format #4

This menu will create an output file of standard or unknown average weight, oxide and atomic percents along with average detection and analytical sensitivity. The user can select averages only, standard deviations only, or averages and standard deviations (x.xxx +/- x.xxx) output.

Save CalcZAF Format

This menu will output x-ray intensity raw k-ratio data for all unknown samples in the current run to a CalcZAF compatible format. Each line in each sample will be output to the file.

Save StrataGem Format

This menu will open a dialog that allows the user to select samples of different voltage conditions for use in thin film calculations in the StrataGem application. The format includes the raw k-ratios for all samples and the standard compositions assigned in the last selected sample. The user has the choice of exporting all data in a sample or just the average of each sample.

Once the file is exported, from StrataGem select File | Import | StrataGem Import and the program will prompt the user whether to import or skip each k-ratio. The program will average all imported values for each element that have the same voltage.

Open Link To Excel

This menu will create an OLE (Object Linking and Embedding) link to an Excel application (if it is available). This link is available to both the StartWin and ProbeWin applications for sending both intensity and quantitative data to an Excel spreadsheet. This feature requires Excel 8 (Office 97) or higher.

From StartWin, each time intensity data is acquired, it will be automatically sent to an Excel spreadsheet along with the column labels. From ProbeWin, the analysis line number and elemental weight percents and totals are sent to an Excel spreadsheet after an analysis is performed and the user clicks the ">>Excel" button in the Analyze! window. In the ProbeWin application the column labels will be sent if they differ from the previous sample.

Close Link To Excel

This menu will close an open OLE (Object Linking and Embedding) link to an Excel application (if it is available).

Help

About Probe for Windows

This menu displays the copyright notice, acknowledgments and contact information for users of Probe for Windows NT.

Help On Probe for Windows

This menu opens this help file for hypertext help file viewing at the main table of contents. Note that both a searchable index and glossary are available for browsing by the user at anytime during a run. Each help topic may be printed. To obtain a complete printout of the help file, ask for an updated copy of the PROBEWIN.PDF file, which is an Adobe Acrobat portable document file that may be viewed and/or printed using the free Acrobat Reader.

In addition, context sensitive help (that is, help that is applicable to the current program context) is available for each window in all applications by simply pressing the F1 key.

Appendix A

Importing Databases from Probe for Windows (16 bit)

The following instructions can be used to import certain database information from the previous version of Probe for Windows (16 bit) to the current Probe for Windows (32 bit) file formats.

The following data may be imported using from the 16 bit version of Probe for Windows and using the instructions described below:

Standard Composition Data

Create a standard composition export file (STANDARD.DAT) in Probe for Windows (16 bit) using the File | Export menu. Copy the file to the new Probe for Windows application directory (usually Program Files\Probe for Windows\). Run the new Probe for Windows (32 bit) Standard application and click Cancel when asked for a database file to open. Then create a new standard database from the File | New menu. Finally, use the File | Import menu to import the previously created standard composition export file.

Element Setup Data

Create a element setup export file (SETUP.DAT) in Probe for Windows (16 bit) using the Export button in the Element setup dialog. Copy the file to the new Probe for Windows application directory (usually Program Files\Probe for Windows\). Run the new Probe for Windows (32 bit) ProbeWin application and open the Element Setup Dialog. Using the Import button, import the previously created element setup export file.

Note that the older 16 bit version of the element setup database did not include fields for wavescan and peaking information. Therefore, those element records that have been imported from the 16 bit version will contain zeros and blanks for those fields, however the program will load the current defaults from the run when those element setups are loaded.

Position Data

Position data is normally imported from ASCII .POS files which should already exist from the 16 bit Probe for Windows files. Simply copy all .POS files from the C:\PROBEWIN directory to new Probe for Windows application directory (usually Program Files\Probe for Windows\) and import them as usual from the Automate! window.

Probe Data

Probe for Windows (16 bit) data is can simply be opened by the 32 bit version of Probe for Windows, no conversion is necessary.

Appendix B

Problems with Light Element Analysis

The analysis of light elements (fluorine - beryllium) present special problems for the microprobe analyst. Some of these problems are technical in substance, relating to the measurement procedure, while others are inherent and are due to physical and chemical effects. The following is just an overview of these concerns. For a more complete treatment the reader is referred to (Bastin and Heijligers, 1986) and (Goldstein, et. al., 1981).

