680 System
Operator's Guide

For AES PC-*ACCESS*, Ver. 7

Part No. 647986 Rev. A

Φ PHYSICAL ELECTRONICS

PHI Safety Notices

Physical Electronics' (**PHI'**s) products are designed and manufactured in compliance with accepted worldwide practices and standards to provide protection against electrical and mechanical hazards for the operator and the area surrounding the product. All PHI products are designed and intended for professional use only, by skilled "**operators**" for their intended purpose and according to all of the instructions, safety notices, and warnings provide by PHI.

Those instructions, notices, and warnings assume that an "operator" will not employ any tool when using PHI products. They further assume that all operators clearly understand that use of PHI products in any manner not specified by PHI may impair the protection provided by the products and expose them to hazards.

A "**technician**" is a qualified servicing individual who:

- Has received training to work with voltages above 50 V,
- Has read and understood the PHI technician's manual for the equipment,
- Observes and under-stands all safety notices on PHI equipment.

The safety symbols that PHI uses are defined on the following page.* To reduce or eliminate hazards, technicians and operators of this equipment must fully understand these symbols.

PHI's products are installed with international-style or **ANSI**†-style safety notices, according to site requirements. International notices are symbols within triangles (alerts) or circles (mandatory actions). PHI's ANSI-style safety notices contain:

- One of three signal words (in all capitals) preceded by the general danger symbol (⚠);
- One of PHI's safety symbols along with a brief description of the hazard and the risk or injury that could occur;
- Short message that observes ANSI's Hazard Alert Trilogy Rule by identifying the hazard, the possible result of ignoring the notice, and how to avoid the hazard.

The three signal words are defined as follows:

- **DANGER**—imminently hazardous situation that, if not avoided, will result in death or serious injury;
- **WARNING**—potentially hazardous situation that, if not avoided, could result in death or serious injury;
- **CAUTION**—potentially hazardous situation or unsafe practice that, if not avoided, may result in minor or moderate injury or damage to equipment.

SEMI‡ standards require identification of type 3, 4, and 5 electrical maintenance tasks in equipment manuals:

- **Type 3** electrical maintenance tasks involve energized equipment, exposed live circuits, and possible accidental contact; potential exposures are less than 30 V RMS, 42.2 V peak, 240 V-A, and 20 J.
- **Type 4** is the same but potential exposures are greater than 30 V RMS, 42.2 V peak, 240 V-A, and 20 J or radio frequency is present.
- **Type 5** tasks involve energized equipment and measurements and adjustment require physical entry into the equipment, or equipment configuration will not allow the use of clamp-on probes.

Only experienced, trained technicians should attempt to perform type 3, 4, or 5 electrical maintenance tasks.

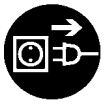
* Many of PHI's safety symbols are provided and copyrighted by Hazard Communication Systems, Inc., Milford, PA.

* American National Standards Institute, 1430 Broadway, New York, NY 10018.

‡ Semiconductor Equipment and Materials International, 805 E. Middlefield Rd., Mountain View, CA 94043-4080.

Voltages may be present that could cause death or personal injury.

A risk of death, personal injury, and/or damage to equipment exists (and a more specific label is not available).



Pulling the plug from its power source before servicing is mandatory.

A pinching point is present that could cause personal injury.

A risk of explosion or implosion may be present that could cause personal injury.

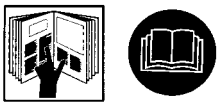
Lifting without assistance or equipment could cause personal injury.

An overhead door is present that could cause personal injury. Do not work under door without auxiliary door supports installed.

Visible or invisible radiation may be present that could cause personal injury.

Hot surfaces may be present that could cause personal injury.

Turning off the power switch before servicing is mandatory.



Refer to the manual(s) before proceeding.

Contents are under pressure.

A harmful or irritant material may be present that could cause personal injury.

Extremely low temperatures may be present that could cause personal injury.

A risk of fire may be present that could cause personal injury.

A potentially dangerous magnetic field may be present.

A spring-loaded door is present. The force of the door opening can cause injury. Use handles to open/close the door and do not stand in the door path.

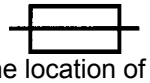
An environment with depleted oxygen may be present that could cause death or personal injury. Open at least 2 doors and wait 2 minutes before entering the enclosure.

Wearing protective gloves is mandatory.

Wearing eye protection is mandatory.

Wearing foot protection is mandatory.

This is the location of the protective grounding conductor terminal.



This is the location of the fuse.

This is the location of an earth (ground) terminal.

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Figures

Limited Warranty

Except as otherwise provided herein, the Seller warrants to Buyer that the equipment sold hereunder, whether it is new equipment or remanufactured (reconditioned) equipment, is, at the time of shipment to Buyer from Seller, free from defects in material and workmanship. As Buyer's sole exclusive remedy under this warranty Seller agrees either to repair or replace, at Seller's sole option and free of part charge to Buyer, any part or parts of such equipment which, under proper and normal conditions of use prove to be defective within 12 months from the date of receipt by the Buyer. Warranty period for equipment requiring installation by Seller will commence on completion of standard installation services. If customer delays installation beyond 45 days after delivery, the warranty period will commence to run 45 days after delivery. After installation, any realignment, readjustment, recleaning or recalibration, provided it does not relate to a proven defect in material or workmanship, shall be performed only at Seller's then current rates for service.

Exclusions and Limitations

It is recognized that some parts by their nature (expendable items) may not function for one full year; therefore, excluded from the foregoing warranty are filaments, anodes, cathodes, multipliers, retard grids, special ceramics, ionizers, along with other such parts mentioned in the applicable operating manual.

The foregoing warranty excludes certain major items or accessories specifically indicated on applicable price lists or quotations, as to which Seller passes to Buyer whatever warranty is provided to Seller by the manufacturer or the specific warranty indicated by the price list or quotation.

This warranty does not cover loss, damage, or defects resulting from transportation to the Buyer's facility, improper or inadequate maintenance by Buyer, buyer-supplied software or interfacing, unauthorized modification or misuse, operation outside of the environmental specifications for the equipment or improper site preparation and maintenance.

Product Service

All claims must be brought to the attention of Seller within 30 days of the failure to perform.

Seller at his option may require the product to be returned to the factory, transportation prepaid, for repair.

Refund of Purchase Price

In lieu of the foregoing, Seller may at any time elect, in its sole discretion, to discharge its warranty by accepting the return of such equipment and refunding any portion of the purchase price paid by Buyer.

Software and Firmware Products

The sole exclusive warranty applicable to software and firmware products provided by Seller for use with a processor will be as follows: Seller warrants that such software and firmware will conform to Seller's program manuals current at the time of shipment to Buyer when properly installed on that processor. Seller does not warrant that the operation of the processor software or firmware will be uninterrupted or error free.

No other warranty is expressed or implied. Seller expressly disclaims the implied warranties of merchantability and fitness for a particular purpose.

Section 1: Introduction

PHI provides three system manuals—this manual, the *AES PC*-ACCESS *Software Manual*, and the *680 System Technician's Manual*—plus a large array of manuals for individual components, including the *MultiPak Software Manual*. The manuals set is depicted in Figure 1-1.

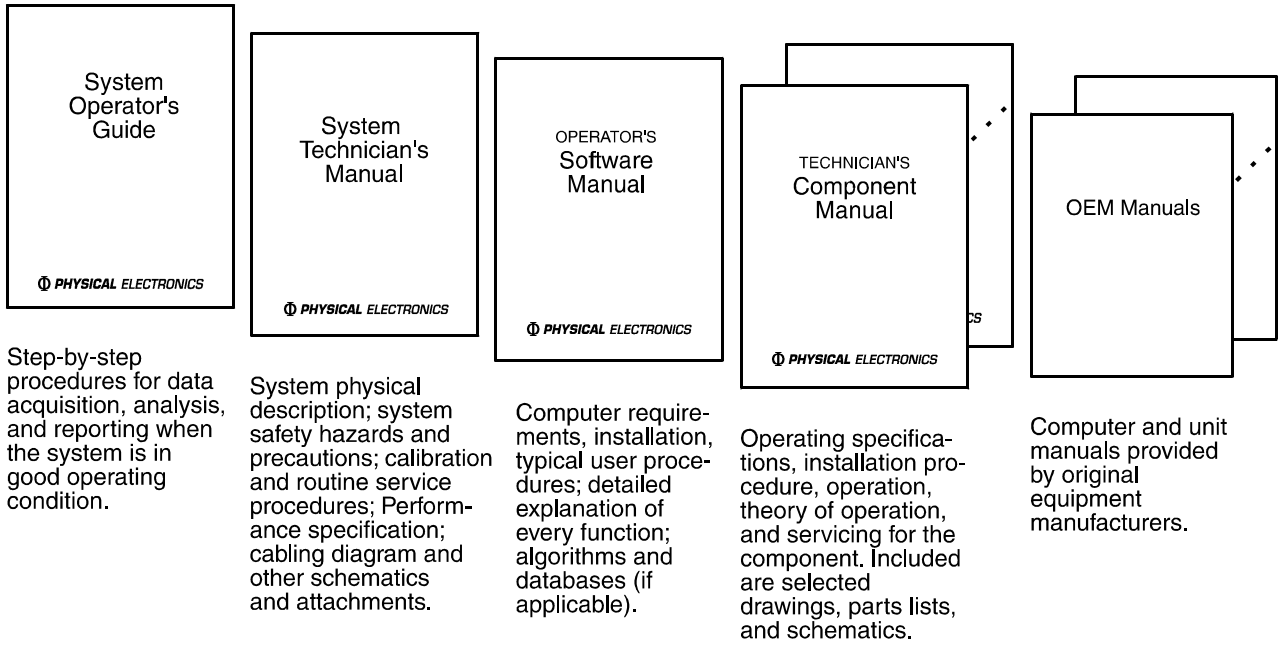

Figure 1-1. Physical Electronics' Manuals for a System.

This *680 System Operator's Guide* takes the user through operation sequences, from system startup to data acquisition and reduction, to system log off and shutdown. It is intended as a resource for getting familiar with how to operate your system under routine conditions.

Physical Electronics' (PHI's) 680 Scanning Auger Nanoprobe is operated using four interacting software applications: PC-*ACCESS,* Stage Control, Ion Gun Control, and Watcher (for vacuum control). The operator uses PC-*ACCESS* to set instrument parameters and perform data acquisition, data reduction, and publishing of results; the Stage Control software to position the stage containing the sample to be analyzed; the Ion Gun Control software to specify ion gun sputtering parameters; and the Watcher software to operate valves and pumps for

sample introduction and retrieval and differential pumping of the ion gun. This manual should be used in conjunction with the *AES PC*-ACCESS *Software Manual*. Detailed descriptions of the Stage Control software and the Ion Gun Control software are included as Appendices A and B, respectively, of this operator's guide. (Details about how Watcher works are given in the System Vacuum Control Component Manual.)

This section presents overviews of the 680 system software user interfaces, Auger Electron Spectroscopy (AES), and data acquisition using the 680 Scanning Auger Nanoprobe, and describes how to reach PHI Customer Service.

Getting Familiar with the System User Interface

The Windows-based interface of the Stage Control software is shown in Figure 1-2, the Windows-based interface of the Ion Gun Control software is shown in Figure 1-3, and the top level of PC-*ACCESS*'s operator interface is shown in Figures 1-4 and 1-5.

Sizing the Window and Selecting Commands

To resize the PC-*ACCESS* window, first click inside the PC-*ACCESS* window to make it active. Press Page Up on the keyboard to make it larger, Page Down to make it smaller, End to make it into an icon, and Home to make the window as large as it gets.

PC-*ACCESS* for AES has a default operator interface that has two "home banks" with pull-down menus of commands, called "command banks." A command is also called a "softkey," because it corresponds to one of the function keys (F1 through F10) on the keyboard. A command can be selected in one of two ways:

- Press the corresponding function key, or
- Point at and click once on the command with the left mouse button.

When a command is red, you can't select it, because it cannot be performed in the current situation.

Selecting the Interface Setting

PC-*ACCESS* is installed with one operator interface, called "PHI" or the "PHI setting" (shown in Figures 1-4 and 1-5). You can define your own sets of home banks and command banks to streamline operation to suit your tasks.

NOTE: The software reopens in the setting that was being used the last time PC-ACCESS was exited.

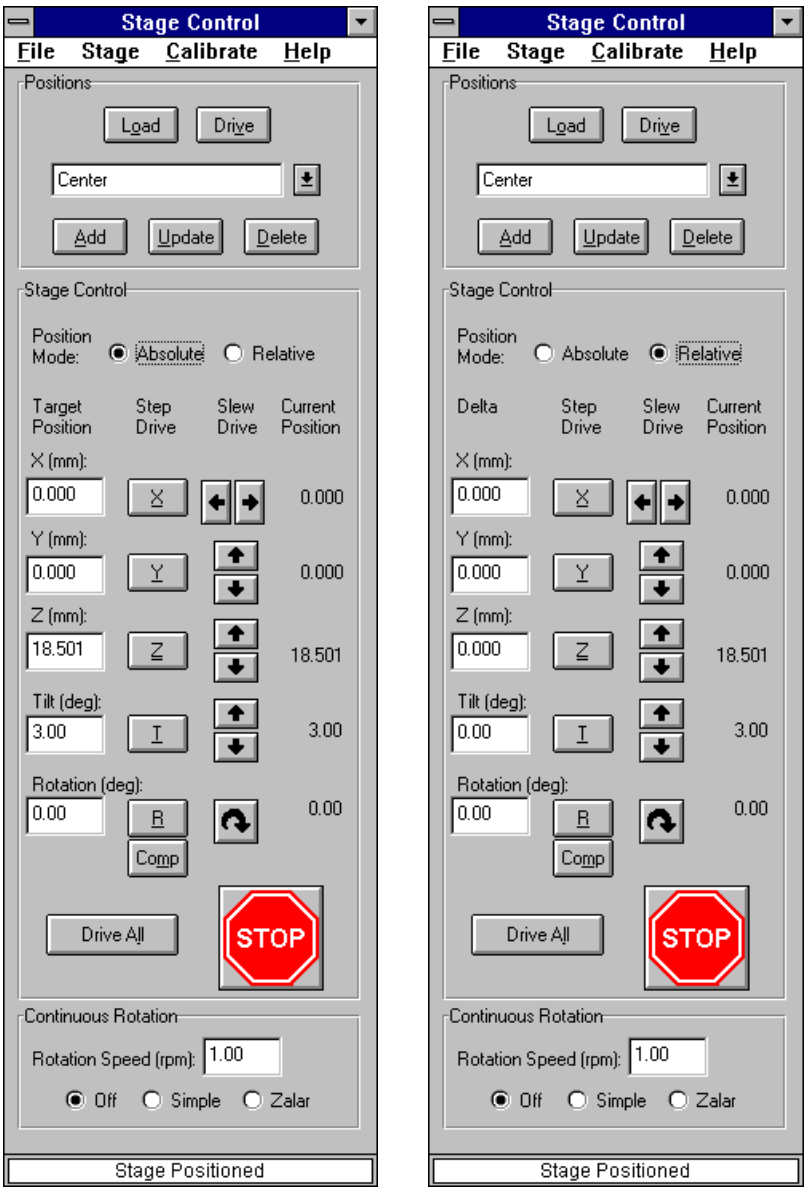

Figure 1-2. *Operator Interface of the Stage Control Software.*

When user-defined settings have been added to PC-*ACCESS*, the first thing to do after launching the software is select the setting to be used for the current session.

To do this, press Command Settings (located on the System Control command bank on the first home bank of the PHI interface). The Command Processor User Settings menu is displayed, showing the list of interfaces available. "PHI" designates the PHI interface. Select PHI to continue your walk-through of the software, and press Enter. (In routine operation, you will select the setting you prefer to use and press Enter.)

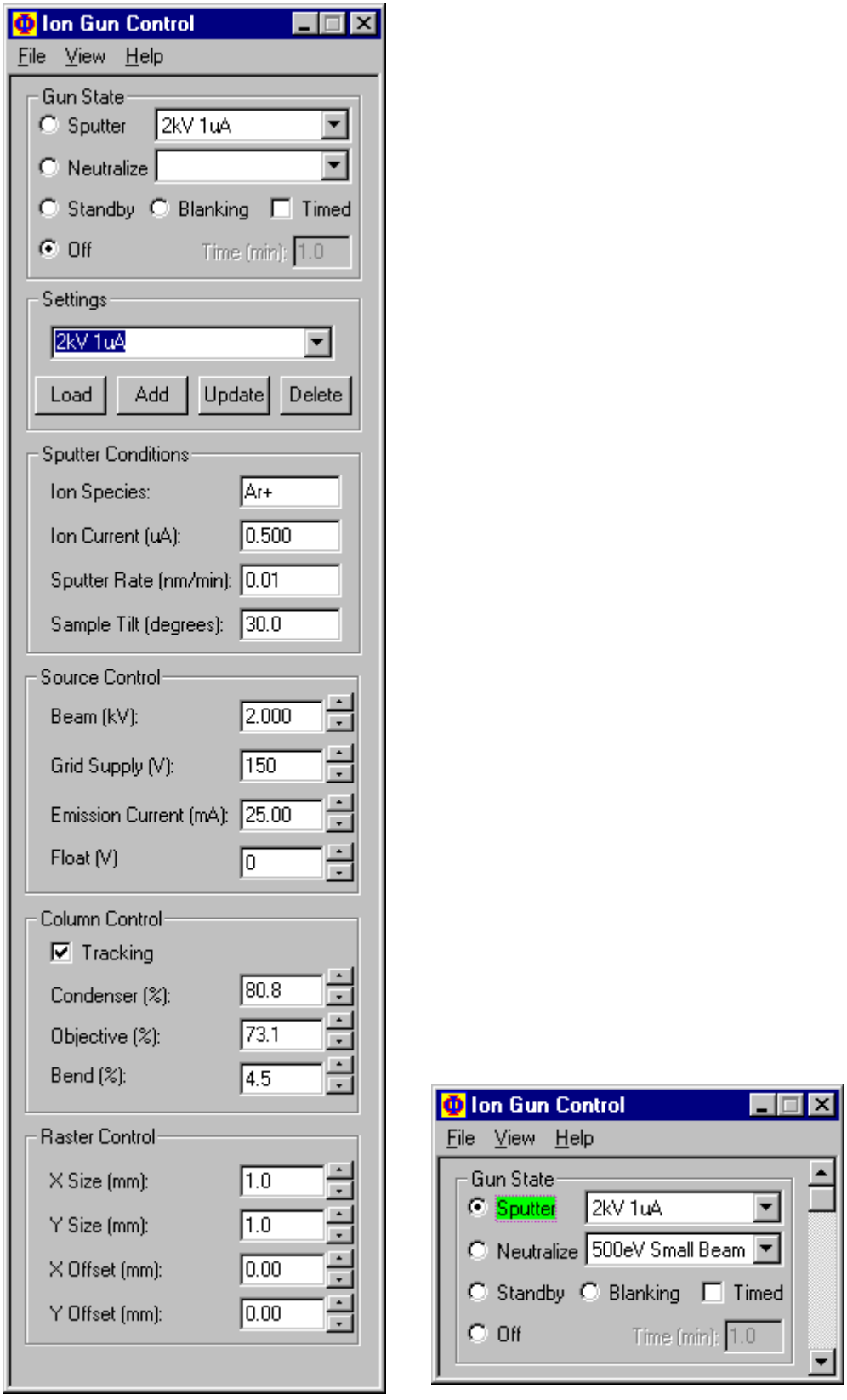

Figure 1-3. Operator Interface of the Ion Gun Control Software.

Hardware	Sample Setup	Setup Acquire	Acquire	Load Display	List Parms	Auto mation	System Control	FE/MCP MCD	NEXT BANK
SEM f1	Sample Setup f1	Setup Survey f1	Acquire Survey f1	Load f1	List Parms. f1	AutoCom f1	Element Table f1	SEM f1	
Manual Z Align f2	Area Define f2	Setup Mult f2	Acquire Mult f2	Display f2	Ion Gun Parms. f2	f2	Hardware Config f2	FE GunSemiperm f2	
Photo f3	Area Select f3	Setup Profile f3	Acquire Profile f3	List Parms. f3	f3	AES f3	SEM Calib f3	Field Emission f3	
Setup Ion Gun f4	Reg Image Setup f4	Setup Line f4	Acquire Line f4	Dir f4	f4	f4	EMS Setup f4	MCPCondSemipermf4	
Timed Sputter f5	Register Image f5	Setup Map f5	Acquire Map f5	Foreground f5	f5	f5	EMS f5	MCP CondRoutine f5	
Stop Sputter f6	Line Define f6	Setup More f6	Acquire More f6	Acq. Display f6	f6	Next Stage Pos f6	Command Settings f6	PSD/MCD Test f6	
AES acq Semipermf7	Line Select f7	Resume Acquire f7	Resume Acquire f7	Resume Acquire f7	f7	f7	f7	Setup MCD Diag f7	
Scan Rate f8	Zalar Setup f8	Change Profile f8	Foreground f8	Display Semiperm f8	List Data Setup f8	Exit Program f8	Exit Program f8	Acquire MCD Diag f8	
f9	Dir f9	Acq. Display f9	Acq. Display f9	Display Colors f9	f9	f9	f9	Acq. Display f9	

Figure 1-4. PC-ACCESS Home Bank #1 and Its Command Banks.

Spectral Massage	Profile Massage	Line Massage	Image Massage	AC Massage	Output Control	Setup Massage	Direct #1 Massage	Direct #2 Massage	NEXT BANK
f1	AC Profile f1	Norm Line f1	Setup Photo f1	AC Setup f1	Display f1	f1	Add f1	Area f1	
Diff Setup f2	Diff Setup f2	Smooth Setup f2	Photo f2	AC f2	Graph Annotate f2	Add/Subt Setup f2	Subtract f2	Differentiate f2	
AC Setup f3	Peak Redefine f3	Shift Setup f3	Image Process f3	AC Profile f3	f3	Baseline Setup f3	BaselineSubtract f3	Smooth f3	
Cursor f4	Montage Setup f4	Cursor f4	Pseudo Colors f4	AC Summary Clear f4	Print Setup f4	Integrate Setup f4	Integrate f4	Montage Display f4	
Display f5	Display f5	Display f5	Display f5	Display f5	f5	Save Data Setup f5	Save Data f5	f5	
Expand f6	Expand f6	Expand f6	Montage Setup f6	AC Summary Setupf6	f6	f6	Norm by E f6	Shift f6	
f7	f7	f7	f7	AC Summary List f7	f7	Edit Data f7	f7	f7	
Print Graphics f8	Time to Depth f8	Print Graphics f8	Print Graphics f8	Print All f8	Print Graphics f8	Hgt/FWHM Setup f8	f8	f8	
Graph Annotate f9	Graph Annotate f9	Graph Annotate f9	f9	Element Table f9	Print All f9	Normalize f9	f9	f9	

Figure 1-5. PC-ACCESS Home Bank #2 and its Command Banks.

Step through the PHI Setting

The PHI setting is used at Physical Electronics' (PHI's) Analytical Laboratories for customer demonstrations and contract analysis, and the PHI Customer Service Engineers use it when operating your system or assisting you with operation. You can't change the PHI setting (except to *add* commands in available locations).

The first of the two PHI home banks has the commands having to do primarily with hardware setup and control, data acquisition, and data and parameter display. For example, the Hardware command bank controls the ion gun, Load Display has the commands for finding, loading, and displaying data files, and Setup Acquire and Acquire have the commands needed to take data.

The second home bank, which is displayed by pressing Next Bank in the first home bank, is mainly for data reduction. Spectral Massage is where you massage the survey scans. Profile Massage is, as it suggests, for massaging depth profiles, Line Massage, for line scans, etc. Under Image Massage is Photo, Image Process, Pseudo Colors, Montage Setup, etc. AC Massage is for atomic concentration information, and so forth.

Finally, there are Setup Massage, Direct Massage #1, and Direct Massage #2. The Setup Massage command bank contains some massage functions that require setup, and the Direct Massage command banks have massage routines that you can just click on to perform tasks automatically. These three banks contain massage functions not included in the previous banks.

What's in the Entire Command Set?

Some commands are available in PC-*ACCESS* but not listed in any of the command banks in the PHI setting. To display the entire set of AES commands, press Command Settings in the System Control command bank (on the first home bank) to display the Command Processor User Settings menu. A list of settings is displayed, one of which is called "PHI" to designate the PHI setting.

Select PHI and press Edit Cmd Bank to display the first of the three pages of available commands. Pressing More Commands displays the successive pages, and pressing Display Home returns the display to the home menu, where the Add Bank, Delete Bank, and Edit Cmd Bank softkeys are displayed.

The commands are listed alphabetically. User-defined commands can be created, named, and added to this list (using the AutoCom feature). When the user creates commands, their names should be in all lowercase letters so they will be listed alphabetically beginning after the last PHI command (Zalar Setup).

In the Command Processor User Settings menu, commands are displayed in red to indicate that that command already resides on at least one command bank in the current interface (setting). Commands in black are not listed in any command bank.

Create Your Own Interface

You can't change the names of the PC-*ACCESS* commands, but you can create and save your own arrangement of commands in home banks and command banks. Any command may be placed in any command bank and in as many command banks as desired. The only constraints are the following:

- The command bank name must fit into the space for its display.
- A maximum of ten commands may be entered into one command bank.
- Only nine command banks (plus a Home Bank or Next Bank softkey) can be entered for each home bank.

It's quite easy to define a personalized setting. A simple modification of the PHI setting is described in the following steps, just to show what the steps are.

1. Press Command Settings in the System Control command bank (on the first home bank). Click on PHI. (Usually, you will start by pressing the Add Bank key, but this example will only add a single command to the PHI setting.)
2. Click on Next Bank, then on Direct Massage #1. Then, press Edit Cmd Bank to display that command bank and the first of the three pages of available commands.
3. Click on More Commands until you see the command Norm by E. Now, click on Norm by E, click on the open line (labeled "f5") of the Direct Massage #2 command bank displayed on the left, and press Select Command.
4. Press Display Home. Press Add Setting, type in "junk," and press Enter. If you press Exit at this point, "junk" will be added to the Command Processor User Settings menu as a setting available for selection as the operator interface. Press Abort instead to not save this new setting.

The command banks Direct Massage #1 and #2 in the PHI setting were devised for the very purpose of making available rarely-but-sometimes-used commands like Norm by E. You may want to add similar command banks to your own setting so you can add certain commands as they become needed.

On occasion, it might be useful to set up a temporary setting. You would create it as described above, but you would press Exit instead of Add Setting. PC-*ACCESS* would save the user-defined setting, but only until PC-*ACCESS* was exited (or until another setting was created). The next time PC-*ACCESS* was started, this setting would not be available, because it was never saved to a setting name.

Brief Introduction to AES

NOTE: Refer to the Handbook of Auger Electron Spectroscopy* *for a detailed review of AES theory, Auger analysis using PHI instrumentation, and reference spectra for 81 elements to assist with identification, quantification, and interpretation of AES data.*

AES is a fast, nondestructive analytical technique used to determine the elemental composition of the top few atomic layers of a surface or exposed interface in a solid material. AES can detect all elements except hydrogen and helium, and it can provide semi-quantitative information with an average detectability limit of 0.1 to 1 atomic percent. Newer AES instruments with field emission electron sources provide rapid characterization of sample features less than 100 Å (10 nm) in size.

AES occurs under ultrahigh vacuum (UHV) conditions, typically by probing the sample with a 3 to 25 keV electron beam. Incident electrons collide with inner shell electrons in the material, leaving atoms in an ionized state. When the ionized atom relaxes to a lower state, an Auger electron can be emitted. Pierre Auger first described this electron emission process in 1925.