Physical and Chemical Effects

1. Low fluorescent yields from these k-lines compounded by a relatively poor reflectivity of analyzing crystals and low sensitivity of detector systems can result in extremely low count rates. Increasing the beam current can produce unacceptably high deadtime corrections for any metal lines present which may also need to be measured. The use of layered dispersive element (LDE) reflectors can improve things somewhat.

2. Errors in the estimation or accuracy of the take off angle or operating voltage of the microprobe. While slight variations in these parameters normally have a little effect on a typical analysis, due to the very high absorption corrections of low energy x-rays, the effect on the ZAF correction can be much larger for the light elements and can sometimes result is large systematic errors.

3. Errors in the mass absorption coefficients (or MACs) for the x-ray absorption cross sections for these elements are considerable. See Appendix C for a table of MACs for the light elements from a variety of sources. There is considerable disagreement among investigators. Probe for Windows allows the user to enter MACs from any source for re-calculation purposes.

4. Chemical bonding effects can results in large peak shift and/or shape changes to the analytical x-ray lines. In fact the degree of shift and/or shape changes in light element K lines and transition metal L lines can sometimes be related to the degree of oxidation in the compound. But normally these effects are a nuisance to the analyst. In a few instances there has even been documented cases of particular lines being strongly enhanced (Ni Lα in NiAl) or reduced (B Kα in nickel borides) (Pouchou and Pichoir, 1988).

5. Volatilization of certain light elements such as sodium and fluorine is often the case in glasses and other amorphous samples. See the use of the Volatile element correction for a powerful software correction to remove this effect.

Problems with the Measurement Techniques

1. Higher order interferences from metal lines are everywhere and must be avoided when possible or corrected for when not. The elements Ti, Cr, Mn, Zr and Nb are well known in this respect. Although it would seem appropriate to use pulse-height analysis (PHA) to reduce these interferences it is often found that narrow PHA settings can introduce more

problems than they solve. See (Donovan, et. al., 1993). These interferences are especially severe for minor and trace level measurements.

2. Carbon contamination is a serious problem for quantitative analysis of light elements, especially of course, carbon but also the other light elements. Contamination is produced by the polymerization of hydrocarbons at the point of impact of the electron beam. Carbon compounds present may include diffusion pump oils, polishing agents, cleaning solvents, among others. The use of an air-jet has been shown to dramatically reduce carbon contamination. It is suggested that the analyst perform measurements to ascertain the actual carbon contamination rate in their microprobe for each element being analyzed.

3. Trace analysis of light elements is particularly difficult, especially oxygen and carbon. Keeping a metallic sample surface from oxidizing is not a trivial matter. The use of an ion mill inside the probe to remove the very top layer of the sample, just prior to the analysis, if available, would be ideal. Note that the increase in temperature of non-conducting samples can be significant enough to cause additional oxidation of the surface. Trace carbon analyses can be difficult without the use of an air jet to reduce the carbon contamination at the point of impact of the beam.

4. Another problem which has not been adequately addressed is x-ray production in non-conductive samples. Effects on x-ray production are seen even when a conductive coating has been applied, possibly due to internal charge buildup below the conductive layer.

5. The selection of standards is also critical due to the already mentioned peak shift and/or shape changes for light element x-rays. The use of Area Peak Factors (APFs) (Bastin and Heijligers, 1986) can provide the analyst with a practical method for treatment of these effects. Program PROBE supports the use of APF factors for analysis of light elements. See the previous sections on using APFs for correcting for peak shape and shift effects when analyzing light elements..

6. Be aware also, that carbide, boride and nitride standards are often not 100 % dense resulting in significant errors in the analysis. If single crystals can be obtained, so much the better.

Appendix C

Use of Empirical MACs

The following is a small compilation of mass absorption coefficients (MACs) from a number of investigators. Some of these MACs may be selected for use in the quantitative calculations in Probe for Windows, from the Analytical | Empirical MACs menu item. Up to MaxEmp% empirical mass absorption coefficients may be defined for each run. These MACs will be saved to the .MDB file for subsequent re-processing and can be added to, changed or deleted again.