Auger electrons escape from the surface with a kinetic energy characteristic of the parent atom. The energy of these escaping electrons is analyzed, resulting in a spectrum of Auger peaks that acts as a fingerprint of the probed surface. Each element has a unique set of Auger peaks.

The kinetic energy of Auger electrons is typically between 40 and 2500 eV. In this energy regime, electrons have an escape depth of approximately 5 to 50 Å (Figure 1-6). This shallow escape depth gives AES its surface sensitivity, enabling analysts to obtain information from the top few atomic layers of the sample. In certain instances, chemical state information for an element can be derived from shifts in energy or changes in line shape. Although AES is normally used to analyze conductive solids, the technique can also be used to analyze inorganic oxides.

Scanning auger microscopy (SAM) is accomplished by scanning an electron beam across the surface of a sample while measuring resultant electron signals. This process generates secondary electron microscope (SEM) images, backscattered electron (BSE) images, and Auger maps. SEM images, which provide a topographic view of the sample by detecting low-energy electrons emitted from the surface, are used to locate specific areas for more detailed study. BSE images, involving higher energy electrons that have undergone scattering

* K.D. Childs et al., *Handbook of Auger Electron Spectroscopy*, Third Edition, Physical Electronics, Inc., Eden Prairie, 1995.

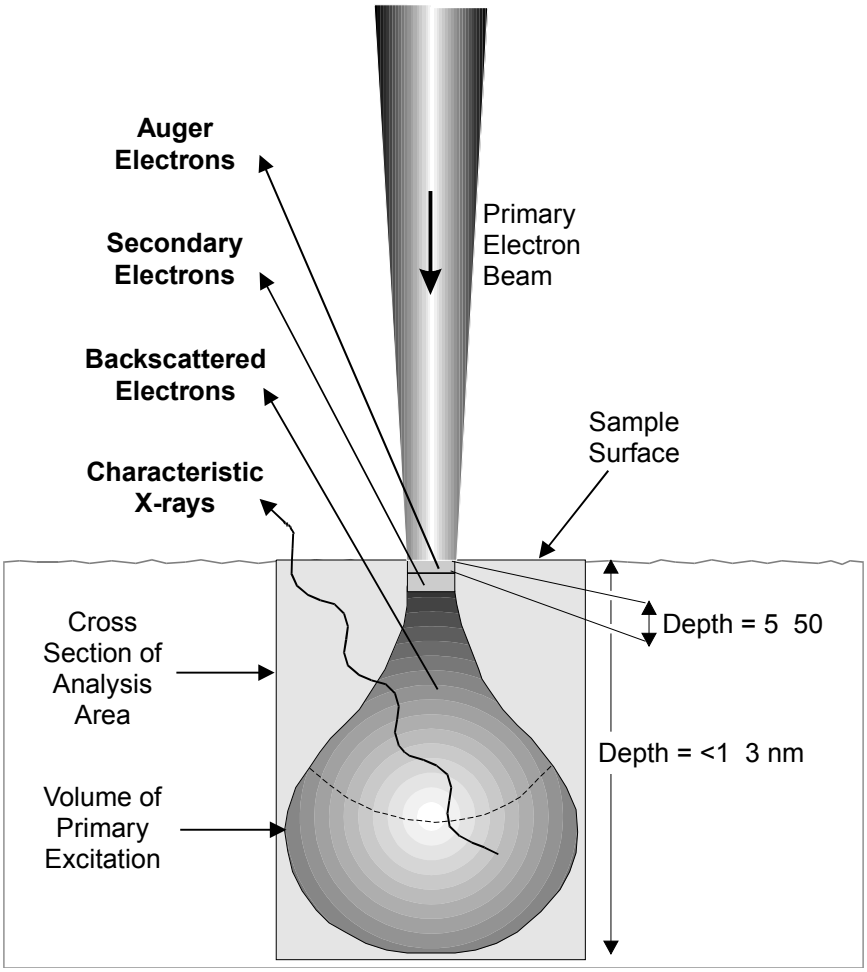

Figure 1-6. *Interaction between an Incident Electron Beam and a Solid Sample. The analysis volumes for Auger electrons, secondary electrons, backscattered electrons, and x-ray fluorescence are shown.*

processes before escaping from the sample, reveal density and crystallographic information. Auger maps, obtained by measuring the emitted Auger electron intensity while scanning the electron beam, reveal the lateral distribution of an element. Acquiring a complementary set of Auger maps provides a thorough characterization of a sample's surface.

Additional information can be obtained by sputter etching the sample surface using inert gas ions. Such sputter etching erodes the sample in a controlled manner and is typically performed with argon ions. A sputter depth profile, which plots the Auger signal as a function of sputter time, shows elemental concentrations as a function of sample depth. This technique is particularly effective for thin-film analysis and for solving materials problems that require identifying such characteristics as the thickness of a thin surface layer, the composition of a thin-film deposit, the presence of interdiffusion between thin films, or the presence of contamination at an interface between two layers.

Dr. Anton Zalar determined that depth profiling is improved when the sample is rotated during sputtering. PHI's Zalar Rotation™ achieves more uniform sputter etching, which minimizes surface roughening, reduces cone formation, and enhances interface resolution.

AES Data Acquisition Modes

The five types of AES data acquisition using PHI systems are survey, multiplex, map, line scan, and depth profile.

Survey

The survey is acquisition of one spectrum from a quick, high-sensitivity scan of a wide energy range (typically 30 to 2030 eV in 1 eV steps) to survey the elements present at a point on the sample or over an area. In *point* analysis, a stationary electron beam is positioned on a specific point. In *area* analysis, the beam is rapidly scanned, or rastered, over an area of the surface.

Multiplex

The multiplex is acquisition of a set of spectra from a series of high-resolution surveys of narrow energy ranges (typically 30 eV wide in 0.5 eV steps). This type of acquisition yields great sensitivity and spectral detail in a short analysis time, because only selected energy regions expected to contain Auger peaks of interest are scanned.

In this manual, the term "multiplex" is used to characterize data (when appropriate), but using the "Acquire–Multiplex" PC-*ACCESS* function is not described, because it is useful primarily when looking for trace elements. The more commonly used multiplex acquisition routines are the window line scan and depth profile, which are described in detail. The 680 Scanning Auger Nanoprobe can acquire as many as 20 elements in one multiplex.

Maps

The map is a set of intensity-value arrays acquired over the area of the SEM to show the surface distribution of specific elements. Each array of intensity values corresponds to an element, and each value in the array corresponds to a point in the map area. The intensity value is obtained by measuring the intensity at a specific Auger peak energy (the energy of the element's principal Auger peak), then subtracting the background intensity.

Acquiring map data for every element identified in the survey will completely characterize the distribution of elements in the analysis area. These maps are then compared to the SEM image.

Line Scans

An Auger line scan is acquisition of data along a single line across a sample. A two- or three-point line scan acquires one spectrum that comprises the peak-minus-background values from the points along the line, and window line scans acquire a multiplex at each point along the line. Window line scan data can be used to extract chemical state information as well as atomic concentration data.

Depth Profiles

An Auger depth profile provides compositional data as a function of depth. To obtain a depth profile, the surface of the specimen is sputtered by inert gas ion bombardment, then Auger spectra are collected from the center of the etched area. Data are acquired (and acquisition parameters like the sputter rate can be adjusted, if desired) alternately with sputtering. Changes in Auger signal amplitudes after etching indicate changes in specimen composition with depth, yielding a depth vs. composition profile.

Typical Analysis Sequence and Corresponding Manual Sections

The following lists describes a typical analysis sequence and the section of this manual that contains those procedures.

Section 2—Start the session and create a file-naming scheme. Ending a session and shutting down are also described.

Section 3—Set up the sample in the chamber, position the area of interest at the focal point of the analyzer, set hardware parameters, take an SEM of the image on the digital storage monitor (DSM), define analysis areas, and set up for image registration.

Section 4—Take a survey scan and perform initial analyses (such as atomic concentrations) to identify what elements are present on the surface.

Section 5—Generate maps of the elements of interest.

Sections 6 and 7—Generate one or more line scans.

Section 8—Generate a depth profile.

Section 9—Set up longer, automated acquisition sequences using PC-*ACCESS*'s AutoCom feature.

Some Analysis Considerations

- Use as much current as possible for good signal-to-noise ratio while (1) still being able to resolve the features of interest (if the beam diameter is larger than the feature of interest, you will get substantial signal from the surrounding area) and (2) not causing electron beam damage to the sample surface.

  Differentiating data makes it easier to identify elements, because Auger peaks are riding on a high secondary electron background. Smoothing data will lessen the impact of noise on the spectrum, but peak resolution is lost as the smoothing function broadens. Typical values are 5, 7, or 9 points.

- Survey spectra can be very noisy when rastering over an area that (1) includes elements of drastically varying secondary electron yields and (2) has rough surfaces with sharply changing angles.

- Longer acquisition times or multiplex spectra are usually needed to identify minor elements.

- Be aware of elemental sensitivity factors when choosing beam voltages. As a general trend, as the beam voltage is increased, the sensitivity to high-energy Auger peaks increases while the sensitivity to low-energy peaks decreases.

- Be aware of the four types of beam damage that the current selected can do to your sample:

  - *Reduction*—Metal oxides and hydrocarbons can be reduced by the electron beam.
  - *Desorption*—Adsorbed material can be removed.
  - *Electron migration*—Mobile ionic material can be repelled or drawn toward the electron beam. For example, PSG glass contains phosphorus, which is attracted to the beam.
  - *Diffusion*—The beam's heat can cause an intermixing of liquids, solids, and gases.

How To Reach PHI Customer Service

If any PHI manufactured or supported controls or equipment fail or other problem-solving is called for, contact PHI Customer Service as follows:

By mail:

Physical Electronics, Inc.
PHI Customer Service, M/S G11
6509 Flying Cloud Drive
Eden Prairie, MN 55344
USA

By e-mail:

service@phi.com

By telephone or fax:

Region	Telephone	Fax
U.S.	1-800-922-4744	1-952-828-6325
Outside U.S.	1-952-828-5831	1-952-828-6325
Japan	81-46-785-6522	81-46-785-4411
Europe	49-89-96275-0	49-89-96275-50

Section 2:
Starting and Ending a Session

This section describes how to get the system started for a data acquisition session. This discussion assumes that the chamber is under high vacuum and that everything has been checked out according to the system maintenance routine. Also included in this section are how to create directories and specify file names, because you will need this information when you acquire data. Ending a session is also described.

A. Perform the daily startup routine.

B. Create a directory for your own data files.

C. Create a file-naming scheme.

D. End the session.

A. Perform the daily startup routine and start the system software.

Normally, the system is turned "down," but not "off" at the end of each day. This involves switching some of the electronics units off or down. At the beginning of your user session, you need to put these units back into operation. Launching the software applications that are necessary to run the analytical system functions is also described.

NOTE: Typically, some units may be turned off at the end of a shift, but it is not *necessary to turn anything off.*

1. Ensure that the following buttons on the Emergency Manual Off box are on:

   – MAIN POWER ON (white when on),
   – AC MAINS (green when on),
   – ELECTRONICS CONSOLE (white when on).

2. Ensure that the system computer and peripherals are on:

   – Monitor on,
   – Personal computer (PC),
   – Printer.

The computer first performs self-diagnostics, then loads the Microsoft® Windows NT® Operating System. The startup sequence allows the user 10 seconds to select an alternate setting; otherwise, the default setting is used.

3. Log on:

   When the operating system has been loaded, the prompt "Press Ctrl+Alt+Del to log on" appears:

   a. Press the Crtl, Alt, and Del keys on the keyboard simultaneously. A dialog box is displayed that lists the "Username" as "phiuser," the computer's name, and "Password."

   b. Type "phiuser" and press Enter.

4. Start the system software by double-clicking on the PC-*ACCESS* icon on the desktop or selecting Start–Programs–PHI–PC-*ACCESS*.

   The system shuts off all of the voltages and resets them according to initialization parameters that ensure your safety.* When initialization is complete, the PC-*ACCESS*, Stage Control, and the Ion Gun Control windows open and displayed, the Watcher software is open and minimized as a button in the Start taskbar, and PC-*ACCESS* is the active window.

   The first thing displayed in the status line of the PC-*ACCESS* window is a warning to be sure that the microchannel plates have been conditioned. If the channel plates have not been conditioned since the system was last pumped down, you'll need to condition the channel plates. The procedure is given in the *AES PC*-ACCESS *Software Manual* (Section 3, MCP Cond Routine). Once the microchannel plates have been conditioned and as long as the system stays under vacuum, you won't have to condition them again.

   ATTENTION: If you don't condition the microchannel plates after pumpdown, you will burn out the channel plates. Replacement will be costly and take valuable time.

   Press Enter when you're sure that the microchannel plates have been conditioned.

   NOTE: The computer screen turns off automatically after 15 minutes of inactivity. Reactivate it by either moving the mouse or pressing a key on the keyboard.

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* These parameters can be changed in the SEM menu, but the installed settings are known to be safe settings for daily system startup.

5. If the message "Stage needs initializing" is displayed in the status line of the Stage Control window, initialize the stage, as follows:

   NOTE: The stage does not have to be initialized unless the status bar in the Stage Control software says to initialize it. The stage should, however, be initialized periodically—perhaps after two weeks without an initialization—to reset the stage's position absolutely to the stage's displayed parameters.

   a. Select Stage–Initialize.

      When Initialize is selected from the Stage menu, the stage begins the initialization process by driving each axis to its limits. Then, the prompt "Rotation needs initializing" is displayed.

   b. Looking through the viewport at the stage, use the keyboard and mouse and/or joystick to align the *dot* on the rotation mechanism with the 0-degree *line* on the fixed base of the stage. This is done in one of the following ways:

      - Press the Rotation Slew Drive button, which moves the stage in a clockwise direction only.
      - Type a value in the Rotation field and press the R Step Drive button.
      - Drive the stage using the joystick (if available).

   c. When the two marks are aligned, press the OK button in the prompt dialog box.

   d. When initialization is complete, the status bar in the Stage Control software says "Stage Positioned."

B. Create a directory for your data files.

To make the software ready for an acquisition, you need to establish a directory and file-naming scheme before doing anything with the hardware.

1. Select DIR under the softkey called Load Display on the first home bank.

   (If you're not using the PHI setting, refer to Section 1, "Selecting an Interface Setting" to select "PHI" before proceeding.)

2. The selected subdirectory is displayed in white. Any data files acquired will be saved there. Press the arrow keys on the keyboard to see all parts of the directory, and look at the subdirectories and subdirectories of the subdirectories. Find the directory named "data." This directory was installed at the top level of the directory when the software was installed.

3. Create a new subdirectory of your own, as follows.

   a. To start, click on the directory "data." It turns white to indicate that it is the current directory.

      (Your system's directory may already have a subdirectory designated for different people to store their own files. If this is the case, substitute the name of that directory for "data" when you use the following procedure, but start by highlighting that directory.)

   b. Select Create Directory. You are prompted for a directory name. Type in "mine." Press Execute.

4. Highlight your personalized subdirectory by clicking on it. Any data files generated are saved in the currently selected directory.

5. The directory only displays directory names and structures. To see data files that are inside a directory, click on the directory name, then press the List Files softkey. Press Enter with an asterisk (*) in the name field to see all the files there, or type "a" (or any letter or short string of letters) in front of the asterisk to see all the files whose names begin with the letter a.

NOTE: A good subdirectory structure makes it easy for everybody to find and use their own data files and keep them separate from other files that might get moved or deleted.

C. Create a file-naming scheme.

Select Sample Setup from the Sample Setup menu (command bank) to define a file-naming scheme for your current data acquisition session, as follows:

1. First, select some letters for the prefix (up to 11), then numbers to define a starting number for files to be acquired.

   For example, PHI users have found this kind of file-naming scheme to be very useful: If the prefix is AES and the number 101, data files acquired from the first sample would be named AES101, AES102, AES103, etc. Acquired files from the next sample could be named AES201, AES202, etc. Data files from sample #3 could have names like AES301, from sample #4, AES401, and so forth.

   NOTE: File and directory names may have up to 14 characters.

2. Enter some brief but useful description in the Comment field of the Sample Setup menu (e.g., "microwave transistor").

   If you need to correct misspellings, just press Edit Comment, then press Start Edit, use the keyboard to change the comment, and press Exit Edit.

3. Set Continuous Stage Rotation to Off.

4. Exit the Sample Setup menu.

At this point, the sample can be introduced and preparation for data acquisition can begin. (Refer to Section 3.)

D. End the session.

The following procedure is necessary only if required by the site's operating procedure or network administrator.

1. In PC-*ACCESS*, select Exit Program from the System Control menu.

   Selecting Yes and exiting the PC-*ACCESS* software also closes the Stage Control, Ion Gun Control, and Watcher programs.

   *NOTE: If you find you cannot exit from a PC-*ACCESS *menu (for example, you cannot enter a valid response to a necessary prompt), abort the current process by right-clicking on the Windows Start taskbar, selecting "Task Manager," highlighting "PC-*ACCESS*," and pressing the End Task button.*

2. Select Start–Shut Down…. A dialog box is displayed. Select one of the options, then press OK.

   

   CAUTION: If the system has shut down, turn the console power on the off, then on before restarting the software system as follows:

   i. Loosen the screw that holds closed the door on the back of the card rack cabinet (usually the right-most door on the back of the electronics console).

   ii. The console power switch is green when on. Turn the switch off, then on.

   iii. Close the console door and retighten the screw that holds it closed.

Section 3:
Setting Up the System for Acquisition

This section describes sample preparation, unloading a sample, loading a new sample into the chamber, and positioning this area at the focal point of the analyzer. The topics are the following:

A. Secure the sample to a sample mount.

B. Isolate the field emitter.

C. Pump down the intro.

D. Extract the sample from the main chamber.

E. Backfill the intro and exchange samples.

F. Put a new sample on the stage.

G. Reopen the valve to the field emitter.

H. Adjust the SEM image.

I. Center and orient the area of interest on the DSM.

J. Position the analysis area at the focal point of the analyzer.

K. Determine what electron gun parameters to use.

L. Determine what current to use.

M. Adjust stigmation, steering, and wobble.

N. Define the analysis area and magnification.

O. Create image files.

P. Draw an outline on the monitor.

Q. Define analysis areas/points.

R. Register the image to compensate for drift.

A. Secure the sample to a sample mount.

Before a sample can be placed into the analysis chamber, you must secure it to a sample mount. To avoid contaminating the sample surface, do not handle the sample with bare hands. Wear gloves. (Aids for sample mounting include tweezers, a small flat-head screwdriver, scissors, and a special tool for holding the sample mount.)

Various sample mounts are available for securing samples. Among them are a 1-inch sample mount, typically used to hold a single sample, and a 2-inch mount, which can hold multiple samples. The multiple-sample mount is typically used for unattended, automated data acquisition from several sample areas over an extended period or for data acquisition from very large samples.

Each sample mount has a molybdenum mask secured by small flat-head screws. Loosen the screws and slip the sample under the mask. You can use the mask to hold the sample in place, and you can see it on the image monitor to help you navigate on the sample.

1. Consider the sample itself and determine which mount to use and how to mount the sample.

NOTE: Wrap insulating samples in aluminum foil or secure them under a mask to reduce charging. (Using PHI's optional pre-tilt sample mount for an increased sample angle also helps alleviate charging problems.)*

*NOTE: Highly reactive samples or samples easily contaminated by the atmosphere should be mounted in a glove box. Such samples can then be transferred and loaded without exposure to the atmosphere using the Model 04-110 Vacuum Transfer Assembly.**

B. Isolate the field emitter.

Protect the field emitter by turning off the beam voltage and closing the valve that connects the field emitter to the main chamber, as follows:

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* This unit is not a standard part of a 680 system. It must be purchased separately.

NOTE: Interlocks are built into the system to prevent damage to the field emitter, but this safety measure is still worth taking the time to do.

1. Turn off the switch labeled Beam Voltage on the 18-195 FE Electron Beam Power Supply Display. Once the display gets to almost 0, the beam voltage is off.

2. ***ATTENTION: When you close the isolation valve, stop as soon as you feel any resistance.*** **Do not overtighten it**.

   Once beam voltage is off, close the isolation valve by turning it clockwise until slight resistance is felt.

   Attempting to overtighten the valve to the field emitter can bend the alignment rod inside, preventing the valve seals from working properly.

C. Pump down the intro.

Before introducing a new sample or removing a sample that is already in the vacuum chamber, you have to pump down the intro, then open the valve to the chamber using the Watcher software, as follows.

1. Press the Watcher button in the Start taskbar to display the Watcher window. Watcher shows the status of the valves: red for closed, green for open.

2. In Watcher, press the TURBO PUMP ON button to start the turbo pump. Allow a few minutes for the turbo pump to come up to speed. (The Time To Go field in Watcher indicates the approximate time remaining for the current action.)

3. Press the PUMP INTRO button to open V3 (the valve between the turbo pump and the intro) so pumping can begin. Pumping begins when the turbo pump has reached 75% of its operating speed. When the intro is pumped down to the same vacuum as the vacuum chamber, Watcher displays: “Task Pump Intro Complete.”

4. Press the TRANSFER SAMPLE button, which closes V3 and opens V1 (the valve between the intro and main vacuum chamber).

D. Extract the sample from the main chamber.

The sample holder is moved from the stage to the intro using the Stage Control software, the intro rod, and Watcher, as follows:

ATTENTION: Make sure the sample holder does not catch on anything that will cause it to slip off the fork or stage! Watch the sample holder closely through the window any time it is moving inside the chamber.

1. ***ATTENTION: Do not move the intro rod until the stage has stopped moving.***

   Select Extract Sample from the drop-down menu at the top of the Stage Control window, and press Drive.

   The status bar of the Stage Control window displays "Moving Stage." When it is done moving, the prompt "Please move intro arm to transfer point" is displayed in a dialog box.

2. ***ATTENTION: Watch the intro rod and sample holder closely through the viewport any time it is moving inside the vacuum chamber.***

   Carefully slide the intro rod all the way in until the "fork" at the end of the intro rod slides all the way into the bottom groove of the sample holder.

   NOTE: The small sample holders—the pretilt, recessed, and flat—all have two grooves: the top groove and the bottom groove. You want the intro rod to be on the bottom groove. This way, the parking stage in the system will intersect with the top groove and the predefined stage position Intro Sample will be correct.

3. Press OK in the Stage Control dialog box when ready.

   The prompt "Please slide intro arm out of chamber" is displayed.

4. ***ATTENTION: Make sure the sample holder does not catch on anything that will cause it to slip off the fork or stage!***

   Carefully slide the intro rod out of the chamber, then all the way back until V1 (the valve between the vacuum chamber and the intro) closes. In Watcher, V1 becomes red, indicating that the valve has, indeed, closed.

5. When V1 has closed, press OK in the Stage Control dialog box.

E. Backfill the intro and exchange samples.

Bring the intro "up to air" (atmospheric pressure) to exchange samples, as follows.

1. Press the BACKFILL INTRO button. This closes V3 (the valve between the turbo pump and the intro) and opens V2 to let dry nitrogen into the intro.

   NOTE: If V2 remains red, check to see that the intro probe is fully withdrawn from the main chamber.

2. In Watcher, press OK when "Introduce Sample Now" is displayed (and the Intro Convectron display in Watcher shows atmospheric pressure), twist and pull the cover to open the intro.

3. Use the sample handler to take the sample and sample holder out.

4. Slide the lower groove of the new sample holder *all the way* onto the fork at the end of the intro rod.

5. Replace the cover on the intro.

F. Put a new sample on the stage.

1. In Watcher, press the PUMP INTRO button.

   The vacuum in the intro chamber pulls the seals on the cover tight. When the intro is pumped down to the level of vacuum in the main chamber, Watcher displays: "Task Pump Intro Complete."

2. In the Stage Control window, select Intro Sample and press Drive. When the stage has moved into position, the prompt "Please move intro arm to transfer point" is displayed.

3. In Watcher, press TRANSFER SAMPLE.

4. ***ATTENTION: Do not move the stage while moving the intro rod.***

   While watching through the viewport, slowly slide the intro rod *all the way* into the vacuum chamber. The sample holder will move into position relative to the stage.

5. Press OK in the Stage Control dialog box. The stage will rise and clip onto the sample holder.

   ATTENTION: If the sample holder and stage do not align precisely, minor adjustments to the parameters in the Stage Control window need to be made and the Intro Sample setting updated to store the adjusted parameter values.

6. ***ATTENTION: Be sure the sample holder is firmly seated on the stage before moving the intro rod again.***

   Slide the intro rod all the way back and out of the chamber. V1 closes automatically.

7. Press the Minimize button in the Watcher window.

G. Reopen the valve to the field emitter.

1. When the pressure is back in the low 10^{-8} range, reopen the valve to the field emitter by turning it counterclockwise. Open the valve only until you feel resistance.

2. Turn on the Beam Voltage switch on the 18-195 FE Electron Beam Power Supply.

H. Adjust the SEM image.

When the valve to the field emitter is open, a scanning electron microscope (SEM) image should appear on the digital storage monitor (DSM), showing the detection of electrons on the sample.

1. To more easily determine your location on the sample, you need to be at a very low magnification. Press Hardware on the home bank, then SEM. In the SEM menu, select Z Align. This will automatically set the electron beam voltage to 1 kV. Highlight the Magnification 1 parameter, type 30, and press Enter or click the right mouse button.

2. Press the Auto Video softkey in the SEM menu to have the SED multiplier voltage, contrast, brightness, and DC Offset automatically adjusted.

I. Center and orient the area of interest on the DSM.

1. Locate the area of interest (where you want to do the analysis) by driving the stage to the location and centering that area on the monitor.

2. Zoom into the area to assure proper location by changing the magnification in the SEM menu.

3. Press the Auto Video softkey as needed.

4. When you are done positioning the sample in the x and y directions, exit the SEM menu.

NOTE: Use the up and down arrow keys on the keyboard with the Shift key to change the magnification in large increments. Using the up and down arrow keys without the Shift key changes the magnification more slowly. (Alternatively, values can be typed into the Magnification field.)

J. Position the analysis area at the focal point of the analyzer.

Adjust the z axis of the stage so the surface of the sample is at the focal point of the analyzer, called "Z Align," as follows.

1. Press Hardware on the home bank, then Manual Z Align.

   The beam voltage is automatically set to 1 keV (1000 eV) for Z alignment. The magnification and current are also automatically adjusted at this point. Acquisition of the energy region that includes the system's "elastic peak" begins. (The elastic peak is an energy at or close to 1000 eV and is unique to each system.)

2. The peak on the spectrum being displayed during this acquisition should be maximized at the value shown in the Elastic Peak Energy field of the Z Align menu.

   Using the Stage Control software, adjust the z axis higher if the acquired peak is lower than the Elastic Peak Energy value, or lower if the acquired peak is higher, until the peak is centered on the Elastic Peak Energy value.

   ATTENTION: Perform the Manual Z Align routine for every sample. The focal point of the analyzer is not only where the best signal is acquired; other system parameters, like ion gun settings, magnifications, and energy scale, are calibrated for this position.

K. Determine what electron gun parameters to use.

Decide what electron gun parameters are needed to optimize the acquisition data. For example, a low beam voltage might be best for an insulating sample, but a higher beam voltage may be best for a metallic or conductive sample. Higher

beam voltages yield better high-energy signal as well as better beam size at any given current.

A 10 kV beam voltage is popular for several reasons: good Auger electron yield for both high and low energy peaks, good beam size, and published sensitivity factors.*

A beam voltage of 20 kV allows for a smaller beam size and is good for penetrating samples that have an insulating layer on top of a conductive layer (so that charging does not become a problem).

1. Select from the settings on Page 1 of the SEM menu or create a setting that is suitable. Many hundreds of settings can be defined and saved by PC-*ACCESS*.

   NOTE: To define a setting, start from an existing setting that already has the same beam voltage *as the one you are defining. Otherwise, a lot of time will be wasted trying to determine the correct steering voltages. A presaved beam voltage brings with it several key settings such as multiplier voltage, image contrast, DC offset, and extractor wobble steering, to name just a few.*

   Pressing Auto Video temporarily changes some settings in the SEM menu, too. The operator may press Update Setting to store the current Auto Video settings to the saved setting.

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* K.D. Childs et al., *Handbook of Auger Electron Spectroscopy*, Third Edition, Physical Electronics, Eden Prairie, 1995.

L. Determine what current to use.

Once the beam voltage is selected, change the gun lens voltage while you watch the current, as follows.

1. Press the Read Beam I softkey on the SEM menu and monitor the current on the Keithley electrometer (a separate unit near the chamber). Change the gun lens voltage by slewing the value up to increase beam current and down to decrease beam current.

2. To obtain optimum beam size at any given current, the beam needs to be centered in the objective lens aperture. Press the Next Menu softkey to go to Page 3 of the SEM menu. Highlight Extractor Wobble Steering. Using the arrow keys, slew the steering values to optimize beam current.

   With these new values, you may need to increase or decrease beam current by slewing the gun lens voltage.

   NOTE: Higher beam currents may not be achievable without proper Extractor Wobble Steering alignment.

3. Once you have the current you want, press the Stop Beam I softkey. Then, press Auto Video to automatically adjust the SED multiplier voltage, gain, contrast, and brightness to optimize the image.

4. Finally, highlight Focus and use the arrow keys to adjust the focus.

   NOTE: Holding the Shift key down while pressing the arrow key changes the focus in large increments. Pressing only the arrow keys changes the focus more slowly. Use the Electron Gun Fine Focus Adjust potentiometer for finer focusing.

M. Adjust stigmation, steering, and wobble.

To achieve the best beam shape for any given current, you need to align the electron beam down the center of the optics column.

1. Highlight the Magnification 1 parameter, type 5000, and press Enter or click the right mouse button.

2. Select a round feature on the sample.

3. Adjust stigmation using the *left and right* arrow keys until the image appears sharper. Then, readjust the Fine Focus Adjust knob on the Electron Gun Control to obtain the best focus possible. Next, use the *up and down* arrow keys to obtain the best image, and refocus.

4. Set the magnification to 2000X.

5. Set the Focus Wobble Percentage field (Page 3 of the SEM menu) to about 10. Highlight Focus Steering (Page 3 of the SEM menu) and use the arrow keys to adjust the steering until the image no longer moves in the x or y direction.

   When the image motion no longer has directional movement, the electron beam is centered on the optics.

6. Return to 5000X magnification and readjust stigmators, as described in step 3.

   NOTE: The higher the magnification used for stigmator adjustment, the better the beam shape.

N. Define the analysis area and magnification.

Select the precise area and magnification at which you will do the analysis.

1. Center the analysis area on the image monitor by changing the x and y coordinates of the stage at a relatively high magnification. Pinpoint the area further using the Image Shift parameter in the SEM menu.

2. Select the magnification that will give you the best information.

3. Press Auto Video again and do a final adjustment of the focus using the SEM menu and the Fine Focus Adjust knob.

O. Create image files.

The first step in documenting an analysis is to create image files of the area displayed on the DSM after sample setup.

As described in Section 1, "Brief Introduction to AES," SEM images provide topographic information and BSE images reveal density and crystallographic information. While SEM images are commonly used to locate specific areas for more detailed study, BSE images of the same area can also be useful. The information from each is quite different (the first primarily topographical, the other primarily elemental), so, in some case, having both can help demonstrate more fully the characteristics of the sample.

The surface sensitivity of the image depends on the beam voltage, with lower beam voltages giving data that are very surface sensitive and higher beam voltages giving data that are more affected by topography.

SEM images can be acquired with beam voltages of up to 25 kV over a range of energies up to 3200 keV, which is the limit of the detector. BSE images detecting elastic electrons at an energy between 0 and 3200 eV must be acquired with a beam voltage that matches the energy to be detected, so the beam voltage must be between 0 and 3.2 eV.

NOTE: SEM images are typically taken at a beam voltage of 10 kV, and BSE images are typically taken at a beam voltage of 3 kV.

BSE images of inelastically scattered electrons are set up to detect an energy less than the beam voltage used. Images of inelastically scattered electrons include some topographic information, so it becomes harder to get clear information from the contrast in these images, but a better beam size can be achieved.

Acquisition of both types have much in common.

Create SEM images.

1. Select SEM from the Hardware menu of the first home bank.

2. Set the Detector field to SEM.

3. Start with a magnification much lower than the magnification at which you will perform your analysis (to 300X, for example).

4. Press Auto Video to automatically adjust the contrast and brightness. If finer adjustment is needed, on Page 2 of the SEM menu, adjust the DC Offset, Brightness, and Contrast Control fields, in that order, as needed to optimize the image on the DSM.

5. Set the Video Calibrate Mode field to Image, then use the Video Calib softkey to check the brightness and contrast. When contrast and brightness have been optimized, the DSM image will be primarily gray with some black, some white, some green, and some red. Press the Stop Video Calib softkey when done.

6. If desired, save the settings using the Add Setting softkey.

7. Press the Photo Setup softkey. In the Setup Photo menu, select the information fields you want to appear on the photo.

8. Highlight Photo, File, Printer, and/or Clipboard in the Output Device parameter, and press Enter or click the right mouse button. Selected devices are displayed in white; non-selected devices are displayed in magenta. Any combination of output devices is valid.

   "Photo" advances the photo number automatically. "File" stores the displayed DSM image to a file with the file name displayed at the bottom of the computer screen when the save is complete. "Printer" sends the image to the printer output selected in the Print Setup menu (from the Output Control command bank). "Clipboard" places a copy of the image in the Microsoft Windows clipboard.

9. Press Output Image. The image will be sent to the device(s) selected. (To print to the Mitsubishi output device, also press Memory and Print on that device.)

10. Repeat this process to document the area at various desired magnifications.

Create BSE images.

Refer to steps 1 through 8 in the previous subsection, "Create SEM Images," for the general procedure, making the following changes in the SEM menu:

1. Set the Detector field to BSE.

2. In the BSE Kinetic Energy field, type the energy that matches the voltage of the incident electron beam (e.g., 3000 V), and click the right mouse button or press Enter.

3. Set the AES Multiplier Voltage field as high as it will go. The image on the DSM will become completely white.

   NOTE: In BSE imaging, elements of lower atomic number appear darker than elements of higher atomic number.

4. Manually adjust the value in the DC Offset field using the arrow keys (and the Shift key, if needed). When the best brightness has been achieved using that field, adjust the value in the Brightness field until the best brightness has been achieved. (The DC Offset field value adjusts the brightness in large increments and the Brightness field value adjusts the brightness in smaller increments.) Then, adjust the value in the Contrast Control field.

   NOTE: If Contrast Control is set to 100% and brightness still needs further adjustment, reduce the value in the Scan Rate field.

P. Draw an outline on the monitor.

Once a sample has been loaded, introduced into the system, positioned at the focal point of the analyzer, and images have been obtained, draw an outline on the DSM along a large, prominent feature with good contrast and sharp edges in both the x and y directions. This will be used later for image registration.

Use a marker that can be removed by wiping with a dry or damp cloth. A white-board marker is a good tool for this.

Q. Define analysis areas/points.

The first step in surface analysis is to find out what is on the sample. Choose analysis areas from the image that are representative of the differing compositions on the sample.

1. Select Sample Setup–Area Define. The image on the DSM will automatically go to a medium scan rate. Press Delete All to delete the areas and points defined in the previous acquisition.

2. Highlight the type of analysis—Point or Area—that will provide the best results for your sample and press Enter.

   If point analysis is chosen, the area analyzed will be the size of the unrastered electron beam. In area analysis, the beam is rapidly scanned or rastered over an area of the surface.

3. To define a point, highlight Coordinates and use the arrow keys to slew to the desired point location for analysis. Press Define Area.

   To define an area, highlight Area and press Enter. Highlight Upper Left in Coordinates, and use the arrow keys to move the top and left bars of the crosshair cursor. (Hold the Shift key down while pressing the arrow keys to move the cursor at a faster rate.) Highlight Lower Right and use the arrow keys to move the bottom and right bars of the cursor. When the box surrounds the desired area, press the Define Area softkey. This will be area 2.

4. When done defining areas, select Exit to leave the Area Define menu.

5. Create another image at this point to show the analysis areas' locations and identifying numbers.

R. Register the image to compensate for drift.

After all the preceding tasks have been completed, the sample may have drifted a bit. By registering the image during an acquisition, you can be sure exactly where the data is being taken.

The reference image size depends upon the features that you're looking at. Locate a feature that has contrast in both the x and y directions. The smaller the feature, the smaller you can make the image reference size. The smaller the reference image size (nominally, between 60 and 100), the less time it takes to do the image registration. The reference image needs to be large enough to fully encompass the registration feature and to include the area over which the feature may drift.

The area defined as the reference image will be saved when you press Save Ref Image then press Exit. Acquisition parameter settings are then used to periodically determine whether this image area has drifted left, right, up, or down and automatically make a correction in the SEM menu for that drift.

The frequency of registration, or whether it is needed, depends on the working magnification, the dimensions of the regions or feature being analyzed, the time needed for the acquisition, and the criticality of sample movement.

1. Before you enter the image registration routine, go back to the SEM menu, and use Image Shift to realign the image with the outline(s) you drew on the monitor earlier.

2. Select Sample Setup, then Reg Image Setup.

3. In the Image Registration Area Define menu, accept or change the reference image size, the search area, and where that is located. PHI usually recommends that the Coarse Search Sampling Interval parameter be set to 2 and the Fine Search Sampling Interval parameter be set to 1.

Section 4:
Take a Survey Scan and Analyze the Data

This section describes acquiring a survey scan and analyzing the results. A survey scan is a quick, high-sensitivity acquisition of Auger data over a wide energy range. A survey scan is used to identify which elements are present in the analysis area. This is typically the first step in any sample analysis, providing an overview of elements present that will guide the design of the remaining analysis session.

The steps described are the following:

A. Set up the survey scan.

B. Register the image.

C. Acquire the survey scan.

D. Differentiate the data (and smooth, if needed).

E. Identify the element peaks.

F. Label the peaks.

G. Print the annotated spectrum.

H. Get atomic concentration data.

NOTE: The following procedure assumes that the analysis area has been positioned at the focal point of the analyzer, the electron gun parameters and current have been set, and the optics have been optimized. These steps are described in detail in Section 3, subsections J through M.

A. Set up the survey scan.

Define the acquisition parameters.

1. Select Setup Acquire–Setup Survey Scan, then select New and press Enter.

2. Define the acquisition parameters. Use the parameter values shown in Figure 4-1 as a starting point. Actual parameters will vary according to system calibration, sample properties, and data needed.

3. Press the Exit softkey.

Some of the considerations that go into exact parameter values are the following:

- If the sample might contain gold, which has its major peak above 2030 eV, a wider range (2100 eV) would be required.
- With 20 kV, 10 nA, spend at least 10 minutes per area when the sample contents are not known. For example, if one cycle for one area will take 0.5 minutes, specify 20 cycles, which will take 10.5 minutes per area.

  NOTE: One cycle is one spectral sweep for each area defined.

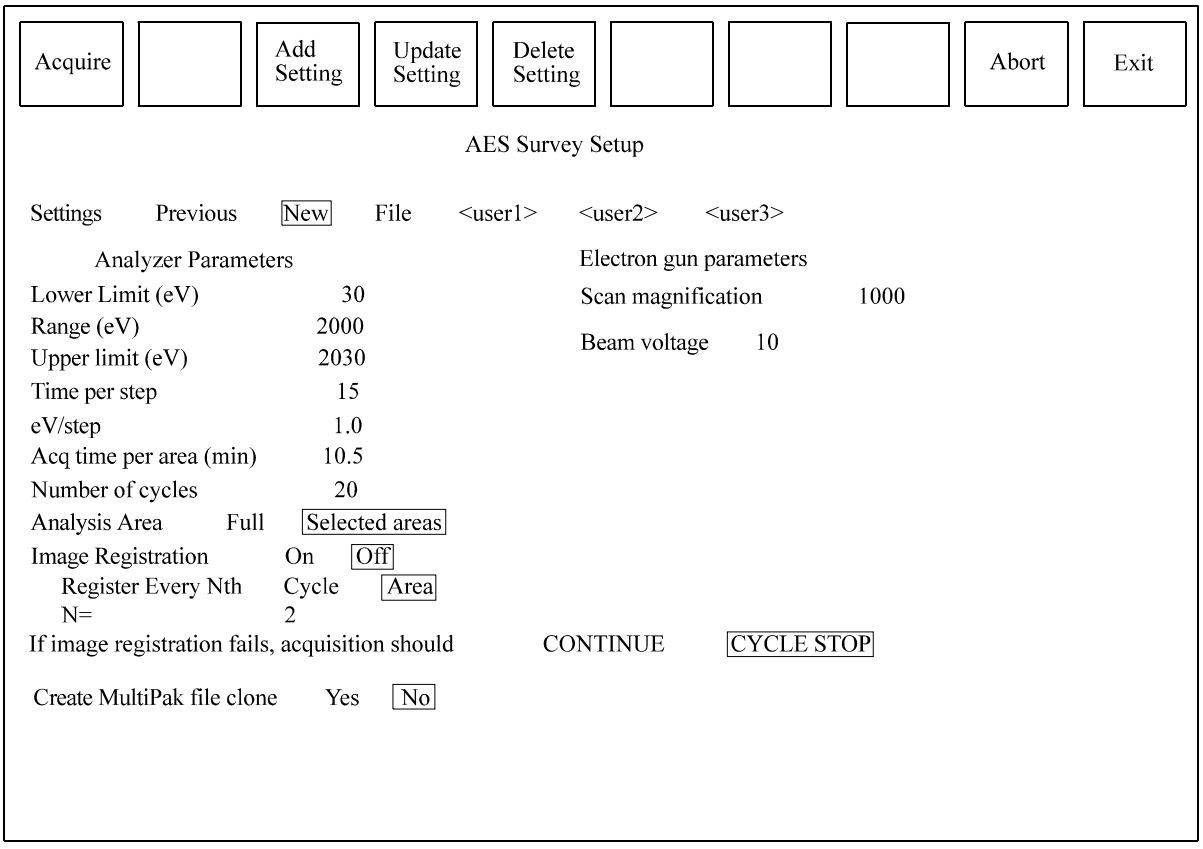


Figure 4-1. Sample Acquisition Parameters for a Survey Scan.

- If the analysis areas have been defined already (described in Section 3), select Selected Areas in the Analysis Area parameter.
- If Image Registration is set to On, calculate the frequency of registration by looking at the number of areas defined and the number of minutes needed to acquired one cycle. For example, if three areas were defined and a cycle for one area takes a half minute, the image would be registered every minute and a half if N is set to 1.

  An alternative is to perform image registration every N area(s), which would allow more frequent registrations. To choose between these options, determine how long each cycle is going to take and how frequently the image should be registered. Low magnifications require less frequent registrations.
- Specify CYCLE STOP if image registration fails, so the data up to that point will be valid. (Were acquisition to continue even though registration has failed, the integrity of the data would be suspect.)

B. Register the image.

If image registration is being used and enough time has elapsed during setup, the last step before starting data acquisition should be image registration. (If Image Registration was set to Yes in step A, the setup parameters will take care of regularly re-registering the image during the acquisition.)

1. Select Sample Setup–Register Image.

C. Acquire the survey scan.

Acquire the data after the analysis points/areas have been defined (as described in Section 3), acquisition parameters have been entered, and the sample's location has been re-registered.

1. Select Acquire–Acquire Survey.

   Acquisition proceeds, and the current cycle number, current area number, and y-axis minimums and maximums are displayed. When acquisition is complete, the survey scan for area 1 is displayed if Yes was selected in the Display Acquisition parameter in the Display Semiperms menu. If No was selected in the Display Acquisition parameter in the Display Semiperms menu, the screen is blanked when acquisition has concluded.

D. Differentiate the data (and smooth, if needed).

Differentiate the curves before identifying the peaks and printing the results.

*NOTE: The remainder of the steps in this section can be performed using PC-*ACCESS *or MultiPak. They are described here using PC-ACCESS, because preliminary data reduction is often appropriate as part of the acquisition process to ensure that the data taken are appropriately representative of the sample being analyzed.*

1. If the acquisition data are not already displayed, display the file that has just been acquired, as follows:

    a. Select Load Display–Acq Display.

    b. Select a point or area, and press Display.

    c. Exit the Display menu.

2. Differentiate the data, as follows:

    a. Select Next Bank, then Spectral Massage–Diff Setup.

    b. In the N-Point Differentiate field, enter 9.

    c. In the Differentiate field, select All.

    d. Press the Differentiate softkey. The derivative data will be displayed in place of the acquired N(E) data.

    e. Press Exit.

*NOTE: Once a point value has been entered in the Diff Setup (and/or Smooth) Setup menu(s), the operator may select Direct Massage #2–Differentiate (or Direct Massage #2–Smooth) to achieve the same result with fewer steps. The values are retained in the Setup menus until PC-*ACCESS *is exited or the values are changed using the Setup menus.*

3. If the data has enough noise that it will be difficult to identify peaks, smooth the data, as follows:

    a. Select Spectral Massage–Smooth Setup.

    b. In the N-Point Smooth field, enter 9.

c. In the Smooth field, select All.

d. Press the Smooth softkey. The smoothed derivative data will be displayed in place of the derivative data.

e. Press Exit.

4. Select Spectral Massage–Expand. Press Full Scale, then press Expand, then press Exit.

E. Identify the element peaks.

Identify the energies of significant peaks.

1. Select Spectral Massage–Cursor.

2. Drag the cursor to the most negative excursion of a peak in the data.

3. Using the *Handbook of Auger Electron Spectroscopy*, find that energy on the chart of Principal Auger Electron Energies (on the inside of the back cover) and in the handbook's Appendix B to narrow the list of possible elements for which this is a peak.

4. Compare the peaks in the acquired data to the standard spectra for each of the possible elements to make a positive identification of the element for which this is a peak.

   NOTE: In the case of low concentrations, the most prominent peaks may be the only ones observable in the survey data.

5. Label the peak, as described in the following subsection.

6. Repeat steps 1 through 5 for each peak in the current spectrum.

7. Repeat step D, "Differentiate the Data," to display and massage the data from the next area or point.

8. Repeat steps 1 through 7 until the peaks of all the areas/points have been identified.

F. Label the peaks.

Label significant peaks for easy reference.

1. Select Spectral Massage–Graph Annotate, press Annotation Text, and press Enter. A box is displayed.

2. Type the desired annotation (e.g., the element abbreviation or transition name from the handbook's Appendix B) in the highlighted field, press Enter, and drag the box to the peak using the right mouse button. Press the Enter Text softkey to add the annotation to the graph.

3. Repeat this for each of the significant peaks on the spectra of each area/point.

4. Press Exit.

G. Print the annotated spectrum.

Print hard copies of the annotated spectra.

1. Select Output Control–Print Setup to find out what the current default printer is and change it, if necessary. (Press the down arrow to see what's available and select the desired printer.) Press OK.

2. Select the Spectral Massage–Print Graphics (or press Ctrl+F12) to get a hard copy of each spectrum.

H. Get atomic concentration data.

Quantify the composition of the analysis areas/points.

1. Select AC Massage–AC Setup.

2. Select the New setting (unless the regions defined in the Previous setting are applicable).

3. Specify the analysis area/point of interest.

4. In the Massaged Data field (if present), select No so a 5-point differentiation will be applied to the raw data automatically prior to calculating the atomic concentration percentages. Select Yes, instead, if you have already differentiated the data and you want to use that massaged data for the AC calculation.

NOTE: The sensitivity factors stored in the database for AC calculations are based on a 5-point differentiation.

5. If Yes was selected in step 4 (and the data has already been differentiated), go to step 6.

   If No was selected in step 4 (or Yes was selected but the data has not yet been differentiated), press the dN(E) softkey to differentiate the data to make it easier to check the analysis window boundaries.

6. In Element Name, enter a "region" name.

   The Element Name field is tied to the database listed in Appendix A, which contains predefined "regions." The database information is automatically placed in the region's parameter fields when the region name is entered. The region's range is also displayed when it is entered. The vertical lines connote the boundaries of the *acquisition* window, and the dashed lines connote the boundaries of the *analysis* window.

7. Press the Expand Region softkey.

   In this case, the x-axis limits are the acquisition window boundaries, and the dashed lines are still the boundaries of the analysis window.

8. Change the analysis window boundaries, if needed, as follows:

   a. Highlight the Left–Analysis parameter and use the right mouse button to move the region boundary, if needed to include the most positive excursion of the region's peak.

   b. Highlight the Right–Analysis parameter and use the right mouse button to move the region boundary, if needed to include the most negative excursion of the region's peak.

      NOTE: Since this is a survey, there are more data to the left and right of the boundaries of the acquisition window. If needed, change the Left (or Right) Acquisition *value to a lower (or higher) energy, then redisplay the region by pressing Display Original, then Expand Region.*

9. Press Add Region, which adds this region to the list of regions to be included in the atomic concentration calculation.

10. Repeat steps 6 through 9 for each region to be included in the AC calculation.

11. When some of the areas/points are known to not contain one or more of the elements, those elements can be deselected for those areas/points by

highlighting the corresponding region name and pressing the Exclude Region softkey. Deselected regions are shown in magenta, and selected regions are displayed in white. The Include Region softkey reselects the highlighted region. (The Delete Region softkey removes the region from the menu entirely.)

12. Generate and print the AC computation in one of the following ways:

    - Press AC Table to display the table in the PC-*ACCESS* window. The percentages are calculated and displayed. Press Ctrl+F10 to print the results. (Press Ctrl+F9 to copy the results to the clipboard.)

    - Press Table to Notepad to display the table in Notepad on the Windows desktop. The percentages are calculated and displayed. Edit the text file, if desired; then, print the results by selecting File–Print from the Notepad menu. (Highlight the contents, then select Edit–Copy to copy the results to the clipboard.)

13. The AC data from more than one area/point can be collected in a single summary file, if desired, as follows:

    a. Press Exit.

    b. Select ACMassage–ACSummarySetup.

    c. Examine the contents of the summary file. If desired, press the Clear AC Summary softkey to remove all the data in the summary file, or highlight the data to be removed and press Delete Entry.

    d. Press Exit.

    c. Press AC to AC Summary to send the AC data from the current area/point to the summary file.

    e. To print the results, select ACMassage–ACSummarySetup and press the Table to Notepad softkey. Edit the text file, if desired; then, print the results by selecting File–Print from the Notepad menu. (Highlight the contents, then select Edit–Copy to copy the results to the clipboard.)

14. Repeat steps 3 through 13 until AC data have been calculated for every area/point.

15. Press Exit in the AC Setup menu when done.

Section 5:
Map the Major Elements and Analyze the Results

Because low concentrations of highly localized elements can be easily overlooked, complete surface characterization requires careful observation. Acquiring elemental maps, then superimposing them upon each other reveals any unidentified areas on the surface. Point spectra acquired from these areas quickly identify one or more new elements, which are then mapped and added to the overlay. This overlay of maps provides a picture of the two-dimensional distribution of surface elements.

This section describes mapping the elements and analyzing the results. A 2-point map, described here, is the most common map acquisition. A 3-point map acquisition is usually used when the signal is small and is riding on a steep background. The steps for map acquisitions are the following:

A. Register the image.

B. Set up for map acquisition.

C. Do a Test Acquire for each element.

D. Re-register the image.

E. Acquire the maps.

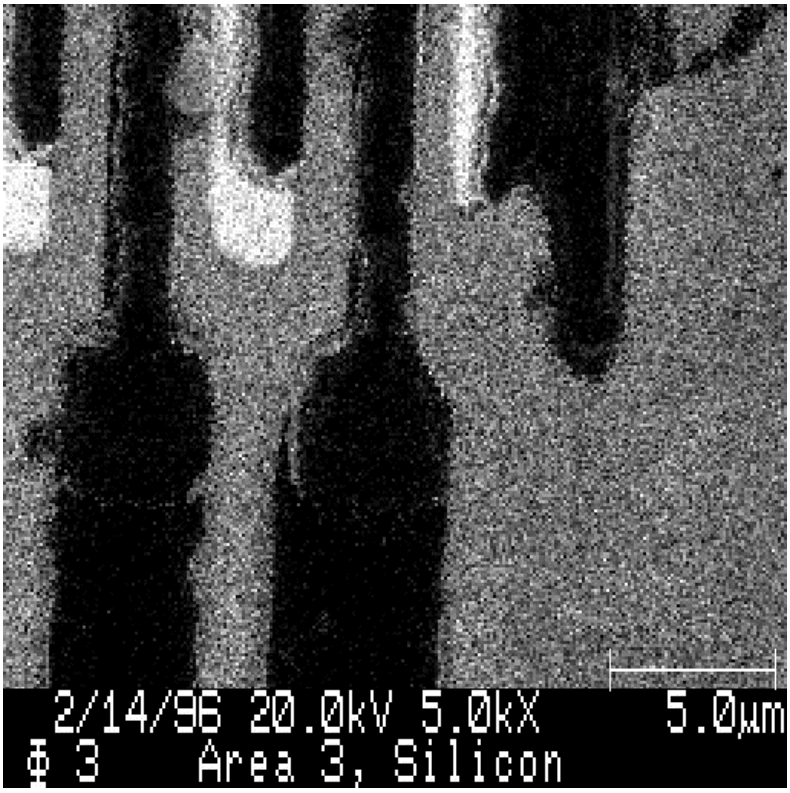
F. Prepare an Auger map file of each region.

G. Create and print a color overlay of maps.

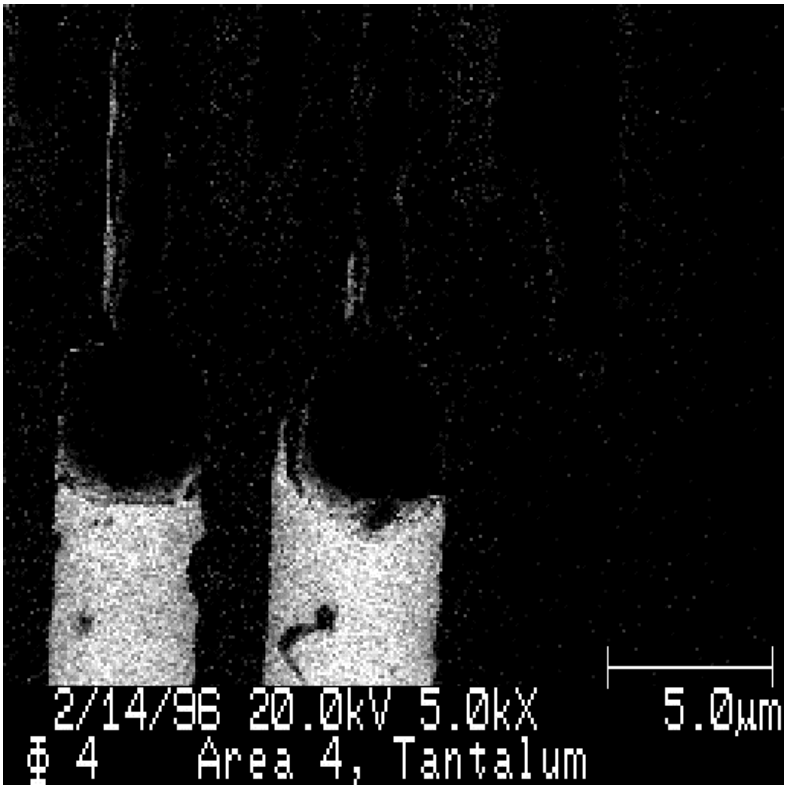
H. Analyze the color overlay of the Auger maps.