Empirical Mass Absorption Coefficients (MACs)

Emitter	Absorber	Henke Ebisu	Ruste	Henke et al.	Bastin Heijligers	Heinrich	Frazer	Pouchou Pichoir
B ka	B	3353		3350	3500			3471
B ka	C	6456		6350	6500			6750
B ka	N	10570		11200	11200			11000
B ka	O							16500
B ka	Al	65170		64000	64000			64000
B ka	Si	74180		84000	84000			80000
B ka	Ti	15280		15300	14700			15000
B ka	V	16710		16700	17700			18000
B ka	Cr	20670		20700	20200			20700
B ka	Fe	25780		27600	27300			27800
B ka	Co	28340		30900	33400			32000
B ka	Ni	33090		35700	42000			37000
B ka	Zr	38410[1]		8270	4000			4400
B ka	Nb	4417		6560	4600	3822		4500
B ka	Mo	4717		5610	4550	4208		4600
B ka	La	3826		3730	2500	11654		2500
B ka	Ta	20820		20800	22500			23000
B ka	W	19660		19700	21400			21000
B ka	U	2247		9020	8200			7400
C ka	B	37020	37020	37000	41000			39000
C ka	C	2373	2373	2350	2373	2147		2170
C ka	Si	36980	36980	36800	37000			35000
C ka	Ti	8094	8094	8090	9400	8290		8097
C ka	V	8840	9236	8840	10100			8850
C ka	Cr	10590	10482	10600	10950			10700
C ka	Fe	13300	13300	13900	13500	14103		13150
C ka	Zr	31130	31130	21600	24000	31304		25000
C ka	Nb	33990[1]	24203	19400	23200	29760		24000
C ka	Mo	32420[1]	15500	16400	19200			20500
C ka	Hf							18000
C ka	Ta	18390	20000	18400	16000			17000
C ka	W	18750	21580	18800	17000			18000
N ka	B							15800
N ka	N							1640
N ka	Al							13800
N ka	Si							17000
N ka	Ti							4270
N ka	V							4950
N ka	Cr							5650
N ka	Fe							7190
N ka	Zr							24000
N ka	Nb							25000
N ka	Mo	23220		20200		26458		25800
N ka	Hf							14000

N ka	Ta							15500
O ka	Li	1602		1600	1600			
O ka	B	7416		7420	8550			
O ka	O	1200		1200	1200			
O ka	Mg	5174		5170	5170			
O ka	Al	6715		6720	6720			
O ka	Si	8790		8790	8790			
O ka	Ti	22140		22100	19900			
O ka	Cr	3143		3140	2900			
O ka	Mn	3468		3470	3470			
O ka	Fe	4001		4000	4000			
O ka	Co	4407		4410	4500			
O ka	Ni	5245		5120	5120			
O ka	Cu	5920		5920	5920			
O ka	Zn	6548		6550	6350			
O ka	Ga	7086		7090	7090			
O ka	Y	15140		15100	15100			
O ka	Zr	16140		14800	16200			
O ka	Nb	17850		15300	17100			
O ka	Mo	18660		16700	18000			
O ka	Ru	19680		19700	19700			
O ka	Sn	23090		23100	15050			
O ka	Ba	4560		4560	4560			
O ka	La	4690		4690	3600			
O ka	Ta	10560		10600	10600			
O ka	W	10990		11000	11300			
O ka	Pb	12470		12500	11000			
O ka	Bi	12690		12700	12100			
Al ka	Al			403		397	404	393
Al ka	Cu			4550		4764	5410	4588
Si Ka	Si			350		347	332	356
Si ka	Ta			3760		1698		1500
S ka	Au					3380	3203	2200
S ka	Hg					1499	932	850
Sc la	Sc							4750
Ti la	Ti							4550
V la	V							4370
Cr la	Cr							3850
Mn la	Mn							3340
Fe la	Fe							3350
Co la	Co							3260
Ni la	Ni							3560
Cu la	Al			1450		1421	1494	1464
Cu la	Ni			11700		11776	12106	11879
Cu la	Cu							1755
Zn la	Zn			1520		1485	1581	1500

Ge la	Ge			1260		1262	1278	1240
As la	Ga							7000
Nb la	Nb			726		718	726	779
Mo la	Au			3680		3437		2200
W la	W					1215	1458	1258
Au ma	Au					1061	1101	1103

[1] extrapolated across absorption edge

Appendix D

Problems With the Analysis

Quantitative analysis is always beset with many difficulties and it is often difficult to pinpoint the cause of "bad" analyses. More often than not, the effect is the sum of more than one problem. However, there are several things that can be checked. First of all start by examining the standards. It is in situations like this that the use of secondary standards can really be helpful in determining what is going on. If the primary and secondary standards do not agree with each other, there is a problem.

First of all, are the standards really "good" standards? That is, have their compositions been determined by a reliable analytical method? In general, major element standard concentrations need to be determined using classical wet chemistry methods. Second, are the concentrations in the STANDARD.MDB database correct and without typographical errors? Third, was the right standard actually acquired? It is easy to get lost at 300-400 magnifications when using a standard mount which contains many standards.