Figure 5-1 shows maps of three regions acquired from the same sample, and Figure 5-2 shows a color overlay of the maps.

NOTE: The following procedure assumes that the analysis area has been positioned at the focal point of the analyzer, the electron gun parameters and current have been set, and the optics have been optimized. These steps are described in detail in Section 3, subsections J through M.

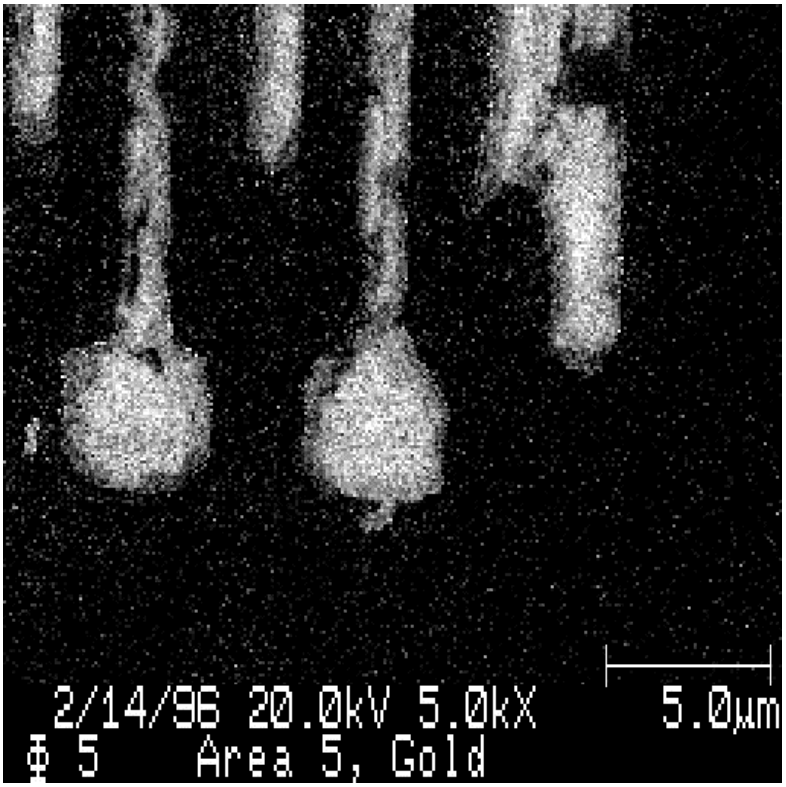


a. silicon (Si2)



b. tantalum (Ta1)

Figure 5-1. Three Maps from the Same Sample.



c. gold (Au3)

Figure 5-1. Three Maps from the Same Sample (concluded).

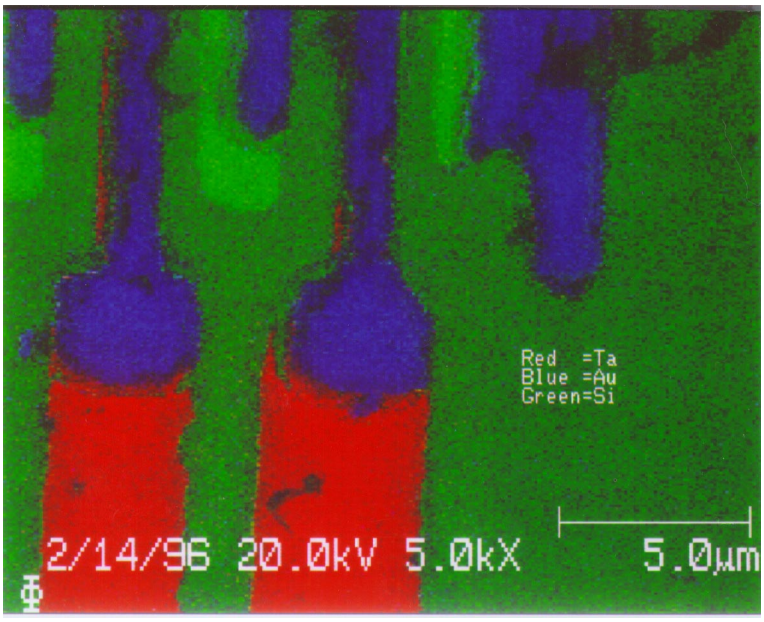


Figure 5-2. Color Overlay of the Three Maps in Figure 5-1.

A. Register the image.

Before setting up to acquire maps, perform image registration to compensate for normal drifting of the stage that may have occurred since the last registration.

1. Select Sample Setup–Register Image (or select Hardware Config–SEM, press Image Shift, and press Exit to return to the previously displayed command bank).

   NOTE: Once image registration is set up for a sample, it is paramount that the image is frequently re-registered. If the sample drifts outside the reference area, image registration will be lost.

B. Set up for map acquisition.

1. Select Setup Acquire–Setup Map, select New, then 2-Point Map, and click the right mouse button (or press Enter). The AES Map Setup #1 menu is displayed.

2. Highlight Element Name and type the name of the region (transition name from Appendix A) to be mapped. The Peak Energy and Background Energy parameters are filled in automatically from the database.

3. Specify the parameters for each region, as follows:

   - Time Per Step—1 ms is typical, since, in most cases, making a map relatively quickly is adequate;

   - Sweeps per Line—1 is typical;

   - Number of Frames—determined by the amount of time to be spent on this region. Consider factors like its concentration, signal strength, and importance relative to the other elements to be mapped. (Since the Next Region or Frame Stop softkey can be pressed during acquisition to stop acquisition when enough data have been acquired, it may be appropriate to enter even more frames than will actually be acquired.)

4. Press Add Region.

5. Repeat steps 2, 3, and 4 until all regions of interest have been entered.

6. Press Next Menu to display the AES Map Setup #2 menu.

7. Choose the number of "lines" (32, 64, 128, 256, or 512). The menu shows the estimated time it will to take to acquire that data.

   NOTE: The lines parameter determines the pixel resolution of the images generated. For example, 128 means 128 points × 128 lines. The resolution selected directly affects the time needed to acquire the map. For example, when 265 is chosen, the time increases by a factor of 4 over the time needed when 128 is chosen.

8. Set Normalized to Yes. Select Gray Level in Display Mode.

9. Set Image Registration to On, if desired, and specify the Register Every Nth parameter. Typical settings are Frames and N = 1. Specify Frame Stop if image registration fails.

10. Select Yes or No in the MultiPak Clone parameter. Yes is specified if the data is to be manipulated further at a later time using PHI's MultiPak software.

C. Do a Test Acquire for each region.

A Test Acquire for each region to be acquired should be taken to make sure that the analysis windows of each region to be acquired are optimal. The region boundaries should accept as much good signal as possible, while excluding data from another peak, if possible.

1. Press the Test Acquire softkey to open the AES Test Acquisition menu.

2. Select the region to look at. (The first region is selected automatically when the menu is first opened.)

3. Select the Point Coordinates fields and use the arrow keys to drive the pointer to identify a place on the sample that has the highest probability of having that element in a high concentration.

4. Specify the number of sweeps for the test acquisition. Five is a typical value.

5. Select dN(E) in the Display mode parameter to show differentiated data to make it easier to check the analysis window boundaries.

6. Press the Test Acquire softkey to begin test acquisition.

   The acquired data is displayed on the right side of the PC-*ACCESS* window, and "Acquisition complete" is shown below that when the acquire is done.

Two sets of vertical lines are also displayed with the data (in addition to the grid lines). The left set of dashed lines represents the peak-energy window*, and the right set of dashed lines represents the background-energy window.

NOTE: The solid line between the dashed lines of each window is not made use of in the Test Acquire routine. It is related to the MultiChannel Detector channel plates.

7. Highlight the Peak Energy parameter in the AES Test Acquisition menu, and use the arrow keys (or right mouse button) to move the peak-energy (left) window so that the majority of the signal from the most positive excursion of the peak lies between the dashed lines.

8. Highlight the Background Energy parameter, then move the background-energy (right) window so that the signal at the most negative excursion of the peak is minimized within that window.

   NOTE: For higher-energy peaks, it may be advantageous to narrow the peak and/or background energy windows. This can be done by turning off some of the channels, although this will reduce total signal strength. If channels are to be turned off, they must be selected in a specific order. One of the following sequences must be used, turning off channels only until the desired window width has been achieved:

   - *1 — 2 — 3 — 4 — 8 — 7 — 6 — 5*
   - *1 — 8 — 2 — 7 — 3 — 6 — 4 — 5*

9. Press Next Element, and repeat steps 2 through 8 for each region, until every region has had a test acquire performed.

10. Press Exit to close the AES Test Acquisition menu.

D. Re-register the image.

Perform image registration to compensate for normal drifting of the stage that may have occurred since the last registration.

1. Select Sample Setup–Register Image to re-register the image.

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* The width of the "window" is approximately 2% of the peak energy, so the window's width increases with increasing energies. That is, if we are looking at a peak energy of 100 eV, the width of the window is ~2 eV; the width of the window at a peak energy of 1600 eV is ~32 eV.

E. Acquire the maps.

1. Select Acquire–Acquire Map.

   Map acquisition begins. The incident electron beam steps point by point along a line while peak intensity is measured at each point. The beam repeats the motion on the same line to measure background intensity, at each point subtracting the background intensity from the peak intensity and saving only the value of the difference.

   This is then repeated for the next line, and so on until all the lines have been acquired. This is one frame. This is then repeated for the next frame, at each point calculating and saving only the *average* of [peak-minus-background intensity at this point on this sweep] and [the value stored at this point during the preceding sweep]. When the specified number of frames is completed, the acquisition for the next region begins.

   As data are acquired, "y-min" and "y-max" values are displayed and continually updated. The "Present" values are the actual minimum and maximum of the data being displayed, and the "Total" values indicate the scale for the y axis of the display.

   During acquisition, the operator can press any of the following softkeys:

   - Background to do data manipulation on another file (excluding SEM or map files).
   - Next Region when satisfied with the signal-to-noise ratio in the map currently displayed on the image monitor. The data collected are saved in a map file. The system will move to acquisition of the next element when the current frame is completed.
   - Frame Stop to terminate acquisition after completing the current frame. The data collected are saved in a map file.
   - Region Stop to stop acquisition after acquiring all frames for the current region. The data collected are saved in a map file.
   - Abort Acq to stop the acquisition almost immediately. Any data acquired are not saved.

   NOTE: The DSM will return to a live image for image registration (if image registration was specified in the setup information).

   When acquisition of all the regions to be mapped is complete, the screen is blanked.

F. Prepare an Auger map file of each region.

When map acquisition is complete, all the region maps have been saved to a single map file. The next step is to optimize the appearance of the data from each region using image processing. Several image processing techniques are available, but the most common and useful technique is Contrast Stretch, which is described here.

Auger maps cannot be displayed both in color and with image processing in PC-*ACCESS*, so (1) processing is done in the Gray mode, and (2) processed maps of individual regions are saved to separate files if they are to be colorized afterward using PC-*ACCESS* (as described in Subsection 5G).

NOTE: Color Auger maps and overlays can be displayed and printed from the raw *data in PC*-ACCESS*, but resolution would not be as quite as good as it would be with image processing of the data first.*

NOTE: MultiPak is the recommended software for map data reduction.

1. Select Hardware–SEM, and ensure that the beam voltage and magnification are set to the same values at which the map was originally acquired.

   These values are read from the SEM menu for (1) display of parameters when outputting maps from the Setup Photo menu, and (2) placement in the header lines of any files generated and saved from the map file.

2. Select Acquire–AcqDisplay. The Map Display menu opens.

3. Specify a region.

4. Choose Gray for Display Mode, and select 256 for the Display Level. Select No for Quadrant Display.

5. Press the Display softkey. The Auger map of the region is displayed on the DSM.

6. Press the Image Process softkey. A histogram of that map is displayed.

7. Do a Contrast Stretch while watching the image on the DSM, as follows:

   a. Press the Contrast Stretch softkey.

   b. Select the High Intensity parameter and press the arrow keys to move the high-intensity (right) vertical cursor.

c. Select the Low Intensity parameter and press the arrow keys to move the low-intensity (left) vertical cursor.

NOTE: Be careful not to manipulate the contrast so much that information is lost.

d. Press the Execute softkey, when the brights are very bright where there is more of the element (more counts at that transition's peak energy) and some of the darker areas are relatively dark.

The DSM display is refreshed with the new contrast values applied to the Auger map.

8. Press Exit.

9. Output the image-processed Auger map of the region, as follows:

NOTE: It can be helpful to revise the file naming scheme at this point (using Sample Setup–Sample Setup) to make it easier later to discern between the acquired map file and the image-processed region files to be created in this step.

a. Press the Photo Setup softkey.

b. Select On or Off for each parameter.

NOTE: The setting of the Annotation Background parameter is the only parameter information saved in the file created using the Setup Photo menu. Annotation Background is typically set to Off when the file is written, because Annotation Background replaces the image at the bottom of the photo with a black band. You might, however, turn Annotation Background on to print a hard copy.

c. Highlight Photo, File, Printer, and/or Clipboard in the Output Device parameter, and press Enter or click the right mouse button.

Selected devices are displayed in white; non-selected devices are displayed in magenta. Any combination of output devices is valid.

"Photo" advances the photo number automatically. "File" stores the displayed DSM image to a file with the file name displayed at the bottom of the computer screen when the save is complete. "Printer" sends the image to the printer output selected in the Print Setup menu (from the Output Control command bank). "Clipboard" places a copy of the image in the Microsoft Windows clipboard.

d. Press the Output Image softkey. The image will be sent to the device(s) selected.

When Output Image is pressed to create the map file, the file name will be displayed at the bottom of the Setup Photo menu. Note the file name associated with each region for later reference.

e. Press Exit.

10. Repeat steps 3 through 10 for each region of interest until done.

G. Create and print a color overlay of maps.

To colorize maps and create overlays from them, maps of individual regions are image-processed and saved to separate files first, as described in Subsection 5F. The following procedure describes creating and printing a color overlay from the individual files.

*NOTE: In PC-*ACCESS*, color maps and overlays can be displayed and printed but not saved in a color file or copied in color to the clipboard. MultiPak can, however, save color images and copy them to the clipboard.*

1. Choose three of the regions for which map files were created in step 5F, and load their files, as follows:

NOTE: Three maps are used to create a color overlay, because three colors are available: red, blue, and green.

a. Press Load Display–Dir, and use the arrow keys or the mouse to highlight the directory containing the map files.

b. Press List Files, then Execute to display the contents of the highlighted directory.

c. Using the right mouse button, click on the file name that contains the massaged data from the first of the three regions to be used in the overlay. The Load menu is displayed, and that file is listed on the loaded files.

d. Click in the next field, type the file name that contains the massaged data from the second of the three regions to be used in the overlay, and press Enter.

e. Click in the third field and type the file name that contains the massaged data from the third of the three regions to be used in the overlay, and press Enter.

2. Highlight one of the three loaded map files, and press Display Menu.

3. In Display Mode, select Color. In Memory Map, select Red, Blue, or Green. In Display Level, select 4, 8, or 16. Select Yes or No in Quadrant Display.

   NOTE: When Blue is selected in Memory Map, always select 8 in Display Level. (Half of the blue plane of memory is used to display the PHI symbol (Φ) and other annotation.)

4. Press Display to display the first of the three maps, but use Display Add Full to display the additional two maps.

5. Repeat steps 2, 3, and 4.

   The three-color map overlay is displayed on the DSM.

6. Press the Photo Setup softkey.

7. Set to On the parameters that are to be displayed on the color overlay.

8. Add text to the overlay to indicate which element is in what color (e.g., Red = Ta, Blue = Au, Green = Si), as follows:

   a. Press DSM Text Annotate.

   b. Choose a font size (2 is typical).

   c. Highlight the Cursor Location parameter and use the arrow keys to locate the beginning of the annotation text.

   c. Press Enter Text and type the annotation text.

   d. Press End Text Entry.

   e. Repeat steps a through d until done.

9. Press Previous Menu to eliminate display of the position cursors.

10. On the Mitsubishi printer, press Memory, then Print.

11. Press Exit to return to the command bank.

H. Analyze the color overlay of the Auger maps.

Examine the overlay(s) to determine whether there are areas that might require further analysis. For example, areas showing little or no color might contain previously unidentified elements.

Section 6:
Perform a 2-Point Line Scan and Analyze the Results

When more definitive structural or position information is needed than a map can give, line scans can provide important additional information, especially for small features. An Auger line scan is a series of data points (in the case of 2- and 3-point line scans) or spectra (in the case of window line scans) collected along a single defined line across a sample. The scans are collected by stepping the electron beam, point by point, along a selected line.

Line scan acquisitions yield a better signal-to-noise ratio from the sample than does a map acquisition, and in a shorter time. Multiple vertical and/or horizontal lines can be scanned, and up to 20 regions can be acquired for each line.

2-point, 3-point, and Window Line Scans

Three kinds of line scans are available: 2-point, 3-point, and Window. Two- and three-point line scans store peak and background energies at every point across the line, whereas a window line scan stores a spectrum at every point along the line.

A 2-point scan saves a single background energy (specified in the E2 parameter) at each point, whereas a 3-point line scan saves two background energies: one is extrapolated from a high-energy background energy (specified in the E2 parameter) and the other is a low-energy background energy (specified in the E1 parameter). Acquiring a 2-point scan is common. The 3-point scan is usually used to measure a small peak intensity on a sloping background, primarily when only a small amount of the element is present.

Having a peak-shape spectrum, which is acquired with the window line scan, makes extraction of chemical state information possible. If the Auger peak energy shifts or the peak changes shape because of chemical state changes or sample charging effects, a 2-point line scan will simply show a change in intensity, whereas such changes will be readily evident in a window line scan.

One benefit of the window line scan is that atomic concentration data can be generated, but, since a window line scan collects much more data, acquisition requires much more time than does a 2- or 3-point line scan.

Acquisition of 2-point line scans is described in this section, and acquisition of window line scans is described in Section 7.

The steps described for acquiring a 2-point line scan and analyzing the results are the following:

A. Define the line.

B. Set up a 2-point acquisition.

C. Do a Test Acquire for each region.

D. Register the image.

E. Acquire the line scan data.

F. Normalize the data.

G. Smooth the data.

H. Expand the data.

I. Make a composite of the line scans.

J. Annotate the image.

K. Print the results.

L. Overlay the spectra on the SEM and print.

NOTE: The following procedure assumes that the analysis area has been positioned at the focal point of the analyzer, the electron gun parameters and current have been set, and the optics have been optimized. These steps are described in detail in Section 3, subsections J through M.

A. Define the line.

The first step is to position the line(s) to include the features of interest.

1. Display on the DSM (digital storage monitor) one of the following images:

   - The SEM of the sample area currently at the focal point of the analyzer;

   - An Auger map of the sample area currently at the focal point of the analyzer. (The map may reveal a feature of interest that cannot be detected from the SEM.)

2. Select Sample Setup–Line Define. Press Delete All, if necessary, to remove any previously defined lines.

3. Specify the orientation of the line to be defined: horizontal or vertical. A line is then displayed on the DSM.

4. Use the arrow keys to place the line where the analysis is to be performed, then press the Define Line softkey.

   The line's position is saved as Line 1 and identified by its coordinate location.

   NOTE: Holding the Shift key down while using the arrow keys will move the cursor in larger increments.

5. Define additional lines, if desired.

6. Press Exit.

B. Set up a 2-point acquisition.

Define the acquisition parameters.

1. Select Setup Acquire–Setup Line, select New, and press Enter or click the right mouse button. The AES Line Setup menu is displayed.

2. Select 2-point in the Acq Method field.

3. Type the name of the region (region name from Appendix A) to scan, and press Enter. The system automatically enters values for that transition into several of its parameter fields.

   Specify the Number of Sweeps and Time per Step for each region. Twenty milliseconds is a typical Time per Step value. The Number of Sweeps values specified depend on the intensity of each element and the desired signal-to-noise ratio.

   For example, when an element's content is expected to be low relative to the other elements, 10 sweeps might be specified. On the same sample, five sweeps might be specified for an element like oxygen if it is relatively abundant. If the sample also has tantalum and gold, whose signals are relatively strong, 2 sweeps might be specified for each.

   On a sample that is expected to have both silicon dioxide and silicon, specify the same number of sweeps for silicon as for oxygen.

4. Press the Add Region softkey, and repeat step 3. Repeat this step until all regions are entered.

5. Press Next Menu. The second page of the AES Line Setup menu is displayed.

6. The acquisition time per line, in minutes, needed for 1 cycle at 128 "pixels" per line is displayed. Use this value as a gauge to select the points per line to acquire. (The higher the number is, the more data is acquired.) When the right mouse button is clicked, a revised acquisition time per line for one cycle will be displayed.

7. Choose the number of cycles to be acquired, and click the right mouse button. The acquisition time per line for one cycle will be revised and displayed again.

8. Turn Image Registration to On or Off. If On is selected, also do the following:

   a. Decide how frequently to register the image, taking into account the acquisition time per line for one cycle, and enter the Register Every Nth parameter accordingly.

      NOTE: If the time between registrations will be too long, adjust the Points per Line value in this menu and/or the Time per Step and/or Number of Sweeps parameter values in the Previous Menu for one or more of the regions until the acquisition time per line for one cycle is appropriate for the frequency of registration needed.

   b. Select CYCLE STOP to indicate that acquisition should stop after the cycle if image registration fails.

9. Once the length of one cycle is known, the number of cycles can be increased according to how long the entire acquisition should last. (A much higher number of cycles can be specified for a 2-point line scan than for an Auger map and still acquire the data in a short time.)

10. Select Yes or No in the last parameter, "Create a MultiPak file clone."

C. Do a Test Acquire for each region.

Perform the Test Acquire procedure given in Section 5C.

D. Register the image.

If image registration is being used and enough time has elapsed during setup, the last step before starting data acquisition should be image registration. (If Image Registration was set to Yes in step C, the setup parameters will take care of regularly re-registering the image during the acquisition.)

1. Select Sample Setup–Register Image.

E. Acquire the line scan data.

Acquire the data after the lines have been positioned, acquisition parameters have been defined, test acquisitions have been performed, and the sample's location has been re-registered.

1. Select Acquire–Acquire Line.

   Data is displayed as it accumulates. The intensity along the line for each region acquired indicates the relative composition of that element at that position. Each line is identified by line number and is shown on the DSM, and each element is identified by region number.

2. If desired, press the CYCLE STOP softkey to stop the acquisition at the end of the current cycle when sufficient data has been acquired. Otherwise, allow the acquisition to run its full course.

   When acquisition is complete, the line scan for region 1 is displayed if Yes was selected in the Display Acquisition parameter in the Display Semiperms menu. If No was selected in the Display Acquisition parameter in the Display Semiperms menu, the screen is blanked when acquisition has concluded.

   Figure 6-1 shows a line scan of oxygen on a sample.

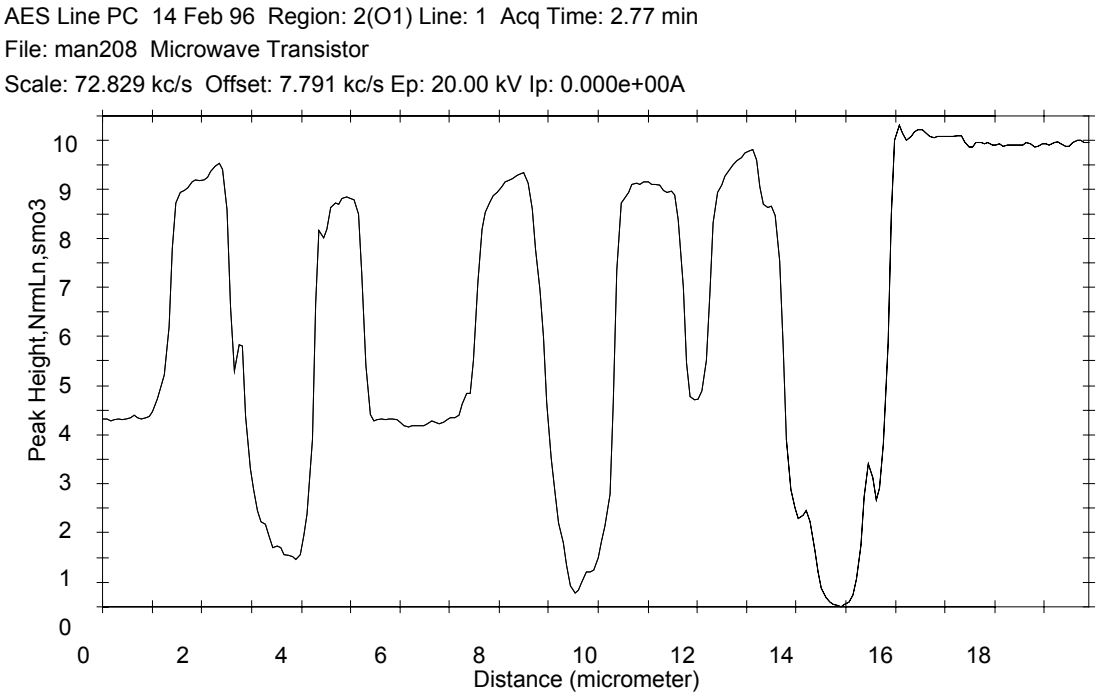

Figure 6-1. Line Scan of Oxygen on a Sample.

F. Normalize the data.

If more than one region was acquired, normalize the data from each region before comparing them to each other to help correct for the variation in signal strength due to topography. Normalizing takes the peak-minus-background data acquired and divides it by the background.

1. Load the line scan data file and display it. The Line Display menu opens.
2. In the Destination parameter, select Here.
3. Highlight the Line parameter, type the line number (e.g., 1), and click the right mouse button.
4. Highlight the Region parameter, type the region number (e.g., 1), and click the right mouse button.
5. Press Display, then Exit.
6. Select Line Massage–Line Norm. The normalized data is displayed.
7. Press Display to reopen the Line Display menu.
8. Repeat steps 2 through 7 until all the regions have been normalized.

G. Smooth the data.

Smooth the data, if desired.

1. Select Line Massage–Smooth Setup.
2. Enter a value in the N-Point Smooth parameter.
3. Select All or Current in the Curve parameter.
4. If Current was selected in step 3, press Pick Curve. The "current" curve is displayed in white. Press Previous Curve or Next Curve until the desired curve is displayed in white, then press Pick.
5. Press Smooth, then Exit.

H. Expand the data.

Three expand routines are available, but the one most commonly used for line display is Full Vertical.

1. Select Line Massage–Expand.
2. Press Full Vertical, then Expand. The selected (white) line is redisplayed using the maximum vertical space possible.
3. Press Pick New Curve, then Previous Curve or Next Curve, then Pick to select another curve in the line scan.
4. Repeat steps 2 and 3 until each line has been expanded.
5. Press Exit.

I. Make a composite of the line scans.

Create a composite of the normalized line scans to show where each element is high in concentration relative to the others.

1. Select Line Massage–Display.
2. Type the line number, and press Enter or click the right mouse button.
3. Type the region name, and press Enter or click the right mouse button.
4. Highlight Yes in the Massaged Data parameter.
5. Press Display if this is the first region, or press Display Add FULL if this is an additional region.
6. Repeat steps 2 through 5 until the composite is done.

   Figure 6-2 shows a composite of normalized line scans.