Some other things to consider :

1. Is the operating voltage correctly specified? Is the correct x-ray line tuned for each element? Check the on-peak position offsets from the Peak/Scan Option dialog and see if they are reasonable. The program will usually type a warning if the actual and calculated peak positions are very different. Be sure that the spectrometer is not tuned on a nearby line of another element if using multi-element standards.

2. Be sure that no "bad" data points are in the standards samples used for the quantitative calibration. The best way to check for this is to analyze each standard and examine the results to look for points with obviously bad or low totals (epoxy, bad surface polish, bad carbon coat, etc.). If a bad point is found, one can delete it. Remember, one can always undelete data later on. A deleted point is simply not used in calculating the analytical calculations but is still present. A good rule of thumb is to only delete points that have low totals since generally most of the problems mentioned above will result in fewer x-ray counts. Avoid deleting points just to get better agreement between the primary and secondary standards. Points that have high totals should not be arbitrarily deleted. It may be necessary to look for other problems such as points with low totals in the primary standards.

3. Look for interferences on the analyzed elements. One easy way to do this is to use the Interferences menu item in the Standard for Windows program. One can also perform a wavelength scan and display possible interfering peak markers. If the element causing the interference is present in significant concentrations in your unknowns, and is not being analyzed for, it may be necessary to add the interfering element to the run by creating a new sample with the interfering element as an analyzed element. Be sure that the proper standards are available to use for the interference correction.

4. If none of the above suggestions seem to help, try acquiring the standards again. Probe for Windows uses an automatic standard drift corrections which can make a significant difference in situations where one or more of your analytical channels is drifting. Note that since the program will perform an automatic drift correction not only on the standards, but also the interference standards and the MAN background standards, it might be also be necessary to run additional sets of those standards or MAN standards.

5. In the case of trace or minor elements, also check to see that none of the off-peak positions are interfered with by another peak. This can cause a reduction in the on-peak counts, sometimes enough to result in a negative k-ratio. Always run at least one wavelength scan on a sample, using the same count time as your quantitative analyses, and if a peak is seen interfering with the off-peak marker, use the Low and/or High buttons in the Graph Data window to select a new off-peak position that is not interfered with.

In addition, to solve problems with trace analysis, it is always useful to run at least one "blank" that is similar to your unknown sample, at least in terms of average atomic number, if not actual chemistry. For example, when analyzing trace elements in quartz, try to run an unknown analysis on a "pure" synthetic quartz sample, one that is known to contain known concentrations of the trace elements. The point is to try to determine how closely one can measure "zero" concentrations.

Glossary of Terms

ANGSTROM

Unit of measure for x-ray wavelengths. Wavescan intensities and KLM markers may be displayed in spectrometer or angstrom units.

The conversion of spectrometer to angstrom units is by default based on the calculated spectrometer offset from the theoretical peak position compared to the actual or measured peak position using the following expression:

$$\Delta P = P_T - P_A$$

where: P_T = the theoretical spectrometer peak position

P_A = the actual or measured spectrometer peak position

This assumes that the spectrometer accuracy is a constant offset regardless of the spectrometer position. Usually this is sufficient for most purposes.

For extremely high accuracy work, use of the multiple peak calibration option in the StartWin application will produce a calibration table that will be used by Probe for Windows in calculating a variable spectrometer offset based on the spectrometer position for each spectrometer and crystal combination and for each K, L or M family. See the PROBEWIN.INI keyword "UseMultiplePeakCalibrationOffset" for more information on the use of this variable spectrometer offset feature.

ALPHA-FACTOR

Alpha-factors are based on the original Bence-Albee correction (Albee and Ray, 1972) and modified to utilize additional fit coefficients (linear and polynomial) for improved quantitative results after Rivers (pers. comm., 1990) and Armstrong (Armstrong, 1991). These factors are derived by fitting concentrations vs. alpha-factors, from calculated or measured k-ratios in a system of binary standards covering a range of composition. See also beta-factors. The expression for the calculation of a single alpha-factor is shown below:

$$\frac{\left(\frac{C}{K}\right) - C}{(1 - C)} = \alpha$$

where : C is the elemental weight fraction of the element

K	is the calculated or measured k-ratio at that concentration

The calculation of the matrix correction for an unknown sample is shown below:

$$C_A^u = \frac{C_A^s}{Beta_{\lambda_A}^s} \frac{I_A^u(\lambda_A)}{I_A^s(\lambda_A)} Beta_{\lambda_A}^u$$

where :	$Beta_{\lambda_A}^u$	is the unknown beta-factor
	$Beta_{\lambda_A}^s$	is the standard beta-factor

ATOMIC PERCENT

The atomic percent is calculated based of the fraction of the atomic weight of the element. Also called "elemental mole" percent. Since the calculation normalizes the data to a 100 % total, it is recommended that the calculation be only performed on samples which total close to 100 %. The calculation is as follows :

$$A.P. = \frac{100}{\sum_{i=1}^{n}\left(\frac{W}{A}\right)} \bullet \frac{W_i}{A_i}$$

where :	W	is the elemental weight percent of the element
	A	is the atomic weight of the element

AVERAGE

Also known as the mean or arithmetic mean. The average is calculated by simply dividing the sum by the number of values. The calculation is shown below.

$$A = \frac{{}_{i=1}^{n}X_i}{n}$$

where :	X	are the values to be averaged
	n	is the number of values to be averaged

AVERAGE ATOMIC NUMBER

Also known as Z-bar. The sum of the elemental weight or atomic fractions of the element atomic numbers of a sample.

The sum of the weight fractions of a sample is applicable for modeling the background continuum and is used in the MAN background correction iterated polynomial calculation by Probe for Windows. The sum of the atomic fractions of a sample is applicable to stopping power calculations and is used in Monte Carlo calculations of electron trajectories. The calculation is quite simply :

$$\bar{Z} = \sum_{i=1}^{n}\left(\frac{W}{\sum_{i=1}^{n} W}\right) \bullet Z$$

where :	Z	is the atomic number of the element
	W	is the elemental weight or atomic percent of the element

BETA-FACTOR

Beta-factors are the normalized sum of the weight fraction weighted alpha-factors for a given compound composition. These are utilized in the quantitative calculation of unknown compositions. See alpha-factor above.

CATION

The metal component of a chemical compound. For example, in the compound SiO_2 the Si or silicon is the metal or cation and the O_2 or oxygen is the anion. In this example, the subscript for Si is 1 and the subscript for oxygen is 2.

CPS

Counts per second. X-ray count rates are always given in cps units to normalize for different count times. To get the actual counts multiply the displayed x-ray counts by the count time used.

DEADTIME

The interval between the arrival of an x-ray pulse signal and the time that the system (including detector and amplifier electronics) is ready to receive the next one is defined as the deadtime. The deadtime correction is only significant at count rates above 10K to 20K counts per second (cps). For example, if the deadtime is 1 us and the count rate is 10K cps, the count rate will be reduced by 1%.

The best way to determine the deadtime constant is to measure it by the function of count rate vs. beam current. The normal deadtime correction is given by the expression below:

$$I = \frac{i}{1.0 - i\tau}$$

Where :	i	is the raw measured counts in cps
	τ	is the deadtime constant in seconds
	I	is the deadtime corrected counts in cps

An optional high precision expression is also available for very high count rates (> 50K cps) and is shown here :

$$I = \frac{i}{1.0 - \left(i\,\tau + i^2\,\frac{\tau^2}{2}\right)}$$

Note that Probe for Windows supports the use of a hardware "enforced" deadtime for rigorous correction of the deadtime effect if available on the microprobe (e.g.

Cameca SX-50/51). In this situation, an arbitrary deadtime constant, that is larger than the largest deadtime measured on the spectrometer, is utilized by the amplifier electronics to gate off any pulses that are received by the amplifier after the initial pulse, within the value specified. This has the net result of masking the "intrinsic" deadtime of the system, which has been noted by several observers to be quite dependent upon the energy of the pulses received from the detector. Hence, the "enforced" deadtime value is used in software to correct the intensities without regard to any dependence upon pulse energy.

To facilitate the measurement of the intrinsic deadtime of your PHA amplifiers, an easy to use but rigorous Excel spreadsheet from Paul Carpenter of Cal Tech is available from Advanced MicroBeam.

FIDUCIAL

Refers to the physical marking of a sample mount with a number of reference marks, so that all data can be referenced directly to those marks on the sample surface. This is used to provide a method of re-locating specific positions even after the sample has been removed from the sample holder. This method can also be used to relocate coordinates from an entirely different coordinate system such as a digitizing microscope stage. In the Probe for Windows software package all samples using this feature must have 3 fiducial marks. This is because the program assumes that the positions are digitized in three dimensions (x, y and x).