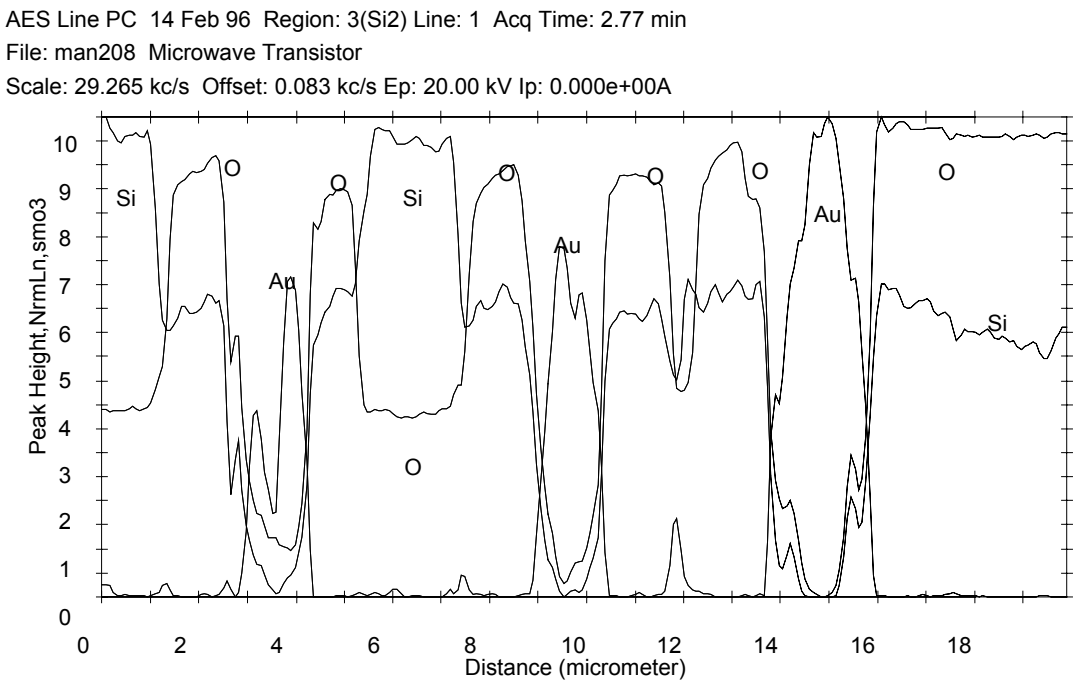

Figure 6-2. Composite of three regions scanned, normalized, smoothed, and expanded.

J. Annotate the overlay.

Add information as appropriate to the composite before printing the data.

1. Select Line Massage–Graph Annotate.
2. Press Annotation Text and press Enter. A box is displayed.
3. Type a label, such as the element abbreviation or region name, in the highlighted field, and press Enter.
4. Using the left mouse button, drag the box to the desired position and release the mouse button.
5. Repeat steps 2 through 4 as needed.
6. Press Exit when done.

K. Print the results.

Print hard copies of the annotated data.

1. Select Output Control–Print Setup to find out what the current default printer is and change it, if necessary. (Click the down arrow to see what's available and select the desired printer.) Click OK.

2. Select Line Massage–Print Graphics (or press Ctrl+F12) to get a hard copy of each spectrum.

L. Overlay the spectra on the SEM and print.

Correlate the features shown on a map or SEM with an individual line scan by creating an overlay.

1. Load and display the appropriate image on the DSM.

2. Select Line Massage–Display.

3. Specify the line number and region name to be displayed with the SEM. Select DSM in the Destination parameter, and highlight Yes or No in the Massaged Data parameter.

4. Press Display.

5. Press Exit.

6. Press the Photo Setup softkey. In the Setup Photo menu, select which fields you want to appear on the photo.

7. Highlight Photo, File, Printer, and/or Clipboard in the Output Device parameter, and press Enter or click the right mouse button. Selected devices are displayed in white; non-selected devices are displayed in magenta. Any combination of output devices is valid.

   "Photo" advances the photo number automatically. "File" stores the displayed DSM image to a file with the file name displayed at the bottom of the computer screen when the save is complete. "Printer" sends the image to the printer output selected in the Print Setup menu (from the Output Control command bank). "Clipboard" places a copy of the image in the Microsoft Windows clipboard.

8. Press Output Image. The image will be sent to the device(s) selected. (To print on the Mitsubishi device, also press Memory and Print on that device.)

9. Repeat steps 3 through 8 as needed.

10. Press Exit to close the Setup Photo menu.

Example of Line Scan Overlay

Figure 6-3 shows an overlay of the oxygen region shown in Figure 6-1 overlaid on an SEM.

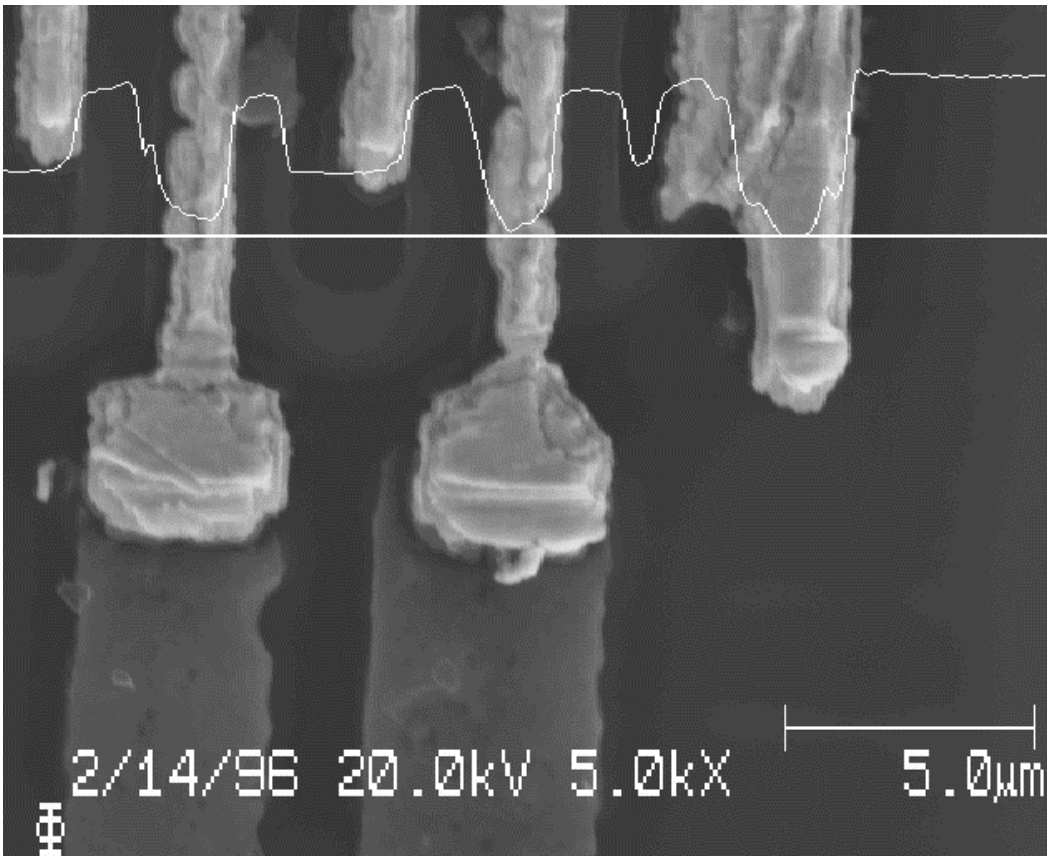

Figure 6-3. *Oxygen Line Scan Overlaid on an SEM.*

Section 7:
Perform a Window Line Acquisition and Analyze the Results

The introduction to Section 6 describes the types of line scans and how to select the appropriate type for acquisition. This section describes acquiring and analyzing a window line scan, as follows:

A. Define the line.

B. Set up a window line acquisition.

C. Do a Test Acquire for each region.

D. Recheck the acquisition setup parameters.

E. Display and register the image.

F. Acquire the line scan.

G. Generate an AC line scan.

H. Analyze the montage display of the data.

NOTE: The following procedure assumes that the analysis area has been positioned at the focal point of the analyzer, the electron gun parameters and current have been set, and the optics have been optimized. These steps are described in detail in Section 3, subsections J through M.

A. Define the line.

Perform the procedure given in Section 6A.

B. Set up a window line acquisition.

1. Select Setup Acquire–Setup Line, select New, and press Enter. The AES Line Setup #1 menu is displayed. Select Window in the Acq Method field.

2. Type the name of the region (from Appendix A) to scan, and press Enter. The system automatically enters values for that transition into several of its parameter fields.

   NOTE: The computer supplies default values for the acquisition parameters. The acquisition window is the energy range over which data will be acquired, and the analysis window is the range used for peak intensity measurements. The limits for the analysis window are usually a few eV inside the acquisition window limits.

3. Specify sweeps and time per step.

   It is useful to define a modest parameter set to get an idea of acquisition times, just as we did at the beginning of the 2-point setup process (described in step 6B.3). Because window acquisitions take a lot of time (to sweep the analyzer across the range of the region) and because it is best to image register frequently, specify short cycles (e.g., 1 ms per point and 1 sweep for all regions).

4. Press the Add Region softkey.

5. Repeat steps 2, 3, and 4 until all regions are entered.

6. Press Next Menu. The AES Line Setup #2 menu is displayed.

7. The acquisition time per line, in minutes, needed for 1 cycle at 128 "pixels" per line is displayed. Use this value as a gauge to select the points per line to acquire. (The higher the number is, the more data is acquired.) When a new value is selected and the right mouse button is clicked, a revised acquisition time per line for one cycle will be displayed.

8. Choose the number of cycles to be acquired, and click the right mouse button. The acquisition time per line for one cycle will be revised and displayed again.

   NOTE: Because a window line acquisition takes a long time but yields excellent data, the acquisition could continue overnight and still be acquiring when work hours begin the next day. (In this case, the operator would then press CYCLE STOP in the morning, then press Exit.) Set the number of cycles to a large number (e.g., 500) to run the acquisition overnight.

9. Turn Image Registration to On or Off. If On is selected, also do the following:

    a. Decide how frequently to register the image, taking into account the acquisition time per line for one cycle, and enter the Register Every Nth parameter accordingly.

    NOTE: If the time between registrations will be too long, adjust the Points per Line value in this menu and/or the Time per Step and/or Number of Sweeps parameter values in the Previous Menu for one or more of the regions until the acquisition time per line for one cycle is appropriate for the frequency of registration needed.

    b. Select CYCLE STOP to indicate that acquisition should stop after the cycle if image registration fails.

10. Once the length of one cycle is known, the number of cycles can be increased according to how long the entire acquisition should last.

11. Select Yes or No in the last parameter, "Create a MultiPak file clone."

C. Do a Test Acquire for each region.

A Test Acquire for each region to be acquired should be taken to make sure that the analysis windows of each region to be acquired are optimal. The region boundaries should accept as much good signal as possible, while excluding data from another peak, if possible.

1. Press the Test Acquire softkey to open the AES Test Acquisition menu.

2. Select the region to look at. (The first region is selected automatically when the menu is first opened.)

3. Select the Point Coordinates fields and use the arrow keys to drive the pointer to identify a place on the sample that has the highest probability of having that element in a high concentration.

4. Specify the number of sweeps for the test acquisition. Five is a typical value.

5. Select dN(E) in the Display mode parameter to show differentiated data to make it easier to check the analysis window boundaries.

6. Press the Test Acquire softkey to begin test acquisition.

   The acquired data is displayed on the right side of the PC-*ACCESS* window, and "Acquisition complete" is shown below that when the acquire is done.

7. Ensure that the full peak (both the most positive and negative excursions of the data) is contained within the analysis window.

   If the window is too wide or to reduce analysis time, reduce the window width, while still ensuring that the positive and negative excursions remain within the window, as follows:

   a. Highlight Lower Limit, type the energy to be the lower limit of the analysis window, and press Enter or click the right mouse button.

   b. Highlight Range, type the amount to add to the lower limit of the analysis window to get the higher energy limit, and press Enter or click the right mouse button.

   c. Press Test Acquire to verify the appropriateness of the analysis window boundaries.

      NOTE: For extracting chemical state information, it is sometimes useful to include a relatively wide energy range below (to lower energies than) the main Auger peak, which could include some of the energy loss structure.

      NOTE: The new Lower Limit and Range values are automatically changed in the AES Line Setup #1 menu.

      NOTE: Remember that the Si2 analysis window may need to accommodate both the Si peak in its elemental state and the Si peak in its oxide state, which is shifted to a lower energy.

8. Press Next Element, and repeat steps 2 through 7 for each region, until every region has had a test acquire performed.

9. Press Exit to close the AES Test Acquisition menu.

D. Recheck the acquisition setup parameters.

Since some region parameters may have changed during the previous step, the acquisition times and resulting frequency of image registration may have changed, too. The AES Line Setup menu parameters should be rechecked.

E. Display and register the image.

If image registration is being used and enough time has elapsed during setup, the last step before starting data acquisition should be image registration. (If Image Registration was set to Yes in step B, the setup parameters will take care of regularly re-registering the image during the acquisition.)

1. Select Hardware–SEM, then press Exit when the menu is displayed. This restores the live image to the DSM, if it is not already displayed.

   NOTE: Changing the Scan Rate parameter to medium, then back to fast also restores the live image to the DSM.

2. Select Sample Setup–Register Image.

F. Acquire the line scan.

Acquire the data after the lines have been positioned, acquisition parameters have been defined, test acquisitions have been performed, and the sample's location has been re-registered.

1. Select Acquire–Acquire Line. The acquisition begins.

2. If desired, press the CYCLE STOP softkey to stop the acquisition at the end of the current cycle when sufficient data has been acquired. Otherwise, allow the acquisition to run its full course.

   When acquisition is complete, the screen is blanked.

G. Generate an AC line scan.

One difference between window line scans and 2- or 3-point line scans is that atomic concentration (AC) information can be calculated from the window line scan data. (AC information cannot be calculated from 2- and 3-point line scan data, because PHI does not supply sensitivity factors for the peak-minus-background N(E) data.)

NOTE: MultiPak is the preferred software for manipulation of window line scan data, because it has noise reduction algorithms that improve the analysis.

This procedure is much like that in Section 4, "Get Atomic Concentration Data," except that:

- Regions are already defined by the window line scan setup,
- AC data can be calculated from a single spectrum from one point on the line (using the AC Setup function) or from all spectra from one line acquisition (using the AC Profile function).

1. Load the window line scan file, and press Display Menu.
2. Select Line number, and press Enter.
3. Specify the regions to be included in the AC calculation, as follows:
   a. Highlight the number of one of the regions, and press Enter.
   b. Select Here as the Destination.
   c. Press the Display softkey if this is the first region, or press Display Add FULL if this is an additional region.
   d. Repeat steps a, b, and c until all the regions to be included in the AC calculation have been added to the display.
4. Press Exit to close the Display menu.
5. Select AC Massage–AC Profile. The percentage of each region is shown as a function of distance from one end of the line to the other.

   If desired, select AC Massage–AC Setup. All regions acquired for Line 1 are listed and displayed in white, meaning they are already selected for inclusion in the AC calculation. Specify the line number in the Area parameter, and specify a point number in the Point parameter. The point number has to be extrapolated by the operator from the number of points acquired and the approximated distance of the point of interest along the line. If desired, highlight any region(s) that should not be included in the AC calculation, and press the Exclude Region softkey. Then, press AC Table to display the table in the PC-*ACCESS* window.

NOTE: All the data have a 5-point differentiation applied automatically as part of the AC calculation routine.

NOTE: The sensitivity factors stored in the database for AC calculations are based on a 5-point differentiation.

6. Press Table to Notepad to display the table in Notepad on the Windows NT desktop.

   If desired, use Table to Notepad to edit and print the AC table

   *NOTE: The table can be pasted into a document from Notepad when Table to Notepad is used or from PC-*ACCESS *when AC Table is used and Ctrl+F10 is pressed.*

   If desired, the AC data from more than one point can be collected in a single summary file, if desired, as follows:

   a. Press Exit.

   b. Select ACMassage–ACSummarySetup.

   c. Examine the contents of the summary file. If desired, press the Clear AC Summary softkey to remove all the data in the summary file, or highlight the data to be removed and press Delete Entry.

   d. Press Exit.

   c. Press AC to AC Summary to send the AC data from the current point to the summary file.

   e. To print the results, select ACMassage–ACSummarySetup and press the Table to Notepad softkey. Edit the text file, if desired; then, print the results by selecting File–Print from the Notepad menu. (Highlight the contents, then select Edit–Copy to copy the results to the clipboard.)

7. Press Exit to close the Atomic Concentration menu when done.

H. Analyze the montage display of the data.

The montage can demonstrate chemical state differences through changing locations and shapes of peaks. For example, a peak where Si and SiO_2 are combined usually has a broader shape than an Si or SiO_2 peak, and the SiO_2 peak is at a lower energy than the Si peak.

The montage display is also an excellent and quick way to verify the completeness and integrity of the data, because observation of significant data at the edges clearly shows where the acquisition window was not wide enough. This might indicate that, for example, sample charging may have caused data to shift out of the window or that an as-yet-unidentified element is present in the sample's composition, indicating the need for further examination.

1. Select Profile Massage–Montage Setup.

2. Specify a line number and region number.

3. Select Baseline Subtract, and unselect Smooth and Differentiate. (White indicates selected, and magenta indicates unselected.)

4. Press Display.

5. Press the Rotation Active softkey, and adjust the perspective of the montage by changing the values in the % of Skew and % of Scale parameters. The display is updated automatically as the parameters are changed.

6. If desired, press Ctrl-F12 to send the display to the printer.

   If desired, press Ctrl-F11 to copy the displayed graph to the clipboard, activate a word processing program, and paste the graphic into a document.

7. Repeat this process for each line and region.

8. Press Exit to close the Montage menu.

9. Evaluate the integrity of the data.

Section 8:
Perform a Depth Profile and Analyze the Results

Depth profiles provide elemental and chemical information as a function of depth. A depth profile is typically used to determine what layers are present, how thick they are, and what contaminants may be present at layer interfaces. There are three types of sputter depth profiles:

- "Alternating," where sputtering and acquisition do not happen at the same time, and, generally, the sample is tilted;
- "Rotating," also called "Zalar," which is an alternating profile where the sample is rotated during sputtering and not tilted;
- "Continuous," where data acquisition and sputtering occur simultaneously. Continuous depth profiles are not typically performed on the 680 Scanning Auger Nanoprobe, because the Auger electron detector quickly becomes saturated unless a very low sputter rate is used.

This section describes setup, acquisition, and data reduction of alternating and rotating depth profiles.

Before setting up for the depth profile, it is important to obtain as much information as possible about the thickness and composition of the layers to be monitored in the profile.

Acquisition from five or more data points per layer is recommended to obtain acceptable layer definition and depth resolution during the profile.

This procedure assumes that the sample has been mounted, inserted into the vacuum chamber, and imaged, as described in Section 3. The steps are the following:

A. Prepare the ion gun.

B. Prepare the Ion Gun Setup menu.

C. Select the Alternating or Rotating acquisition method.

D. Prepare the Stage Control software.

E. Set up regions in the Profile Setup menu.

F. Set up profile parameters in the Profile Parameters menu.

G. Perform Test Acquires on the regions selected.

H. Register the image.

I. Acquire the profile.

J. Suspend the depth profile.

K. Change the profile's parameters, then resume acquisition.

L. Load and display the depth profile.

M. Create a montage display.

N. Adjust the analysis window boundaries.

O. Get atomic concentration data of the profile.

P. Get AC data from individual cycles.

Q. Create a Time to Depth Conversion.

A. Prepare the ion gun.

Depth profile information is obtained by alternating data acquisition and bombardment of the specimen with inert gas. This involves the ion gun, Ion Gun Control software, the Thermovalve Control, and the Watcher (SVC) components of the system.

At the beginning of this process, all the AVC valve lights except valve 7 (V7) must be red (indicating closed).

1. In the Ion Gun Control software, select View–Extractor Pressure to open the display of the Extractor Pressure window. The Gun State should be Off and Emission Current should be 0.

2. Make sure the turbo pump is on and up to speed, which takes about 5 minutes.

3. Press the Watcher button in the Start taskbar to display the Watcher window, and press the DIFF PUMP ION GUN - ON button.

   The ion gun is being differentially pumped when V4 (the valve between the ion gun and the turbo pump) is shown green and V3 (the valve between the turbo pump and the intro) is shown red.

   NOTE: Valve 4 is interlocked so it will not open if there is insufficient vacuum between the turbo pump and the ion gun. This safety feature ensures that the system will not accidentally be brought up to air.

4. Achieve an Extractor Pressure of 25 mPa, as follows:

   a. Throughout this step, monitor the reading in the Extractor Pressure window, and monitor the Chamber Ion Gauge readout on the Vacuum Gauge Control to ensure that the value stays below 1×10^{-7} torr.

   b. Slowly turn the leak valve on the ion gun manually in a counterclockwise direction to let gas into the system.

   c. The Thermovalve Control regulates the leak valve and is used to maintain constant gas pressure in the ionization chamber, thereby providing consistent and repeatable sputter rates. Since the Thermovalve Control can only *close* the leak valve from its unregulated position, it is necessary to initially open the valve manually to allow more gas flow than desired during ion gun operation.

      When the Extractor Pressure reaches 40 or 50 mPa, stabilize the pressure at 25 mPa, as follows:

      i. On the Thermovalve Control, turn on the power switch. (Nominal settings on this control are typically 5 for RESET TIME, 10 for PROP(ortional) GAIN, and 10 for LIMIT.)

      ii. Push the Set Point switch down.

          After several seconds, the Extractor Pressure will stabilize at the previously stored Set Point pressure.

      iii. If the set point pressure is different from 25 mPa, turn the SET POINT potentiometer clockwise to raise (or counterclockwise to lower) the pressure, again allowing several seconds for the new pressure to stabilize.

      iv. When the Extractor Pressure has stabilized at 25 mPa, proceed to step 5.

5. In the Ion Gun Control software, select a predefined set of ion gun parameters from the Settings drop-down list (or create a new setting by entering the desired parameters, pressing the Tab key to enter each new value, entering a Setting name, and pressing Add). The setting selected should be the setting known to approximate the sputter rate desired. (Refer to Appendix B, Ion Gun Control Software, and the *Model 06-350 Ion Gun Component Manual* for information on ion gun parameters and corresponding sputter rates.)

6. Click on Standby in the Gun State part of the Ion Gun Control window.

   Pressing Standby begins warming up the ion gun, which includes achieving the Emission Current and Grid Supply parameter values displayed.

7. Because the Emission Current and Extractor Pressure values are interrelated and the Emission Current rises after Standby is pressed, the Extractor Pressure changes accordingly. Further adjustment of the SET POINT potentiometer on the Vacuum Gauge Control may be required at this point to ensure that (1) the Chamber Ion Gauge reading stays below 1×10^{-7} torr, and (2) the target Extractor Pressure and Emission Current are achieved and stabilized before proceeding to the next step.

   The Thermovalve Control will automatically adjust the leak valve to maintain the SET POINT pressure for operating during depth profile acquisitions.

8. Position the sample to be at the focal point of the analyzer, which is where the ion gun has been aligned. (Refer to the *Model 06-350 Ion Gun Component Manual* for ion gun alignment procedures.)

NOTE: PC-ACCESS *will control turning the ion gun on and off during the acquisition.*

B. Prepare the Ion Gun Setup menu.

The values entered in the Ion Gun Setup menu in PC-*ACCESS* are saved with the data acquisition file. The values from the Ion Gun Control window must be entered manually in the PC-*ACCESS* menu if the correct values are to be saved in the file. (Note that some of the parameters in the Ion Gun Control window do not have a place in the PC-*ACCESS* menu and cannot be saved with the file.)

1. Enter the ion gun parameters in PC-*ACCESS*'s Ion Gun Setup menu, pressing Enter or clicking the right mouse button after each value is typed.

C. Select the alternating or rotating acquisition method.

The three options available to optimize depth resolution are the following:

- Use a low ion gun beam voltage,
- Use a low "grazing" angle,
- Rotate the sample during sputtering.

Determine whether the depth profile acquisition will be alternating (for tilted samples) and the angle needed or rotating (with 0-degree tilt).

NOTE: Changing the tilt moves the area of interest, so the operator will have to relocate the area of interest and reposition the area at the focal point of the analyzer, which is where the ion gun is aligned. Refer to Section 3, steps I and J, for procedures, or refer to the Stage Control Software Manual *for "Eucentric Tilt" procedures.*

D. Prepare the Stage Control software.

Set the parameters in the Stage Control window, as follows.

- For alternating profiles, click on the Tilt text field, then type the angle to be used. (30 degrees is typical.)

  NOTE: Relocate the area of interest, as described in the previous step.

- For rotating profiles, click on the Tilt text field, then type "0." Then, calibrate the center of rotation, as described in the *Stage Control Software Manual*.

  NOTE: The stage is optimized to acquire Zalar Rotation profiles without any tilt.

E. Set up regions in the Profile Setup menu.

NOTE: Before setting up the depth profile acquisition parameters, it is important to obtain as much information as possible about the thickness and composition of the layers to be monitored in the profile so the depth profile can be set up to include them.

1. Select Setup Acquire–Setup Profile, select New, and press Enter.

2. Set up the depth profile acquisition, as follows:

   NOTE: Always put the most volatile elements first, because they are susceptible to electron beam damage.

   a. Type the name of the region (from Appendix A) to scan, and press Enter. PC-*ACCESS* automatically enters values for that transition into several of its parameter fields. Figure 8-1 shows a sample depth profile setup menu.

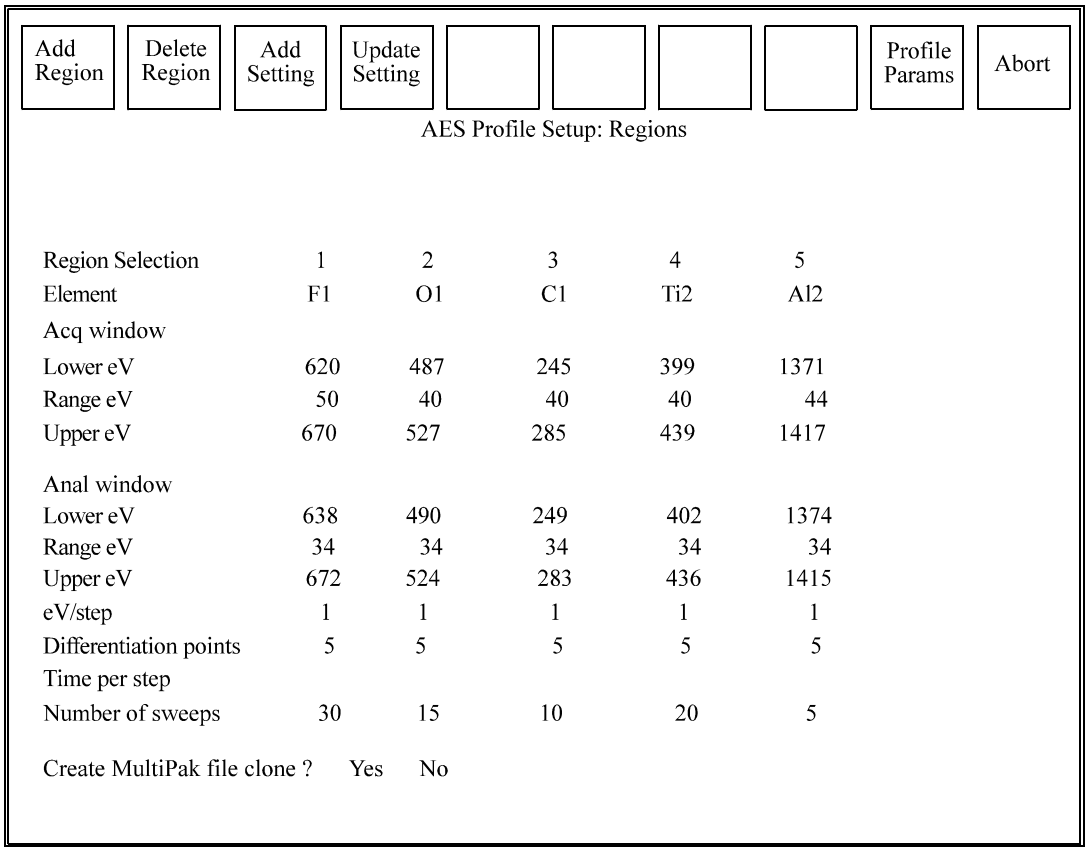

Figure 8-1. Profile Setup Regions Menu with Sample Settings.