GRID

(Grid File) A file format used by SURFER (Golden Software) to store single precision floating point data with 3 coordinates of information (X, Y and Z). The file is a binary file and may be read from VB, FORTRAN, C or Pascal. A grid file always has the extension ".GRD". The format is as follows :

cdum	a 4 byte string containing the characters 'DSBB' (not null terminated)
ixgrid, iygrid	two integer*2 values containing the grid density
xmin, xmax	two real*8 values containing the X data minimum and maximum
ymin, ymax	two real*8 values containing the Y data minimum and maximum
zmin, zmax	two real*8 values containing the Z data minimum and maximum
data	ixgrid * iygrid real*4 values containing the Z data values

The grid file format is also compatible with Micro-Image for Windows, a full featured image acquisition and analysis application available from Advanced MicroBeam.

INTERFERENCE

An analytical situation when a peak from another element overlaps a primary analytical line used in the analysis. Ignoring this interference will often result in an incorrect analysis. This is sometimes seen as a high total when the interference is large. However when the interfered element is only a minor or trace quantity, even a small interference can result in a large error on the interfered element without affecting the overall total significantly. These primary line interferences can be completely and quantitatively corrected for using Probe. Be aware however, that interferences on your off-peak positions need to be checked for, before acquiring any off-peak sample data, especially for trace and minor element analyses.

INTERFERENCE STANDARD

A standard used for the quantitative interference correction in Probe for Windows. A standard used for the interference correction can also be used for the analytical calibration and need only be analyzed once, although additional interference standardizations can be used in the automatic drift corrections if they are available. To use the quantitative interference corrections in Probe for Windows you must analyze for both the interfered element (obviously) ***and*** the interfering element. To use a standard as an interference standard, it must contain a known concentration of the interfering element, ***and*** it must not contain the interfered element, nor can it contain any other elements that interfere with the interfered element. You can add additional standards to a Probe for Windows run using the Standard | Add Standards To Run menu.

ITERATION

A technique which is used to calculate a result when you have more unknowns than coefficients. Probe for Windows used an iterative loop for the ZAF and Phi-Rho-Z matrix calculation and an additional iteration loop around that to calculate other compositionally dependent parameters such as the spectral interference, MAN background correction and the APF correction.

K-FACTOR

The standard k-factor is the concentration of the element in a standard divided by the ZAF correction of the element in the standard. See also raw k-ratio, unknown k-ratio and ZAF. The expression is :

$$K_{std} = \frac{C_A^s}{[ZAF]_{\lambda_A}^s}$$

where : C_A^s is the concentration of element A in the standard

$[ZAF]_{\lambda_A}^s$ is the ZAF correction for element A in the standard

K-RATIO

See raw k-ratio, unknown k-ratio, k-factor and ZAF.

MAC

Mass Absorption Coefficient. A physical constant in units of cm^2/gm, which describes the amount of absorption of an x-ray of specific energy in a pure element matrix. Used by the ZAF and Phi-Rho-Z matrix corrections.

MAN

Mean Atomic Number. The average atomic number of a sample used in the on-peak background correction. A calibration curve type of background correction in which the background count rate is measured while the spectrometer is tuned to the element of interest on samples of varying atomic number each not containing the element of interest. A function is then fitted to a plot of counts vs. average atomic number and used in the compositional iteration to calculate a background correction. This method works quite well for major and even minor elements in low to moderate average atomic number matrices, since the background of a sample is almost entirely dependent upon atomic number. See the above section on MAN backgrounds.

MATRIX CORRECTION

An algorithmic calculation to correct for the effects of other elements present in the analyzed sample. These can include absorption, fluorescence and atomic number effects. Probe for Windows supports several types of both ZAF and Phi-Rho-Z matrix corrections. See also ZAF.

MULTI-SELECT

Multi-select means that the list box supports the selection of more than one item. To select a range of items click the first item in the list and then holding the <shift> key, select the last item to be selected. To select individual items, click while holding the <ctrl> key down.

NOMINAL

Nominal is defined in the Oxford American Dictionary as "in name only" or "a token fee". However, in the Probe for Windows software it is meant to indicate that the value referred to is not necessarily the actual value. This use of the word is also seen in the lumber trade, where a 2 by 4 is the nominal dimension, but the actual dimension is often less.

For example, in this sense, the *nominal beam current* is not the actual measured beam current but only a close approximation that is used to calibrate the beam drift correction. The beam drift correction only uses the nominal beam current to set the magnitude of the correction. If the nominal beam current is close to the actual measured beam current then the correction is close to 1.0 and the beam drift corrected counts displayed will be close in magnitude to the counts displayed on the screen scalers.

ONE SIGMA

A predicted standard deviation based on the count rate and counting time. It is calculated by dividing the square root of the count rate by the square root of the average counting time. It is equivalent to the square root of the total counts. Use the one sigma value to evaluate whether a given average count rate represents a homogeneous set of sample data. Standard deviations larger than the one sigma value indicate the possibility of actual compositional variation within the data set.

$$\sigma = \frac{\sqrt{i_{cps}}}{\sqrt{t_s}}$$

where : i_{cps} = intensity in counts per second

t_s = count time in seconds

OXIDE

Generally refers to a traditional stoichiometric formula with oxygen as opposed to any specific chemical compound with oxygen. For instance SiO_2 is the oxide formula for Si. This nomenclature is generally used only by geologists. In a Probe for Windows run, the sample compositions are reported as oxides if the Display As Oxides option is selected for that sample.

P/B

Peak to Background Ratio. A measure of the sensitivity of the analysis. To improve your detection limit, you will want to increase the peak to background ratio. This can be accomplished by using the analyzing crystal with the highest theta available (for example run K Kα on LiF rather than PET) and using lower noise flow detectors rather than sealed detectors. The P/B is calculated as :

$$\mathrm{P} / \mathrm{B} = \frac{\mathrm{I_P} + \mathrm{I_B}}{\mathrm{I_B}}$$

where : $\mathrm{I_P}$ is the background corrected peak intensity

$\mathrm{I_B}$ is the background continuum intensity

PHA

(Pulse Height Analyzer). This is the electronics used to convert the analog signal from the detector pre-amp into digitized pulses for input to the x-ray counter scalers. Used to filter low energy noise and high energy interferences from the signal. Because Probe for Windows utilizes a sophisticated interference correction, it is best to use a "loose" PHA filter to avoid energy shift effects in the analysis, rather than try to eliminate interferences with the PHA settings.

PHI-RHO-Z

A method used to correct x-ray intensities for the effects of matrix by combining the absorption and atomic number corrections into an expression describing the true x-ray distribution with depth. The expression is often written as $\phi(\rho z)$. This is an alternative method to the ZAF matrix correction which can be more easily verified using so called tracer and wedge experiments.

POLYGON

An enclosed shape consisting of a number of line segments. Probe for Windows NT supports the automated acquisition of data from within user defined polygon boundaries. The digitized polygon coordinates are automatically saved to the run file as they are acquired and are automatically output when the Surfer .BAS option is selected.

POLYNOMIAL

A mathematical expression of the form : $Y = A + BX + CX^2$ +.... The MAN background correction used in Probe for Windows NT uses an iteration of this expression to calculate for major element backgrounds without the need for off-peak measurements. The user may select a 1st order (straight line) or 2nd order (parabolic) fit to the MAN data. The parabolic fit is the default.

PRIMARY STANDARD

An analytical standard which is ***assigned*** as the calibration standard for one or more analyzed elements in a quantitative run. Elements in standards not assigned as the primary calibration, but which contain an analyzed element can be considered secondary standards.

RAW K-RATIO

A k-ratio is the ratio of unknown intensity relative to a reference intensity. Several varieties of these are defined. The raw k-ratio is defined as the ratio of the unknown counts to the standard counts for a element. The intensities are usually corrected for beam drift, dead time, background and standard drift. See also unknown k-ratio, k-factor and ZAF. The expression is :

$$K_{raw} = \frac{I_A^u(\lambda_A)}{I_A^s(\lambda_A)}$$

where : $I_A^u(\lambda_A)$ is the unknown intensity for element A at λ

$I_A^s(\lambda_A)$ is the standard intensity for element A at λ

RELATIONAL DATABASE

A data file storage and retrieval model based on a set of defined relationships between two dimensional tables consisting of fields (table items) and rows (data items). From both the programmer and end user point of view, these tables behave like spreadsheets where each row is a record and various operations such as browsing and searching can be performed without knowledge of the actual physical file structure.

SCANNING

(Spectrometer) A spectrometer that can be tuned to a range of analytical x-ray lines. Usually contains two to four analyzing crystals. Scanning spectrometers are required for off-peak background corrections. MAN background corrections can be used for either fixed or scanning spectrometers.