*NOTE: PC-*ACCESS *supplies default values for the acquisition parameters. The acquisition window is the energy range over which data will be acquired, and the analysis window is the range used for peak intensity measurements. The limits for the analysis window are usually a few eV inside of the acquisition window limits. To measure peak intensity, PC-*ACCESS *performs a 5-point differentiation on the data and measures the peak-to-peak height.*

b. Specify sweeps and time per step.

   It is useful to define modest parameters to get an idea of acquisition times (as described in Sections 5 and 6).

c. Press the Add Region softkey.

d. Repeat steps a, b, and c until all the regions are entered.

e. Press the Profile Parameters softkey.

F. Set up profile parameters in the Profile Parameters menu.

After setting up for the element region parameters, the sputtering parameters are entered in the Profile Parameters setup menu.

1. Press the Profile Params softkey in the Regions setup menu to open the Profile Parameters setup menu.

2. Select the sputter time. (The desired sputter depth and the sputter rate will determine the required sputter time.)

   NOTE: Different materials have different sputter rates. For example, gold can sputter up to four times faster than Ta_2O_5. *If exact thickness measurements are required, reference materials of known thickness must be sputtered and used as a calibration for the sputter rate, or a profilometer trace must be made to measure the depth of the sputter crater.*

3. In Sputter Mode, select Alternating for alternating profiles, or select Rotating for a rotating profiles.

   *NOTE: Rotating must be selected in order for PC-*ACCESS *to tell the Stage Control software when to start and stop rotation.*

4. Enter a Sputter Interval time, taking into account the sputter rate and the Sputter Time.

   NOTE: For rotating profiles, take into account the rotation speed when defining the Sputter Interval. Typically, the sputter interval is equal to one revolution of the sample. For example, if the sample rotates once per minute, the Sputter Interval would be set to 1 minute. If the sample rotates half a revolution per minute, the Sputter Interval would be defined as 2 minutes.

   For example, if the Ion Gun Control will sputter at 25 Å per minute and a Sputter Interval time of 0.5 minutes was entered, data points will be acquired after every 30 seconds of sputtering, or approximately every 12 Å.

   Below the Sputter Interval field is a readout of the Total Acquisition Time, which includes both the sputter time and the data acquisition time.

5. Specify the Image Registration and Analysis Area parameters (e.g., Selected Areas) as appropriate for the current sample.

6. On some types of samples, a charge can build up on the sample surface during sputtering, causing the image to shift. If there is a delay after sputtering, the charge is given a chance to drain away before the analysis begins. Usually, a delay after sputtering is not required for conductive samples. Enter the appropriate value in the Delay After Sputter field.

7. For rotating depth profiles, select Yes or No in the Rotate Home After Sputter field. Yes is the recommended selection.

G. Perform Test Acquires on the transitions selected.

Test Acquire provides an opportunity to determine whether the acquisition windows set up for each of the elements are appropriate.

NOTE: It is important to make each window wide enough to include the entire Auger peak. Since Test Acquires can be performed only for the elements on the surface, the operator needs to consider the windows of the buried elements, especially on samples where there may be charging layers below the surface. The windows need to be wide enough so that, if the sample charges and the Auger peaks shift upward in energy, the peaks will not move outside the acquisition windows.

1. Press the Test Acquire softkey in the Profile Parameters setup menu to open the Test Acquisition menu.

2. Adjust the menu parameters as needed:

    a. So the analysis area for the profile does not suffer any beam damage, use the Point Coordinates parameter in the Test Acquire menu and the arrow keys to move the incident electron beam slightly aside from where the actual acquisition is to take place.

    b. Adjust the number of sweeps parameter, if desired.

    c. Select Point in the Area Type parameter.

    d. Set the Display Mode parameter to dN(E), because this is how the actual data will be acquired.

3. Press the Test Acquire softkey.

    Differentiated data for the first region is displayed on the right side of the screen as it is acquired.

4. Press the Abort Acq softkey when enough data has been acquired.

5. Press the Regions softkey to redisplay the Regions setup menu and make the desired changes to the parameters to be used during actual acquisition.

6. Press the Profile Params softkey in the Regions menu, then the Test Acquire softkey in the Profile Parameters menu.

7. Press the Next Element softkey in the Test Acquisition menu.

8. Repeat steps 2 through 8 until all the regions to be acquired have been examined and their parameters adjusted as needed.

H. Register the image.

If Image Registration was selected, the last step before beginning the depth profile is to make sure the image is still aligned with the registration marks drawn previously on the DSM. Once these registrations are lined up, acquisition may begin.

I. Acquire the profile.

1. Press Acquire to begin the depth profile acquisition after the regions and acquisitions parameters have been defined.

   To determine a base level for each of the elements being monitored, two presputter data cycles are acquired before sputtering begins. These are displayed on the left side of the depth profile graph. The y axis is an arbitrary peak-to-peak height intensity scale, and the x axis is sputter time in minutes. The lines on the graph will appear after the second cycle.

2. To look at the individual windows as they are being acquired, press the N(E) or dN(E) Display softkey. The white cursors inside the window being displayed represent the analysis window. To return to the profile display, press the Profile Display softkey.

3. The profile can be stopped at any time by pressing the CYCLE STOP softkey. The profile acquisition will stop at the end of the cycle currently being acquired.

   NOTE: At any time during the depth profile, you can change the sputter rate by changing the relative raster sizes on the Ion Gun Control or by changing the ion current. It is also possible to change the data intensity in depth by changing the sputter interval.

   For most profiles, start out at a slow sputter rate or a higher data density (smaller sputter interval). If there are thicker layers underneath the

surface contamination, increase the sputter rate or increase the sputter interval.

When acquisition is complete, the screen is blanked.

J. Suspend the depth profile.

It is possible to suspend the depth profile, acquire a survey scan, multiplex, map or line scan, and then resume the depth profile. This may be useful to (1) determine if any new elements are present, or (2) fully characterize an interface of buried structure that is revealed during the profile.

As an example, the Suspend Acquire softkey was pressed during a depth profile when the oxygen signal had dropped off to about half its maximum intensity, and a survey scan was acquired. The results are shown in Figure 8-2.

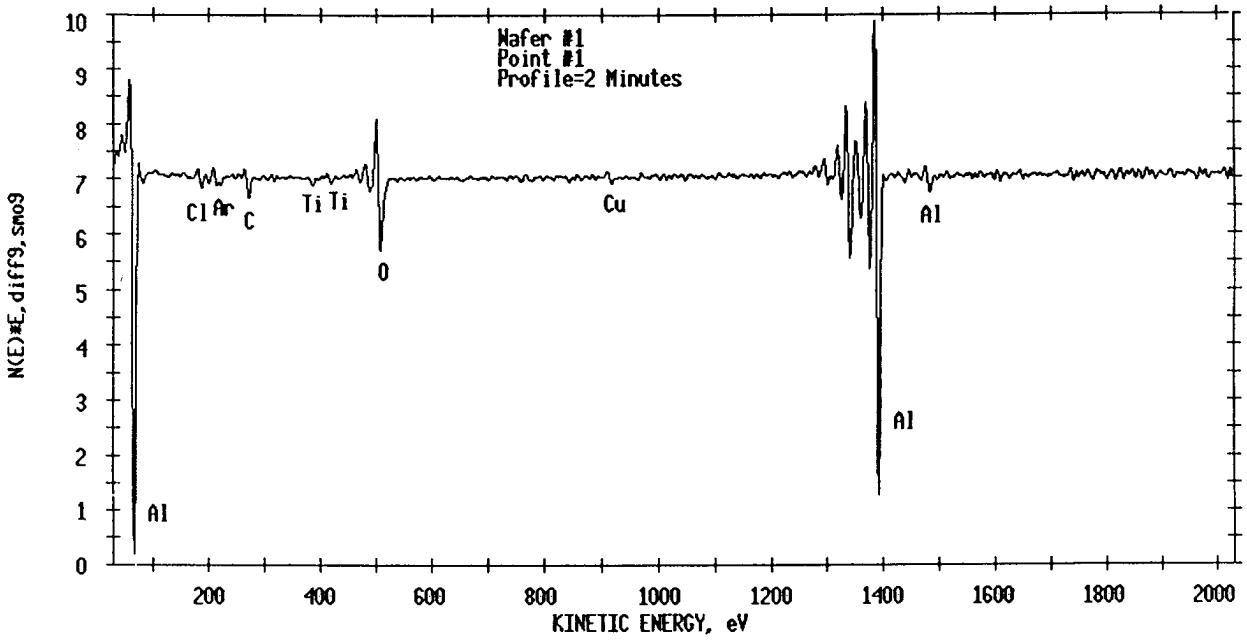

Figure 8-2. *Two Minutes into an Auger Depth Profile, acquisition was suspended and this survey scan was taken.*

K. Change the profile's parameters, then resume acquisition.

Another function available when a depth profile is suspended (or after a CYCLE STOP) is Change Profile. This feature allows the operator to add or remove elements and change certain acquisition parameters.

1. Select Setup Acquire–Change Profile. The file name of the currently suspended acquisition will appear.

2. To access the parameters for the current acquisition file, press Enter or click the right mouse button. The Previously Defined Regions setup menu will appear.

3. Set any previously defined region(s) to On or Off to include or exclude that region during the remainder of the profile. It is also possible to change the lower limit of the acquisition window, both the lower limit and range of the analysis window, and the number of sweeps for each of the elements.

4. Press the New Regions softkey to add new regions for acquisition during the remainder of the depth profile.

5. Press the Profile Parameters softkey to see the file name of the currently suspended depth profile, the sputter time set up for the acquisition, the elapsed sputter time, and how many cycles have been acquired. At this point, the operator may change the total Sputter Time, Sputter Interval, Sputter Mode, Image Registration, and Delay After Sputtering parameters. The Test Acquire softkey is also available to check the acquisition and analysis windows and revise them, if necessary.

6. Press the Resume Acquire softkey.

L. Load and display the depth profile.

After acquisition, load and display the depth profile.

1. Select Acquire–Acq Display.

2. Specify the display parameters:

   a. Click on the file name.

   b. Highlight Profile on the second line. (To display individual regions, highlight Region instead.)

   c. Highlight "All" to display all of the regions. (When multiple areas and regions are acquired, these numbers can be changed.)

d. Choose minutes for Display Units.

e. Press Display. The derivative peak height data is displayed.

M. Create a montage display.

The montage display shows all the cycles' spectra for each region on one graph. This can show, among other things, whether any peak shifted out of the acquisition window due to chemical shifts or charging effects and clearly shows the relative shapes of peaks, which is an easy way to recognize chemical state differences. The display can be shown at different angles or from the end of the profile to the beginning, if desired.

NOTE: PHI MultiPak is the preferred software for data reduction.

1. Select Profile Massage–Montage Setup.

2. Select the desired parameter settings, as follows:

   a. Highlight the desired Area Number, then press Enter. Repeat for the Region Number parameter.

   b. On the Display Every Nth Cycle line, enter the desired Start and End Cycle numbers. (When 1 is entered, every cycle would be displayed when Display is pressed. If 2 is entered, every other cycle would be displayed.) Press Enter.

   c. Enter values for Percentage of Skew and Percentage of Scale.

      Percentage of Skew determines whether the display of the cycles is skewed positively or negatively from vertical. This has the effect of changing the apparent *azimuthal* orientation. Percentage of Scale dictates the height of the maximum intensity spectrum from each region, relative to the plot area. This has the effect of changing the apparent *elevation* of the view.

   d. Highlight Yes or No in the Grid parameter. Selecting Yes will display a constant-energy grid that connects each of the displayed cycles.

   e. The Massage Before Display parameter includes the options Smooth, Baseline Subtract and Differentiate. Any combination of these can be selected for the display. Those appearing in magenta are turned off. Those appearing in white are selected. Use the Select Massage and Unselect Massage softkeys for selection and deselection.

For example, to display smoothed N(E) data, highlight Smooth and press Select Massage.

NOTE: Ensure that Differentiate is not selected.

3. Press Display.

   NOTE: The montage display can be printed using the Print Graphics command.

4. Repeat steps 1 through 3 to display each region, if desired.

5. Press the Exit softkey to close the Montage menu.

Example

Figure 8-3 shows a sample montage display of the aluminum peak at every cycle in a profile. The parameters set were: Start Cycle = 1, End Cycle = 18, Region Number = 5 (for the aluminum region), and Skew = –50%.

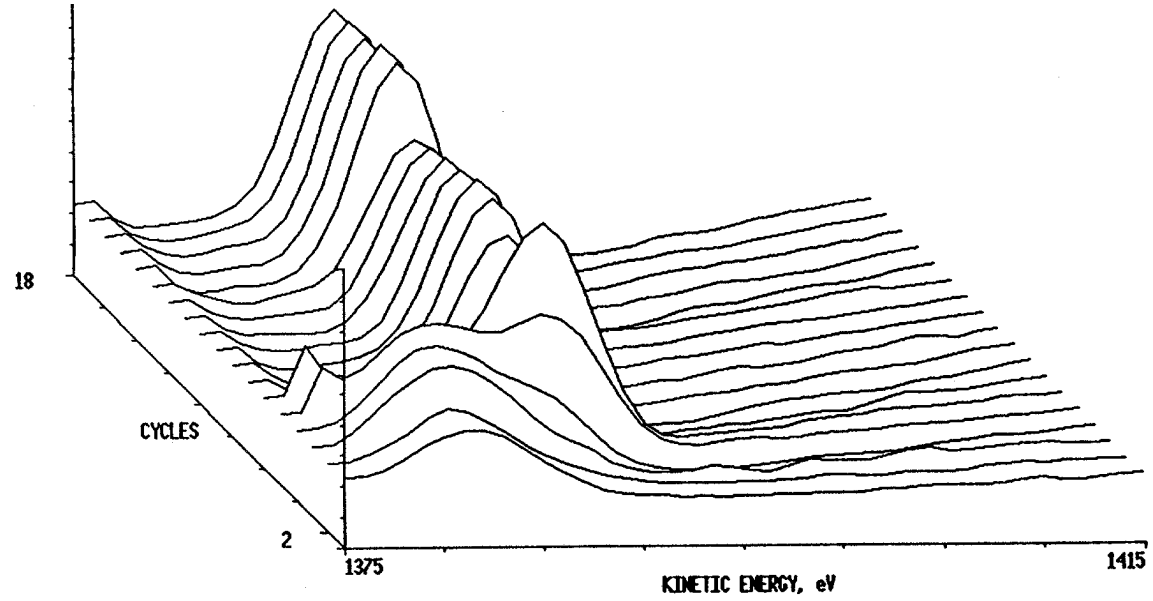

Figure 8-3. Montage Display of the Al Region in a Depth Profile. The y axis is cycle number (1 through 18), the x axis is kinetic energy (1375 through 1415), and the z axis is relative intensity.

Example

Figure 8-4 uses a specific data file to show how displaying overlays of different cycles of the same element can be informative. First, Cycle 1 of Region 5 (oxygen) was specified and the Display softkey pressed. Then, Cycle 18 of Region 5 was added by pressing the Display Add FULL softkey.

This shows the data in the aluminum analysis window at Cycle 1 before any sputtering occurred, plus the aluminum analysis window at Cycle 18 at the end of the profile acquisition. The energy shift indicates that Cycle 1 detected oxidized aluminum and Cycle 18, elemental aluminum.

NOTE: PHI MultiPak software is preferred for separation of profiles into different chemical states.

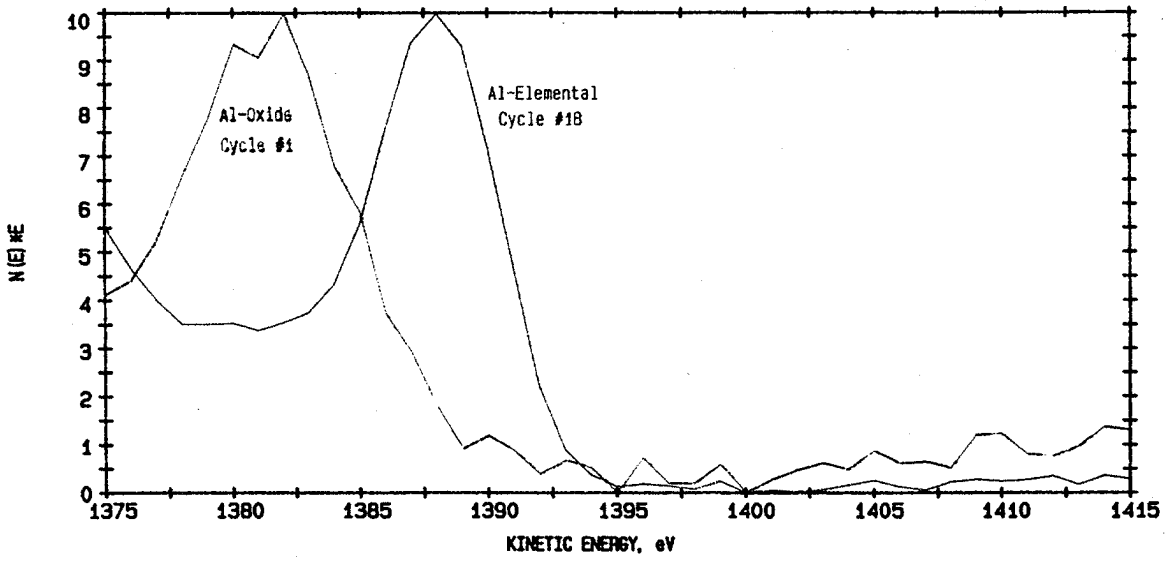

Figure 8-4. *Comparison of Al Windows at Different Cycles during the Depth Profile. The display contrasts the kinetic energy shift due to different chemical states of Al.*

N. Adjust the analysis window boundaries.

Having determined that all of the peaks acquired fell within the acquisition window, the limits of the *analysis* windows may need to be adjusted to include the positive and negative excursions of the appropriate dN(E) Auger peaks and to exclude neighboring peaks from other elements.

For example, if a sample has a partial overlap of oxygen and chromium, these two elements would be set up with very wide *acquisition* windows. After data acquisition, it might be appropriate to narrow the *analysis* windows (or even to divide a single acquisition window into two analysis windows) for measurements of the two elements.

NOTE: PHI MultiPak is the preferred software for data reduction.

1. Select Profile Massage–Display.

2. Select Region as the Display mode. Choose the region number and a cycle in the profile where the desired element exists. Display that spectrum.

   It may also be useful to display additional cycles of that element, using the Display Add FULL or Display Add AUTO softkeys, to detect possible energy shifts.

3. Select Profile Massage–Diff Setup to differentiate the spectra. Enter a 5-point differentiate, and apply it to All.

4. Once representative cycles have been displayed, select Profile Massage–PeakRedefine, then select Window Define.

5. The Peak Redefine display shows the lower and upper limits for the analysis window. The correct positive and negative excursions for each peak need to be within those limits. Highlight the desired parameter(s) and use the arrow keys to adjust the boundaries as needed.

6. Divide a single acquisition window into two (or more) analysis windows as follows:

   a. Select Add Window.

   b. Enter the region name, and change the lower limit and range, if desired.

   c. Press Apply, then Store to Table, then Redefine Datafile.

   d. Press Exit.

7. Repeat steps 1 through 6 for every region.

O. Get atomic concentration data for the profile.

AC calculations are performed on the data specified by the parameters in the Atomic Concentration menu, which is opened by selecting ACMassage–AC Setup. The softkeys in this menu also are used to send the AC data to a Notepad file for display, copying, or printing.

1. Select Profile Massage–Display. Select Profile Mode and All Regions, and press the Display softkey. Then, press Exit.

2. If there are some regions to be excluded from the AC calculation, proceed as follows:

   a. Select Profile Massage–Expand.

   b. Press Pick New Curve, then press Next Curve or Previous Curve to highlight (in white) the curve to be removed. The area and region of the highlighted curve is displayed at the bottom of the PC-*ACCESS* window.

   c. Press Remove.

   d. Repeat steps b and c until the only region profiles displayed are the ones to be included in the AC calculation.

   e. Press Pick, then press Exit.

3. Select Profile Massage–AC Profile to calculate the atomic concentrations.

   The y axis is now converted to an atomic concentration percentage scale from 0% to 100%.

4. For color output of the AC profile, proceed as follows:

   a. Press Output Control–Print Setup.

   b. Select “deskjet.”

   c. Select “Properties,” then “Landscape” in the Orientation field. Press OK, then press OK again.

   d. Select Output Control–Print Graphics.

      NOTE: Unless the Print Setup is changed again, future printouts will go to the DeskJet printer.

P. Get AC data for individual cycles.

This subsection describes how to get atomic concentration values for individual cycles of the profile.

1. Select AC Massage–AC Setup. The regions of the profile will automatically be entered.

2. Change the cycle number as appropriate and press Enter.

3. Select "No" in Massaged Data, and select dN(E) Display. (A 5-point differentiation is automatically performed on the data as part of the atomic concentration calculation.)

4. Click on a specific region name, and adjust the analysis region boundaries as necessary (as described in Section 4H, step 8).

5. Press the Include Region or Exclude Region softkeys as appropriate.

6. Repeat steps 4 and 5 for all regions.

7. Select Profile Massage–AC Profile to calculate the atomic concentrations for the selected curves, regions, and cycles.

8. Press the Table to Notepad softkey. The results are displayed in the Notepad window.

9. Select Print from the File menu in the Notepad window to obtain a printout of the atomic concentration table.

10. Select File–Exit to close the Notepad application.

11. Press Exit to close the Atomic Concentration menu.

Q. Create a time-to-depth conversion.

Another way to display the depth profile is to change the x axis from time units to depth units.

1. Press Profile Massage–Display. Make sure that Profile Mode and All Regions are highlighted. Leave Display Units in minutes initially and press Display to show the peak-to-peak-height intensity display of the depth profile. Exit the Display menu.

2. Select Profile Massage–Time To Depth.

3. Choose Initial in User Settings and press Enter. Page one of the menu is displayed.

4. Select Angstroms in the Units for Depth field.

5. Press Define Layers. In the menu, specify an end time and a Rate in depth per minute (D/min) for each layer, then press Enter. A corresponding Thickness will be displayed. (You can, instead, specify a Thickness, causing a corresponding Rate and Depth for each layer to be entered automatically.)

   NOTE: As many layers can be defined for a specific profile as are necessary to correctly characterize the data in terms of depth.

   NOTE: To save this conversion, press the Add Setting softkey and type a file name. Later, when the Time to Depth function is activated again and the setting's file name is selected, the computer will automatically set up a conversion using the same parameters.

6. Press Display Depth.

7. Press Exit.

Example

The sputter rate used to acquire the example profile was 25 Å per minute calibrated with Ta_2O_5 as a reference standard. Because a single sputter rate was used for this profile, one layer was defined with an end time of 8 minutes and a sputter rate of 25 Å per minute, which resulted in a total thickness of 200 Å for the profile. To display this in depth, the Display Depth softkey was pressed. As shown in Figure 8-5, the y axis was displayed in peak-to-peak-height intensity, and the x axis was displayed in angstroms for depth.

The example profiles were annotated and printed using the Graph Annotate and Print Graphics functions. The result is shown in Figure 8-6.

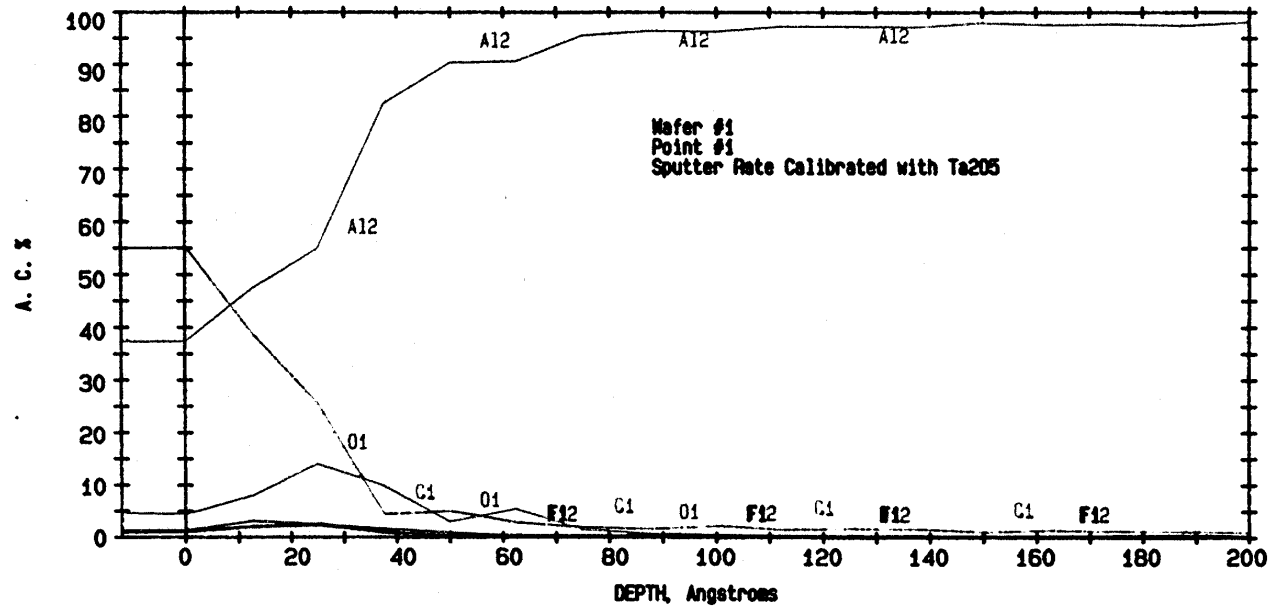

Figure 8-5. Atomic Concentration Percentage vs. Depth in Angstroms of the Depth Profile.