SECONDARY STANDARD

A standard which is not assigned as a primary standard. Not used in the standard calibration. If the secondary standard contains a known quantity of the analyzed element, the quality of the secondary analysis can be judged by observing the "%VAR" line when an analysis is performed. The "%VAR" is calculated as follows :

$$\%\,\mathrm{VAR} = \frac{W_{AVER} - W_{PUBL}}{W_{PUBL}} \bullet 100$$

where :	W_{PUBL}	is the published concentration of the element
	W_{AVER}	is the measured average concentration of the element

SPECIFIED CONCENTRATION

A concentration which is used in the matrix correction calculation to provide a more accurate matrix correction. These concentrations are used in two ways in Probe for Windows. Standards which contain elements which are not being analyzed for, will have their concentrations automatically loaded by the program for possible use in analyzing a standard.

Second, if you are analyzing an unknown which contains elements that you are not analyzing for, but whose concentrations you know or can estimate, you can add these elements as specified elements and specify their concentrations. This will result in an improved matrix correction for those elements that you are analyzing for.

SPECIFIED ELEMENT

An element included in sample matrix corrections that is constant in concentration, and therefore, not being analyzed. It is included to account for its effect on x-ray absorption and fluorescence in the sample matrix.

STANDARD DEVIATION

A calculation that essentially describes the error for a single point of a sample. The calculation is :

$$\mathrm{S.D.} = \sqrt{\frac{{}^{n}_{i=1}(X_i - \mu)^2}{n-1}}$$

where :	X_i	is the data observations
	μ	is the mean of the data
	n	is the number of observations

STANDARD ERROR

A calculation that essentially describes the error for the average of a sample. It is calculated by dividing the standard deviation by the square root of the number of data points. The calculation is :

$$S.E. = \frac{\sqrt{\dfrac{\sum_{i=1}^{n}(X_i - \mu)^2}{n-1}}}{\sqrt{n}}$$

where :	X_i	is the data observations
	μ	is the mean of the data
	n	is the number of observations

TIFF

(Tagged Image File Format) An image file format consisting of rasterized pixel color information. Probe for Windows can create an OLE script that will automatically create .GRD files that can be imported by Micro-Image for Windows for fully registered TIFF images.

TUNABLE SPECTROMETER

A spectrometer that can be tuned to a range of analytical x-ray lines. Usually contains two to four analyzing crystals. See SCANNING (spectrometer) above.

UNKNOWN K-RATIO

The unknown k-ratio can then be defined as the product of the raw k-ratio and the standard k-factor. See also raw k-ratio, k-factor and ZAF. The expression is :

$$K_{unk} = \frac{C_A^s}{[ZAF]_{\lambda_A}^s} \frac{I_A^u(\lambda_A)}{I_A^s(\lambda_A)}$$

where :	$I_A^u(\lambda_A)$	is the unknown intensity for element A at λ
	$I_A^s(\lambda_A)$	is the standard intensity for element A at λ
	C_A^s	is the concentration of the element in the standard
	$[ZAF]_{\lambda_A}^s$	is the ZAF correction for the element in the standard

WDS

(Wavelength Dispersive Spectrometry) X-ray analysis based on Bragg's law of x-ray diffraction.

$$n\lambda = 2d \sin\phi$$

where :	n	is the order of the x-ray (1st, 2nd, etc.)
	λ	is the wavelength in angstroms
	2d	is the analyzing crystal 2d spacing in angstroms

ϕ	is the angle of diffraction

ZAF

A method for the matrix correction of fluorescent x-rays. The method calculates the effects of atomic number (Z), absorption (A) and characteristic fluorescence (F) separately, hence the name ZAF. To put all of this in perspective, here is the complete ZAF correction expression for x-ray intensities solved for the concentration of an unknown concentration :

$$C_A^u = \frac{C_A^s}{[ZAF]_{\lambda_A}^s} \frac{I_A^u(\lambda_A)}{I_A^s(\lambda_A)} [ZAF]_{\lambda_A}^u$$

where : $\frac{C_A^s}{[ZAF]_{\lambda_A}^s} \frac{I_A^u(\lambda_A)}{I_A^s(\lambda_A)}$ is the unknown k-ratio and

$[ZAF]_{\lambda_A}^u$	is the ZAF correction factor of the unknown
$I_A^u(\lambda_A)$	is the unknown intensity for element A at λ
$I_A^s(\lambda_A)$	is the standard intensity for element A at λ
C_A^s	is the concentration of the element in the standard
$[\,ZAF\,]_{\lambda_A}^s$	is the ZAF correction for the element in the standard

Because the ZAF correction factor for the unknown is exactly that : unknown, the calculation requires a iterative solution. See also Phi-Rho-Z.

Z-BAR

See Average Atomic Number.

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