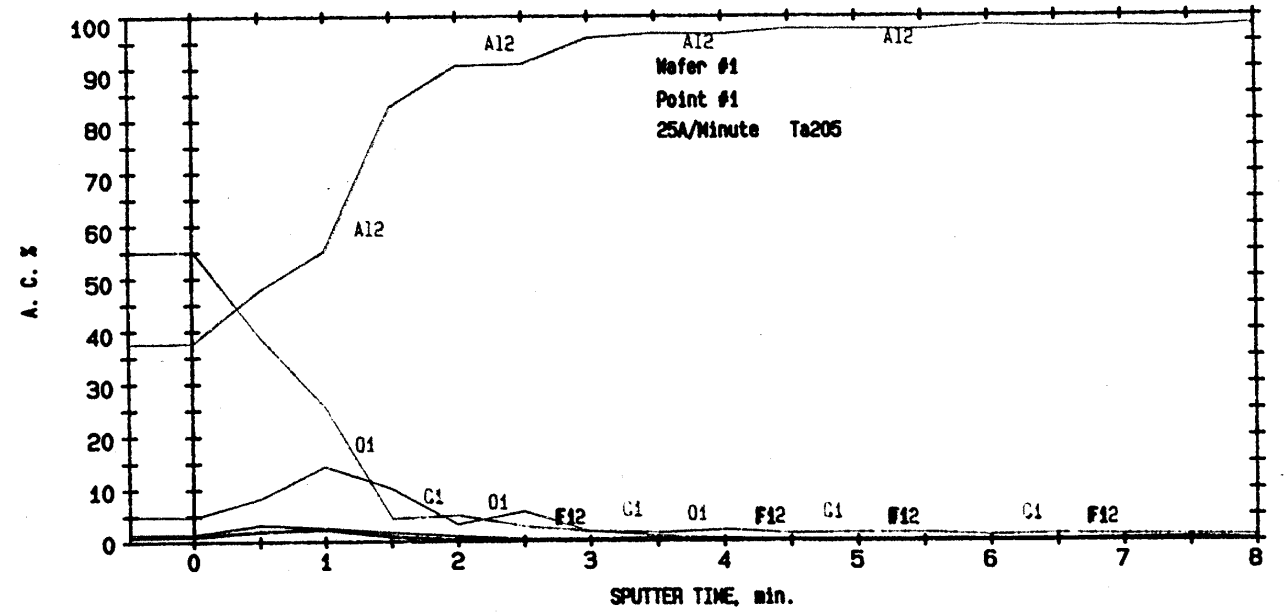

Figure 8-6. Annotated Atomic Concentration Display of the Auger Depth Profile vs. Sputter Time in Minutes.

Section 9:
Using AutoCom

This section describes the AutoCom feature and how to use it. Examples are provided.

NOTE: Refer to the AES PC-*ACCESS* Software Manual *for details about specific fields and softkeys of AutoCom menus (Section 3, AutoCom, Display, Load, Expand, and Sample Setup).*

AutoCom Menus

The AutoCom command (Automated Command Sequencing) is a software feature that makes routine or lengthy sample analysis and data massage possible without an operator present. AutoCom can be used for data acquisition and data reduction.

The AutoCom command is located on the Automation command bank in the first home bank of the PHI setting. It is accessed by highlighting it and clicking with the left mouse button or by pressing the F1 key when the Automation command bank is selected. AutoCom has three primary menus: Defined Sequences menu, Edit Sequence menu, and Runtime Report.

The Defined Sequences menu lists sequence names that have been already defined. Use the arrow keys or left mouse button to select the desired sequence name, or press Add Sequence, then New Sequence to define a new sequence. The Defined Sequences menu is also used to delete, edit, and execute sequences.

The Edit Sequence menu is displayed by pressing the Edit Sequence softkey from the Defined Sequences menu. This menu is used to create new sequences and change existing sequences.

Runtime Report provides a synopsis of what happened during actual processing of the AutoCom sequence. Each function in the sequence is listed along with a brief message telling whether the function was completed or some problem occurred during processing.

Using the Edit Sequence menu.

Sequences are built and edited using the Edit Sequence menu. When this menu is displayed, the lower row of softkeys displayed is the same as the first home bank of the currently selected interface setting. Commands are added to the sequence by selecting command banks from the lower row, then selecting commands from the command bank displayed on the left side of the PC-*ACCESS* window. (Click with the left mouse button to select the command or hold down the Shift key and press the function key corresponding to that command.)

Before selecting the command to be added, highlight (using the arrow keys or left mouse button) the position where you want the command added, and press the desired softkey (Insert Before or Insert After). (The state of the insert mode is shown in the lower right corner of the PC-*ACCESS* window.) The command selected next will be positioned accordingly.

NOTE: Some of the commands may be displayed in red, indicating that the command is not available in AutoCom. Refer to Section 3 in the Software Manual *for information about the use of specific commands in AutoCom.*

NOTE: If parts of the sequence cannot be seen on the screen, use the arrow keys to scroll through the sequence.

Some commands have parameters that can be edited. To edit the parameter values in the menu of the selected command, highlight the command, and press the Edit Parms softkey.

NOTE: Some parameters in the menu(s) associated with individual commands can be defined before, during, or after defining the AutoCom sequence. Before executing a sequence, use the Open Sequence and Edit Parms softkeys from the Edit Sequences menu to verify that all the settings are appropriate for the acquisition to be performed.

Adding loops to an AutoCom sequence.

Looping within an AutoCom sequence can be achieved using the Display, Load, and/or Sample Setup commands. These commands have different menus when opened from the AutoCom function. When one of these commands is selected when defining an AutoCom sequence, press Edit Parms to show its AutoCom menu.

Selecting Yes in the Loop Control field will add a Loop command to the sequence. A Loop must be terminated by one of the Loop End commands. Pressing the Loop Ends softkey in the Edit Sequence menu, then pressing the appropriate softkey adds a Loop End command to a sequence. (Load Loop End

terminates a Load loop; Sample Loop End terminates a Sample loop; and Region Loop End, Cycle Loop End, or Area Loop End terminates a Display loop.)

Selecting No in the Loop Control field will add a Fixed or Next command to the sequence. In this case, only a Load Next command will need to be terminated by a Load Loop End.

Embedding sequences into other sequences.

Existing sequences can be embedded into other sequences by pressing the Insert Sequence softkey, then pressing the Sequence Function or Sequence Name softkey in the Edit Sequences menu. The first embeds the sequence of commands and allows *different* parameters to be used in different sequences. The other (Sequence Name) embeds the sequence name and causes the *current* parameter settings to be used in all instances of that sequence.

NOTE: A sequence that is embedded in other sequences cannot be deleted, and a sequence cannot be embedded within itself.

Running the AutoCom sequence.

After an AutoCom sequence has been defined, enter the Defined Sequences menu and select either the Partial Process or Process Sequence softkey to run the sequence. The commands will be performed in the order in which they are listed.

An AutoCom sequence can be interrupted at any time in any of the following ways:

- Function Stop, which stops execution of the sequence after it completes the function currently running.

- Abort, which stops execution immediately. If an acquisition is in progress, no data is saved.

NOTE: If you abort a sequence while it's processing and a sample loop function is being executed, it may be necessary to go into the Sample Setup menu and reselect the desired samples before processing the sequence again.

- When acquisition is being performed as part of an AutoCom sequence, the same acquisition control is available as during a normal acquisition: Cycle Stop, Region Stop, and Frame Stop. When one of these commands is used during an AutoCom sequence and the acquisition is resumed, the sequence will continue execution with the next command.

Setting up sequences.

To define an AutoCom sequence, select Automation–AutoCom from the home bank. The AutoCom Defined Sequences menu is displayed. Press Add Sequence. Type in a new name, press Enter, and press Accept Name.

The following tables detail how to build some sample AutoCom Sequences. Each sequence can be used as a building block to be used in combination with others to define additional sequences.

Table 9-1 describes building an AutoCom sequence named "acq_sur" that will acquire, load, display, differentiate, and print a survey scan. Table 9-2 describes building an AutoCom sequence named "acq_mult_print" that will display and print all regions of a file of multiplex data and then perform an atomic concentration (AC) routine using all the regions in the file.

Table 9-3 describes building an AutoCom sequence named "acq_mult_ac" that will acquire multiplex data for all selected regions, use the acq_mult_print AutoCom sequence to print all regions and generate AC data using all regions, and display the AC results. Clear AC Summary is inserted early in the sequence to clear out the previous AC information before the new summary table is prepared.

Table 9-1. Building an Example Sequence ("acqsur") Using AutoCom.

AutoCom Sequence	Action Performed To Insert the Command into the Sequence
Setup Survey	Select Setup Acquire from the lower home bank display, then select Setup Survey from the command bank displayed on the left side of the PC-*ACCESS* window. Highlight Setup Survey on the AutoCom sequence list, press Edit Parms, and select Previous in the Settings parameter.
Acquire Survey	Select Acquire from the lower home bank display, then select Acquire Survey from the command bank displayed on the left side of the PC-*ACCESS* window.
Load Acquire	Select Load Display from the lower home bank display, then select Load from the command bank displayed on the left side of the PC-*ACCESS* window.
Display	Select Display from the command bank displayed on the left side of the PC-*ACCESS* window.
Differentiate	Select Next Bank from the lower home bank display. Select Direct Massage #2 from the lower home bank display, then select Differentiate from the command bank displayed on the left side of the PC-*ACCESS* window.
Print Graphics	Select Spectral Massage from the lower home bank display, then select Print Graphics from the command bank displayed on the left side of the PC-*ACCESS* window.

Table 9-2. Building an Example Sequence ("acq_mult_print") Using AutoCom.

AutoCom Sequence	Action Performed To Insert the Command into the Sequence
Display Loop	Select Load Display from the lower home bank display, then select Display from the command bank displayed on the left side of the PC-*ACCESS* window. Press the Edit Parms softkey in the AutoCom menu. Highlight Loop Control and select Yes. Highlight "Profile" mode, and select a region. Press Exit in the Display menu.
Print Graphics	Select Next Bank from the lower home bank display. Select Spectral Massage from the lower home bank display, then select Print Graphics from the command bank displayed on the left side of the PC-*ACCESS* window.
Region Loop End	Press the Loop Ends softkey in the AutoCom Edit Sequences menu. Press Region Loop End, then press Done.
AC	Select AC Massage from the lower home bank display, then select AC from the command bank displayed on the left side of the PC-*ACCESS* window.

Table 9-3. Building an Example Sequence ("acq_mult_ac") Using AutoCom.

AutoCom Sequence	Action Performed To Insert the Command into the Sequence
Setup Mult	Select Setup Acquire from the lower home bank display, then select Setup Mult from the command bank displayed on the left side of the PC-*ACCESS* window. Highlight Setup Mult on the AutoCom sequence list, press Edit Parms, and select Previous in the Settings parameter.
AC Summary Clear	Select Next Bank from the lower home bank display. Select AC Massage from the lower home bank display, then select AC Summary Clear from the command bank displayed on the left side of the PC-*ACCESS* window.
Sample Loop	Select Next Bank from the lower home bank display. Select Sample Setup from the lower home bank display, then select Sample Setup from the command bank displayed on the left side of the PC-*ACCESS* window. Press the Edit Parms softkey in the AutoCom menu. Highlight Loop Control and select Yes. Press Exit in the Sample Setup menu.
Acquire Mult	Select Acquire from the lower home bank display, then select Acquire Mult from the command bank displayed on the left side of the PC-*ACCESS* window.
Load Acquire	Select Load Display from the lower home bank display, then select Load from the command bank displayed on the left side of the PC-*ACCESS* window.
acq_mult_print	Press the Insert Sequence softkey in the AutoCom Edit Sequences menu. Highlight acq_mult_print, then press the Sequence Function (or Sequence Name) softkey.
Sample Loop End	Press the Loop Ends softkey in the AutoCom Edit Sequences menu. Press Sample Loop End, then press Done.
AC Summary List	Select Next Bank from the lower home bank display. Select AC Massage from the lower home bank display, then select AC Summary List from the command bank displayed on the left side of the PC-*ACCESS* window.

Appendix A:
Stage Control Software

This appendix describes the Windows-based stage control software. The operator controls the stage using the keyboard and mouse and/or joystick with the software interface shown in Figure A-1. PC-*ACCESS* acts as the behind-the-

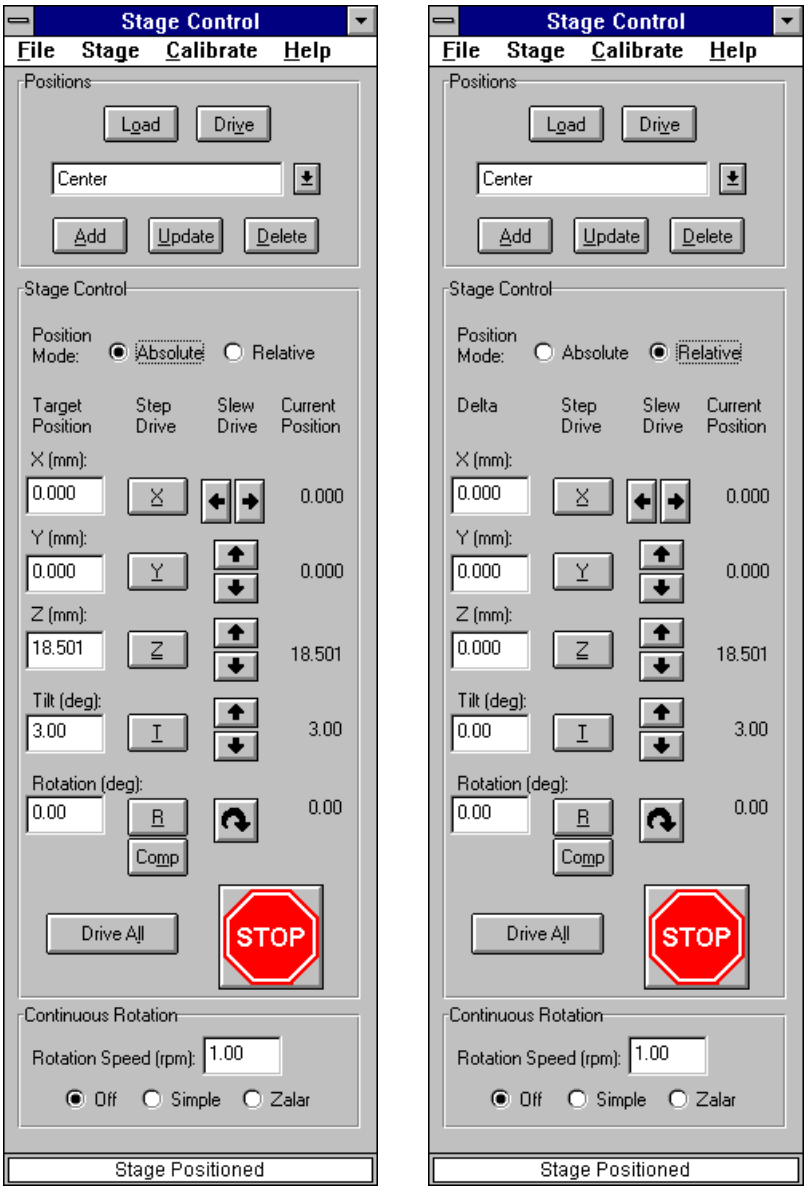

Figure A-1. *Interface of the Stage Control Software, Shown in Absolute (left) and Relative (right) Modes.*

scenes interface to the Stage Control software, which controls the stage hardware, and can directly control some stage control software activity.

The interface has four main menus—File, Stage, Calibrate, and Help—and four main areas of the window—Positions, Stage Control, Continuous Rotation, and status line. The main menu functions, shown below, and each feature of the interface are discussed under main headings (which are sorted alphabetically) in the remainder of this section.

File	**Stage**	**Calibrate**	**Help**
Exit	Initialize	Rotation Center	About Stage Control
	Properties	Eucentric Tilt	Help Topics

The software opens and closes automatically with opening and closing the PC-*ACCESS* software.

Getting Started

The stage is used to locate one or many features of interest at the center of the video image on the system monitor, save the features' locations, and drive to those locations, called Position "settings," on demand. The stage is installed with certain Position settings (sets of coordinates), and the operator can easily define and save additional settings for precise repeatability and operational flexibility.

When used in conjunction with data acquisition using PC-*ACCESS*, the stage can drive to any or all of the saved locations. In this way, acquisition from several positions can be performed using a single Acquire command. Even compucentric Zalar Rotation™ depth profile acquisitions can be performed automatically at several locations as part of a single Acquire, because the stage can also rotate the sample continuously during ion sputtering.

An almost unlimited number of Position settings can be stored in the Stage Control software and/or in PC-*ACCESS*.

In its simplest form, the operator can drive the stage to position a single feature of interest at the center of the video image on the system monitor, then use PC-*ACCESS* to acquire data from that location. In this scenario, no coordinates need to be saved by the operator.

PC-ACCESS's Control of the Stage Control Software

PC-*ACCESS* can control stage movement during data acquisition from multiple positions using the parameters entered by the operator in the Sample Setup and Setup Profile menus.

The operator "drives the stage" to position a feature of interest at the center of the video image on the system monitor. Then, in PC-*ACCESS*, the operator opens the Sample Setup menu and, if the Multiple Position menu is not already displayed, presses the Variable Point softkey. The stage parameters are displayed at the bottom of the PC-*ACCESS* window. The operator presses the Store Position softkey, which stores the coordinates by a name the operator assigns. The operator repeats the positioning of the stage and saving of coordinates until all the positions have been stored. (If desired, the Move to Position may then be used to drive to any of the stored positions.)

When ready to acquire data, the operator uses the "Sel" column in the Sample Setup menu to specify which positions are to be included in the Acquire. Then, the appropriate Setup Acquire menu is used to establish the other acquisition parameters and the Acquire softkey is pressed to begin acquisition.

When setting up for depth profiling, select "Rotating" for Sputter Mode and select "Yes" for Rotate Home After Sputter. These selections allow PC-*ACCESS* to (1) turn stage rotation on during ion sputtering and off during acquisition, and (2) return the stage to the starting rotation coordinate after rotation. *NOTE: Zero tilt of the sample is recommended with rotation when keeping the location at the focal point of the analyzer is important (such as when acquiring data).*

Moving the Stage

The operator may move the stage in any of the following ways:

- Using the **Step Drive** button associated with the X, Y, Z, and Tilt axes moves the stage either (1) to the coordinate specified in that axis' text field, when Absolute is selected for Position Mode, or (2) in the increment specified in that axis' text field, when Relative is selected for Position Mode.

  (1) to the location of that degree on the stage, when Absolute is selected for Position Mode, or (2) the number of degrees (positive or negative) specified in that axis' text field, when Relative is selected for Position Mode.

  The Rotation Step Drive button labeled "Comp" starts compucentric rotation, which sequentially moves the rotation axis, then the x axis, then the y axis. This motion places the point of rotation at the same (or roughly the same) location on the video image as it was before compucentric rotation began.

- Using one of the **Slew Drive** buttons associated with the X, Y, Z, and Tilt axes "slews" (continuously moves) the stage along the axis associated with that button—without regard to values in the axes' text fields.

  The only Slew Drive button associated with the Rotation axis starts continuous, clockwise (positive) rotation around the stage's rotation center. (There is no compucentric-rotation Slew Drive button.)

- Using the **Drive All** button moves all five axes to achieve the values in the axes' text fields.
- Selecting a position setting in the Positions list box, then pressing the **Drive** button.
- Using the **joystick**. When the joystick is tilted, the stage starts moving in that axis direction. When the joystick is released, the stage slows, then stops moving. The greater the angle of the joystick from its upright position, the faster the stage movement. With the Enable Rotation button turned on in the Stage Properties dialog box, turning the joystick's knob rotates the stage clockwise.
- Using the **arrow keys** on the keyboard (or on the numeric keypad when the Num Lock key is off). When the operator clicks in any of the axis text fields, some subset of the arrow keys (illustrated in Figure A-2) becomes enabled for stage movement.
- Using **PC-*ACCESS***, as described in the preceding subsection.

See Arrow Keys, Drive, Drive All, Slew Drive, and Step Drive later in this section for more detail.

NOTE: Regardless of operator action, stage movement stops when a Z- or Tilt-axis limit (defined in Stage Properties) is reached.

NOTE: Whenever rotation is continuous, rotation is clockwise. Rotation is continuous when slewing or when the Simple or Zalar button is on. Slewing occurs when (1) a Slew Drive button is pressed, (2) the joystick is used, or (3) an arrow key on the keyboard is held down.

Using the Joystick

The joystick for the stage can also be used to change the stage's x, y, and rotation axes. The ALL LOCK, Y LOCK, and X LOCK buttons on the joystick's platform, shown in Figure A-3, are used to prevent inadvertent moves in the y (Y LOCK) or x (X LOCK) direction or any movement of the stage (ALL LOCK) by the joystick. Rotation using the joystick, which is done by rotating the joystick's knob, is also enabled or disabled using the Enable Rotation button in the Stage Properties dialog box.

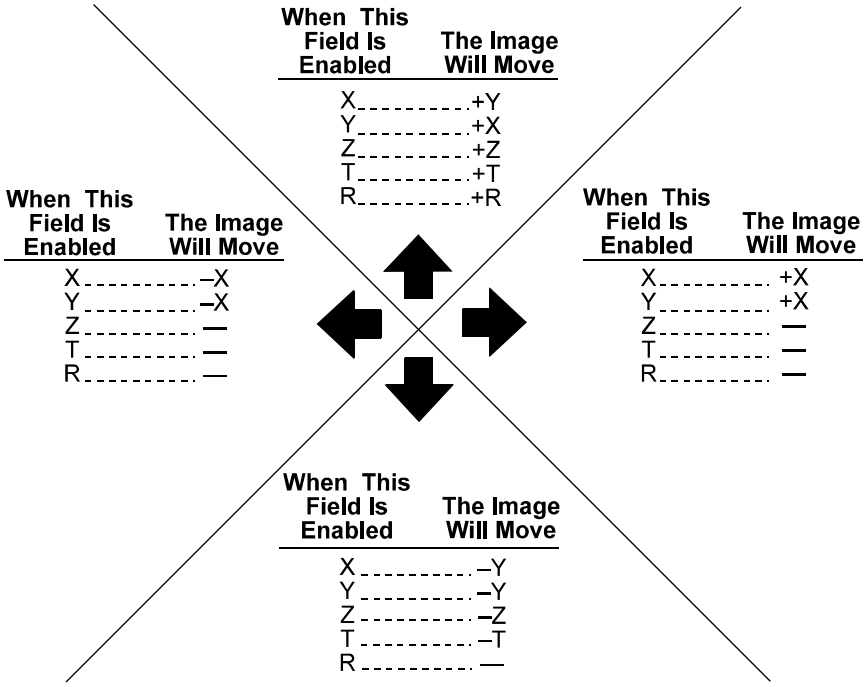

Figure A-2. *Image Movements with Keyboard Arrow Keys (or the arrow keys on the numeric keypad when the Num Lock key is off).*

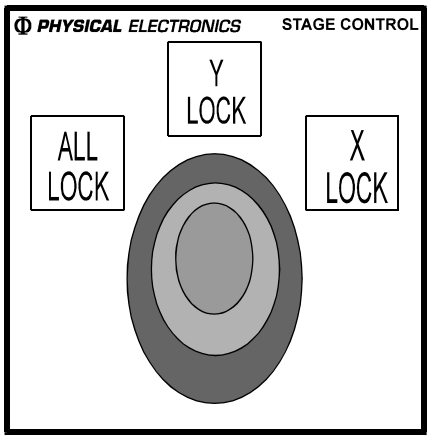

Figure A-3. *Illustration of the Stage's Joystick.*

Motion when using the joystick is called "slewing," which is a continuous series of incremental moves. The size of these increments depends on the angle of the joystick, so that the farther the joystick is pushed away from vertical, the faster the stage moves.

The Two Motor Groups

Axis movements are divided into two motor groups due to a limitation of the motor hardware. Only one group can move at any time. X, Y, Rotate are in one group. Z and Tilt are in the other group.

While any of the axes in one group are moving, the Stage Control window becomes inactive except for the Target Position/Delta fields in that same group and the Stop button.

About Stage Control

Selecting the function called About Stage Control from the Help menu displays the version of the Stage Control software.

Add

A new setting that uses the current values in the Target Position fields is created by typing a name in the Position list box and pressing the Add button.

See also Positions.

Arrow Keys

The arrow keys on the keyboard (or on the numeric keypad when the Num Lock key is off) may be used to move the stage. When the operator clicks in any of the five axis text fields, some subset of the arrow keys (shown in Figure A-2) becomes enabled for stage movement.

A single press of an active arrow key causes one Step Drive move. Holding down that arrow key causes a single Step Drive move followed by a Slew Drive move (continuous movement) until the key is released.

When the Shift key is held down and Absolute Position Mode is selected, a single press of an active arrow key causes one Step Drive move that is ten times larger than the minimal Step increment for that axis. When the Shift key is held down and Relative Position Mode is selected, the Step move is ten times larger than the *Delta* value for that axis.

When the Shift key is held down *and* an arrow key is held down, first a Shift-Step Move occurs, which is described in the preceding paragraph, then the stage slews at a speed that is ten times the current slewing speed.

See also Slew Drive; Slewing; Step Drive.

Calibrate

Calibrate is one of the software's four main menus. The functions listed on this menu are Rotation Center and Eucentric Tilt.

See also Rotation Center; Eucentric Tilt.

Continuous Rotation

Continuous Rotation is the third of the four main areas of the Stage Control window. It contains three buttons—Off, Simple, and Zalar—and a text entry field.

The Off button turns off the current rotation movement. Clicking on the STOP sign slows, then stops rotation.

The Simple button turns on continuous rotation around the stage's Rotation Center. (A tilt of 0° is recommended for Simple rotation during sputtering.)

Zalar Rotation turns on continuous *compucentric* rotation; that is, the specified set of x and y coordinates is the center of stage rotation. The stage adjusts the x, y, and rotation axes *continuously* to keep the coordinates at the center of rotation.

The speed of rotation is determined by the value in the Rotation Speed text field.

NOTE: Whenever rotation is continuous, rotation is clockwise. Rotation is continuous when slewing or when the Simple or Zalar button is on. Slewing occurs when (1) a Slew Drive button is pressed, (2) the joystick is used, or (3) an arrow key on the keyboard is held down.

See also Table A-1; Rotation Center.

Current Position

The values in the Current Position fields are updated as the stage moves to indicate the actual position of the axes.

NOTE: The values displayed at the bottom of the PC-ACCESS Sample Setup menu are the actual Current Position values only when the Store Position softkey is pressed.

Delete

The setting showing in the Position list box is deleted when the Delete button is pressed.

The PHI-defined positions Intro Sample, Extract Sample, Park Sample, and Retrieve Sample cannot be deleted.

See also Positions.

Delta

The values in the axis (X, Y, Z, Tilt, and Rotation) text fields are Delta values when Relative Position Mode is selected (and Target Positions when Absolute Position Mode is selected). A Delta value is the amount (in millimeters or degrees) by which the stage is to be moved from its Current Position when:

- Step Drive button is pressed,
- Drive All button is pressed,
- Arrow key on the keyboard (or on the numeric keypad when Num Lock is on) is pressed once.

The stage moves ten times the Delta value when the Shift key is held down and an arrow key is pressed.

Delta values are *not* considered when the Load or Drive button is pressed, the Simple or Zalar button is turned on, or stage movement is controlled by PC-*ACCESS*.

The current Delta values are remembered for subsequent selections of Relative Position Mode, are stored on disk upon normal exit, and are retrieved upon startup.

See also Arrow Keys; Drive All; Step Drive; Target Position.

Drive

Pressing the Drive button both loads the displayed Position's values and drives the stage to those coordinates.

See also Positions.

Drive All

Pressing the *Drive All* button moves all five axes to the positions indicated in the Target Position/Delta fields.

See also Arrow Keys; Delta; Drive; Slew Drive; Step Drive; Target Position.

Eucentric Tilt

The Eucentric Tilt function is located on the Calibrate menu of the Stage Control window. Selecting Eucentric Tilt opens the Eucentric Tilt Calibration dialog box (Figure A-4). This dialog box is used to compute values for sample thickness and beam offset.

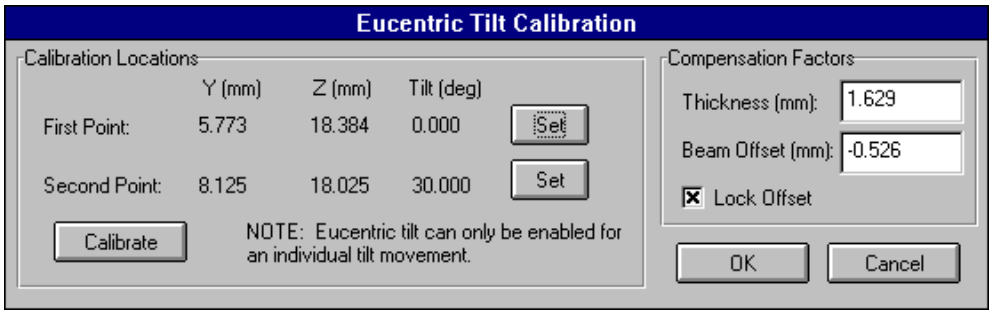

Figure A-4. *Eucentric Tilt Calibration Dialog Box.*

Beam Offset is a physical dimension relating to the individual system's geometry and, once calculated, does not need to be recalculated unless the stage or analyzer has been physically removed from the system. Turning on and leaving on the Lock Offset button after calibration of Beam Offset ensures that the value is not overwritten in subsequent calibrations of Thickness.

The sample Thickness parameter, on the other hand, is computed for each sample for which Eucentric Tilt is to be used. Eucentric Tilt uses the current Thickness value to adjust the y and z axes during tilt adjustment to keep the sample feature of interest centered (or close to centered) at the focal point of the analyzer.

NOTE: Eucentric tilt is performed properly only if (1) Thickness has been calibrated for the current sample, (2) Enable Eucentric Tilt is selected in the Properties dialog box, and (3) the Tilt Step Drive or Slew Drive button is used to move the stage.

The following procedure describes how to "calibrate" the stage for the thickness of the current sample. (The same seven steps are the beginning of the beam offset calculation procedure. Since it is usually performed only once, a footnote at the end of the procedure provides the remaining steps required to "calibrate" beam offset.)

NOTE: This procedure must be performed to determine the Thickness value for every new sample for which Eucentric Tilt will be desired.

1. In the Stage Control window, adjust the Tilt to "0."

2. In the PC-*ACCESS* SEM Menu Page 1, position the sample, as follows:

    a. Type 0 in Image Shift X and Y, and press Enter.

    NOTE: Do not use Image Shift to center features of interest. Doing so will cause an error in the Y positioning of the stage that will interfere with proper motion during Eucentric Tilt moves.

    b. Set Magnification 1 to 200 and Magnification 2 to 1000, pressing Enter after typing each value.

    c. Using Magnification 1 (200X), find a specific feature (10 to 50 µm in size) on the sample and center it on the monitor.

    d. Using Magnification 2 (1000X), refine the position of the sample at the center of the image.

    e. Adjust the focal point of the analyzer to the feature in one of two ways:

    - Adjust the value in the Focus parameter of the PC-*ACCESS* SEM Menu Page 1 (and press Enter);

    NOTE: Once focus has been established in this step, do not repeat this step until this Eucentric Tilt "calibration" procedure has been completed.

    - Adjust the Z parameter in the Stage Control window.

3. In the Stage Control window:

    a. Select the Properties function from the Stage menu. In the Stage Properties dialog box, turn off the Eucentric Tilt Enable button (if it is on). Press OK to close the dialog box.

    b. Select the Eucentric Tilt function from the Calibrate menu. In the Eucentric Tilt Calibration dialog box, press the Set button next to the First Point parameters.

    c. Enter a value between 30 and 45 in the Tilt parameter in the Stage Control window, and press the Step Drive button labeled "T." (A value of 30° is recommended, because no interference from other objects is likely.)

4. In the PC-*ACCESS* SEM Menu Page 1:

   a. Select Magnification 1 and press Enter; then, readjust the centering and focus by adjusting the Y and Z values in the Stage Control window.

   b. Select Magnification 2 and press Enter; then, readjust the centering and focus again by adjusting the Y and Z values in the Stage Control window.

5. In the Eucentric Tilt Calibration dialog box, calibrate the values as follows:

   a. Press the Set button next to the Second Point parameters.

   b. Proceed to step c unless calibrating the beam offset.

      Press the Lock Offset button to unlock (uncheck) the Beam Offset value.

      NOTE: Leaving the Lock Offset button on after calibrating Beam Offset ensures that the Beam Offset value will not get overwritten in subsequent calibrations of Thickness.

   c. Press the Calibrate button.

      Values are displayed for Thickness and Beam Offset, and the Lock Offset button becomes enabled.

   d. Press OK to close the Eucentric Tilt Calibration dialog box.

6. Select the Stage–Properties function. In the Stage Properties dialog box, turn on the Eucentric Tilt Enable button. Press OK to close the dialog box.

7. In the Stage Control window, return Tilt to 0°. The feature should return to the center of the screen and be in focus.*

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* 8. Verify the accuracy of the Beam Offset value by setting the Tilt parameter to a value half of that used for calibration (for example, 15° if 30° was used for calibration), and press the T Step Drive button.

If the sample is centered (to within 10 mm) and in focus, no additional calibration is necessary. If the positioning is not accurate, proceed to the next step.

9. Manually modify the value of Beam Offset as follows:

   a. Set the Tilt parameter back to 0°,and press the Step Drive button labeled "T."

   b. Select the Calibrate–Eucentric Tilt function. In the Eucentric Tilt Calibration dialog box, click on the Lock Offset button to uncheck it.

   c. Increase or decrease the Beam Offset number by approximately 0.1 mm. (Write down the value(s) used.)

   d. Turn on the Lock Offset button in the Eucentric Tilt Calibration dialog box.

   e. Press the Calibrate button. The Thickness value will change slightly.

Exit

The Exit function is located on the File menu of the Stage Control window. It closes the application, but it is not necessary to use the Exit button. The Stage Control software closes automatically when the PC-*ACCESS* software is exited.

Closing the application saves the current stage positions, calibrations, and properties, then closes the application window. If the stage motor is moving upon exit of this application, it decelerates until it stops.

File

File is the first main menu of the Stage Control window. Its submenu contains the Exit function.

See also Exit.

Help

Help is the fourth main menu of the Stage Control window. Its submenu contains Help Topics and About Stage Control.

See also Help Topics; About Stage Control.

Help Topics

Help Topics is listed on the Help menu. This function opens on-line help.

Initialize

The Initialize function is located on the Stage menu of the Stage Control window. This function "initializes" the stage movement motors; that is, sends the stage to a known position and resets the position registers to zero. When complete, the stage will be located at the "Intro Sample" position setting. Initialization of the stage must be performed any time that:

---

f. Press OK to close the dialog box.

g. Repeat steps 8 and 9 until the sample is centered and in focus at the end of step 8.

10. When the half-way angle positioning has been optimized, tilt the Stage to the Calibration angle (e.g., 30°) to verify that positioning accuracy here has not degraded.

    If it has degraded too much, repeat steps 12 and 13 until the best compromise value has been achieved.

- "Stage needs initializing" is displayed in the status line in the Stage Control window,
- Vacuum chamber is opened,
- Stage is moved manually (typically by a service technician),
- Position parameters displayed are suspected not to match the actual position of stage.

When Initialize is selected from the Stage menu, the stage begins the initialization process by driving each axis to its limits. The user is then prompted to initialize rotation.

When the prompt "Rotation needs initializing" is displayed during the stage initialization process, the operator looks through the viewport at the stage and drives the stage to align the dot on the rotation mechanism with the 0° line on the fixed base of the stage.

When the two marks are aligned, the operator presses the OK button in the prompt dialog box. The Rotation Current Position value is set to zero automatically. Initialization proceeds automatically.

Joystick

See "Getting Started—Using the Joystick" earlier in this section.

Load

Position values are loaded automatically when a Position setting is selected or a setting name is typed, then the Load button is pressed. The stage does not *move* when a Position setting is loaded using the Load button.

See also Positions.

Position Mode

The Position Mode buttons are Absolute and Relative. The selection of Position Mode determines the type of values in the text entry fields: Target Position (Absolute mode) or Delta (Relative mode).

NOTE: Slewing is not affected by the selection of Position Mode.

See also Delta; Slewing; Target Position.

Positions

Position is the first of the four main areas of the Stage Control window.

The Position settings allow the user to save, load, and drive to saved stage positions using predefined and user-defined names. These settings are saved to the hard disk whenever the Add, Update, Delete or Exit menu items are selected and are retrieved upon application startup. The predefined position settings are described in Table A-1.

Position parameters are loaded automatically when (1) a Position setting is selected, or (2) a setting name is typed and the Load button is pressed. The stage does not *move* when the Load button is pressed. (Pressing the Drive button both loads the Position parameters and drives the stage to that Position.)

A new Position setting that uses the Current Position values is *created* by typing the name in the list box and pressing the Add button. The setting showing in the

Table A-1. PHI-Defined Position Settings

Setting	Purpose
Center	If the Rotation Center Calibration function has been performed for the current sample, pressing Drive when this position setting is showing in the list box moves the stage to the software-stored rotation center point of the stage.
Intro Sample	Used for placing a sample onto the stage. Pressing Drive when this position setting is showing in the list box moves the stage to the intro-unclip position (aligned below the intro arm destination). The operator is notified to slide the intro arm in. When OK is selected, the stage moves up and clips onto the sample holder (intro-clip position). The operator is then notified to slide the intro arm out. When OK is selected, the operation is complete.
Extract Sample	Used for removing a sample from the stage. Pressing Drive when this position setting is showing in the list box moves the stage to the intro-clip position (aligned with the intro arm destination). The operator is notified to slide the intro arm in. When OK is selected, the stage moves down, unclipping itself from the sample holder (intro-unclip position). The operator is then notified to slide the intro arm out. When OK is selected, the operation is complete.
Park Sample†	Used for moving a sample from the stage onto the parking attachment. Pressing Drive when this position setting is showing in the list box moves the stage to the park-clip position (aligned with the parking attachment destination). The operator is notified to position the parking attachment. When OK is selected, the stage moves down, unclipping itself from the sample holder (park-unclip position). The operator is then notified to retract the parking attachment. When OK is selected, the operation is complete.
Retrieve Sample†	Used for moving a sample from the parking attachment onto the stage. Pressing Drive when this position setting is showing in the list box moves the stage to the park-unclip position (aligned below the parking attachment destination). The operator is notified to position the parking attachment. When OK is selected, the stage moves up and clips onto the sample holder (park-clip position). The operator is then notified to retract the parking attachment. When OK is selected, the operation is complete.

* The only difference between “clip” and “unclip” is the Z axis value. This Delta Z value is modifiable by the user in the Stage Properties dialog box.

† This setting is available only if the user has indicated in the Stage Properties dialog box that the system is configured with a parking attachment.

list box is *changed* to the Current Position values when the Update button is pressed. The position setting showing in the list box is *deleted* when the Delete button is pressed. (The predefined positions Intro Sample, Extract Sample, Park Sample, and Retrieve Sample cannot be deleted.)

> *NOTE: The predefined positions Intro Sample, Extract Sample, Park Sample, and Retrieve Sample can be changed or updated, but this is not recommended, because this will change the intro-clip and park-clip positions.*

Properties

The Properties function is located on the Stage menu of the Stage Control window. Selecting Properties opens the Stage Properties dialog box.

See Stage Properties.

R

See Step Drive.

Rotation

See Delta; Target Position; Continuous Rotation; Rotation Center; Rotation Speed; Initialize; Slew Drive; Slewing; Step Drive.

Rotation Center

The Rotation Center function is selected from the Calibrate main menu of the Stage Control window. Selecting this menu item opens the Rotation Center Calibration dialog box (Figure A-5).

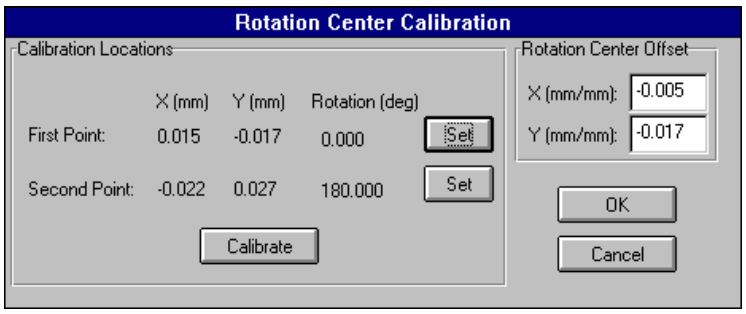


Figure A-5. Rotation Center Calibration Dialog Box.

The Rotation Center Calibration dialog box allows the user to calculate the Rotation Center Offset values so that the sample will rotate in the center of the user's SEM image. The calibration is performed as follows:

1. Use Manual Z Align in PC-*ACCESS* to ensure that the sample surface is perpendicular to the electron beam. This is done by measuring the elastic peak at or near the extreme edges of the sample, especially in the y direction. Tilt should be adjusted such that the elastic peak energy is the same at the y extremes.

2. Find a specific feature on the sample. Drive the stage so that this feature is centered in the SEM image using the Stage Control parameters or the joystick.

3. In the Rotation Center Calibration dialog box, press the Set button corresponding to First Point.

4. The operator message, "Rotate the sample by 180 degrees and center the feature in the SEM image," is displayed. Click OK to close the message. Change the Stage Control parameters again to rotate the stage 180 degrees and re-center the feature. (The Tilt parameter must be the same as the First Point Tilt parameter.) Then, press the Set button corresponding to Second Point.

5. Press the Calibrate button in the dialog box. The Rotation Center Offsets are calculated and displayed.

   Pressing the OK button stores the new calibration values (and performs the calibration if not already done). (Pressing the Cancel button will ignore all changes.)

6. Verify the calibration by performing the following steps. The SEM image should keep the center point steady as it rotates.

   a. Enter a value of 0 in the X and Y Target Position parameters.

   b. Press the Drive All button.

   c. Press the Simple button in Continuous Rotation.

Rotation Speed

The Rotation Speed text entry field allows the user to adjust the speed of rotation when (1) Drive or Drive All is pressed, (2) any of the Slew Drive buttons is pressed, (3) Simple or Zalar (Continuous Rotation) is pressed, and (4) slewing rotation using the arrow keys on the keyboard.

Holding down the Shift key while slewing rotation using the arrow keys on the keyboard rotates the stage ten times faster than the value in the Rotation Speed field.

See also Arrow Keys; Slewing.

Simple

See Continuous Rotation.

Slew Drive

The Slew Drive buttons are the arrow buttons in the Stage Control window. Except for the Rotation axis, the buttons come in sets of two: positive movement and negative movement. (Rotation slewing is always positive (clockwise).)

NOTE: The Slew Drive buttons (and the joystick) are not affected by the selection of Position Mode or by any values in the axes' text fields.

Using any of the Slew Drive buttons associated with the X, Y, Z, and Tilt axes moves the stage continuously in the direction associated with that button until the button is released. (The Rotation Slew Drive button moves the stage clockwise around the stage's rotation center only. There is no Slew Drive button for compucentric rotation.)

The x- and y-axis slewing speeds depend on the current image magnification from the PC-*ACCESS* software and on the X-Y Slew Control Ratio in the Properties dialog box. The z-axis slewing speed depends on the Z Slew Ratio in the Properties dialog box. The speed of movement for the Tilt axis is fixed. The Rotation slew speed is specified in the Rotation Speed field in the main window.

The slewing speed increases tenfold when the Shift key is held down while using an active arrow key on the keyboard.

The Slew Drive buttons are not available while the stage is performing a step drive.

See also Arrow Keys; Slewing.

Slewing

"Slewing" is a continuous series of incremental moves. Slewing occurs when (1) a Slew Drive button is pressed, (2) the joystick is used, or (3) an arrow key on the keyboard is held down.

NOTE: Slewing the Rotation axis causes clockwise (positive) rotation only.

See also Moving the Stage; Using the Joystick; Arrow Keys; Slew Drive.

Slewing Speed

See Arrow Keys; Slew Drive.

Stage

Stage is the second main menu in the Stage Control window. Its menu contains Initialize and Properties.

See also Initialize; Properties.

Stage Control

Stage Control is the second of the four main areas of the window.

See also Arrow Keys; Delta; Position Mode; Target Position; Slew Drive; Step Drive; Current Position.

Stage Properties

Selecting Properties from the Stage menu opens the Stage Properties dialog box (Figure A-6). Table A-2 describes the fields of the dialog box.

The OK button will save the new property values. The Cancel button will ignore the new values.

When the user changes the X-Y Slew Ratio value, the operator is notified of the minimum magnification that can be used to obtain the maximum motor speed.

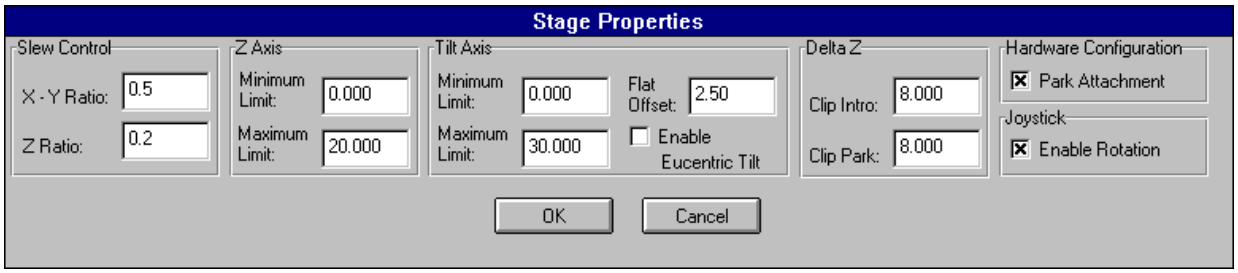


Figure A-6. Stage Properties Dialog Box.

Table A-2. Stage Properties Dialog Box Fields.

Field	Purpose
Slew Control	
X–Y Ratio	Specify a ratio between 0.1 and 10.0, which affects the slewing speed of the x and y axes. The slewing speed of the x and y axes is also affected by the current Magnification value in the PC-*ACCESS* SEM menu, Page 1.
Z Ratio	Specify a ratio between 0.1 and 10.0, which affects the slewing speed of the z axis.
Z Axis	
Minimum Limit Maximum Limit	Specify the minimum and maximum z-axis limits, within the range of the hardware limits. The stage will not move if the requested values are outside these limits.
Tilt Axis	
Minimum Limit Maximum Limit	Specify the minimum and maximum tilt-axis limits, within the range of the hardware limits. The stage will not move if the requested values are outside these limits. ***ATTENTION: The Tilt axis has a default software limit of 30 degrees, which can be extended in this dialog box to the hardware limit of 60 degrees. This is not recommended, however, because a tilt between 30 and 60 degrees can cause the sample to hit the analyzer.***
Flat Offset	A value established at installation that compensates for individual instrumentation. The value is the adjustment necessary for the *stage* to be perpendicular to the incident electron beam.
Enable Eucentric Tilt	Click to turn on this box to automatically adjust the y and z axes when the tilt axis is changed (thereby keeping the feature centered at the focal point of the analyzer). The adjustments of y and z are made using the Thickness and Beam Offset values that were generated using Eucentric Tilt Calibration. *NOTE: Use the Tilt Step Drive or Slew Drive button.*
Delta Z	
Clip Intro	Specify the clearance needed (the movement of the z axis needed) to clip/unclip the stage to/from the intro arm for the current sample height.
Clip Park	Specify the clearance needed (the movement of the z axis needed) to clip/unclip the stage to/from the parking attachment for the current sample height. (The Clip Park field is available only when the Parking Attachment option has been selected in the Hardware Configuration section of this dialog box.)
Hardware Configuration	
Park Attachment	Click to turn on this box on if the system has a parking attachment.
Joystick	
Enable Rotation	Click to turn on this box to control the action of the rotating knob on the joystick. When this box is set to on, turning the joystick knob will rotate the stage clockwise. The ALL LOCK button must be off to use this feature.

Status Line

The status line is the fourth main area of the window, located at the bottom of the window.

The following are some of the possible status messages: "Stage Positioned"; "Stage Moving"; "WARNING: Stage needs to be initialized!!"; "Initializing stage"; and "Registering Stage Positions."

Step Drive

After typing a value in the Target Position (Absolute Position Mode) or Delta (Relative Position Mode) field for the X, Y, Z, or Tilt axis, the user presses the associated Step Drive button to move the stage one step along that axis. When Delta values are displayed, the stage *changes* position in the amount of the value in that Delta field. When Target Positions are displayed, the stage moves so it is located at the coordinate shown in that Target Position field.

When Target Positions are displayed, the *Rotation* Step Drive button labeled "R" rotates the stage 1° clockwise around the stage's rotation center. The Rotation Step Drive button labeled "Comp" rotates the stage 1° clockwise and automatically adjusts the x and y axes (compucentric rotation) to allow the desired feature (Current Positions of the x and y axes) to remain centered (or close to centered) in the SEM image.

The Step Drive buttons are not available while the stage is performing a step drive.

See also Arrow Keys.

STOP

The STOP button will decelerate the moving stage until it stops.

T

See Step Drive.

Target Position

The values in the text entry fields (X, Y, Z, Tilt, and Rotation) are Target Positions when Absolute Position Mode is selected (and Delta values when Relative Position Mode is selected). A Target Position value is the axis coordinate (in millimeters or degrees) at which the stage should be located after the move.

The current Target Position values are stored on disk upon normal exit and are retrieved upon startup.

NOTE: To determine the current location of the stage, refer to the values displayed for Current Positions, not Target Positions, because the Target Position values are updated after a move only *when the stage is* slewed *along that axis.*

See also Arrow Keys; Current Position; Delta; Drive All; Slew Drive; Step Drive.

Tilt

See Eucentric Tilt; Stage Properties; Getting Started: "The Two Motor Groups"; Continuous Rotation; Delta; Target Position; Slew Drive; Step Drive.

NOTE: Regardless of operator action, stage movement stops when a Tilt- axis limit (defined in Stage Properties) is reached.

Update

The setting showing in the Position list box is changed to the values in the Current Position fields when the Update button is pressed.

See also Positions.

X

See Stage Properties; Getting Started: "The Two Motor Groups"; Delta; Target Position; Slew Drive; Step Drive.

Y

See Stage Properties; Getting Started: "The Two Motor Groups"; Delta; Target Position; Slew Drive; Step Drive.

Z

See Stage Properties; Getting Started: "The Two Motor Groups"; Delta; Target Position; Slew Drive; Step Drive.

NOTE: Regardless of operator action, stage movement stops when a Z- axis limit (defined in Stage Properties) is reached.

Zalar

See Continuous Rotation.

Appendix B:
Ion Gun Control Software

The front panel of the Ion Gun Control unit has an on/off switch and a power indicator light. When the power is on (normal operation leaves the unit on), the control unit is operated using the Ion Gun Control window on the PC's desktop.* The features and operation of the Ion Gun Control window are described in the following paragraphs.

Overview

The features and operation of the Ion Gun Control window are described in the following paragraphs.

Starting and Closing the Ion Gun Control Software

Ion Gun Control software is started automatically when PHI's system software (e.g., PC-*ACCESS*) is opened, and exited automatically when the system software is exited. If PC-*ACCESS* is not available, software is started by clicking on the Ion Gun Control icon in the PHI group on the PC's desktop.

NOTE: The software opens in the Column Tracking mode, which is the recommended mode of operation. This feature allows the Column Control parameters to scale automatically with changes in beam voltage, which allows the ion beam to remain in focus and the beam size to remain unchanged.

NOTE: If the Model 11-066 hardware is turned off, then on, software detects this and reinitializes hardware with the current setting, if possible.

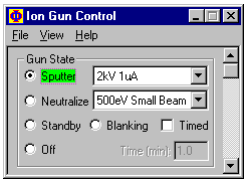
Using the Tab Key on the Keyboard

The Tab key on the keyboard works like an Enter key: It activates an operator entry.

Features of the Ion Gun Control Window

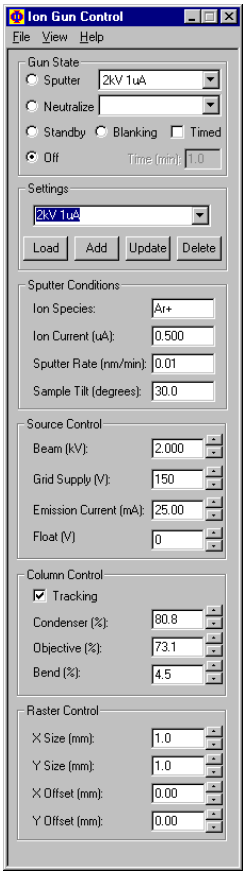
Selecting Short Menu from the View pull-down menu toggles the long and short Ion Gun Control menus.

* The Ion Gun Control window on the Sun workstation is documented through on-line help on the PHI Quantum 2000.



Use the short menu is for routine operation. When Ion Gun Control software is started, the short menu is displayed in its last location.

The short version displays Gun State; using the scroll bar displays other portions of the window.



Use the long menu to align the ion gun and calibrate ion gun parameters with sputter rates. When the long menu is displayed, it is shown in its last location.

The long menu has the following groups of information.

- **Gun State** is used to turn the ion gun to Sputter, Neutralize, Off, Standby or Blanking. The Settings fields next to the Sputter and Neutralize buttons are used to select a setting to be used for operation.
- **Settings** is used to define, name, change, and delete sets of ion gun parameters.
- **Sputter Conditions** allow you to enter information like sputter rate and gas species to be saved with the setting for future reference.
- **Source Control** and **Column Control** display current parameter settings for ion gun optics. **Raster Control** parameters define the size of the raster area. Up and down arrows to the right of the **Source**, **Column**, and **Raster Control** parameter fields may be clicked on with the mouse button to increment values. Holding down the mouse button on an arrow accelerates the rate of change of the value displayed. Clicking in a parameter field also allows the up and down arrows on the *keyboard* to change the parameter, and holding down the keyboard arrow button accelerates the rate of change. Pressing the keyboard's Tab key moves the cursor to the next parameter field.

Interaction between Ion Gun Control Software and PC-ACCESS

While PC-*ACCESS* is able to affect Ion Gun Control software, the Ion Gun Control software *cannot* affect PC-*ACCESS*. Physical Electronics recommends operation of the ion gun as follows.

1. In PC-*ACCESS*:
   a. Open the Ion Gun Setup menu from the Hardware menu.
   b. Manually enter the values from the Ion Gun Control window into the corresponding fields in the Ion Gun Setup menu. (The values of the fields in the PC-*ACCESS* Setup Ion Gun menu are stored with the data acquisition file.)

NOTE: Not all fields of the Ion Gun Control window are represented in the PC-ACCESS Ion Gun Setup menu.

c. Set the ion gun to Standby.

d. Exit the Ion Gun Setup menu.

2. In the Ion Gun Control window, set the ion gun to Blanking or Neutralize.

   ATTENTION: PC-ACCESS switches the ion gun from Blanking or Neutralize to Sputter automatically when Acquire–Profile is started. It automatically switches back when Sputter Time ends.

   The ion gun is on when the Gun State is Neutralize. The Neutralize mode should be used only when an ion beam is desired during data acquisition.

3. In PC-*ACCESS*, select Setup Acquire–Setup Profile to define the acquisition parameters. Then, select Select Acquire, Acquire–Profile, and press the Acquire softkey.

   Data acquisition begins, using the parameters defined in the Profile Setup menu. PC-*ACCESS* switches the Ion Gun Control window to Sputter, running the ion gun for the length of Sputter Time defined in the Profile Setup Menu. When that time ends, PC-*ACCESS* switches the Ion Gun Control window to Neutralize or Blanking—whichever Gun State the ion gun was in before the acquisition was started.

   The status of the ion gun is displayed both at the bottom of the PC-*ACCESS* window and in the Ion Gun Control window.

To run the ion gun without acquiring data, put the Ion Gun Control window's Gun State in Standby, then use the PC-*ACCESS* Setup Ion Gun menu to start (On) and stop (Standby) the ion gun.

Getting Started

This section provides the following information on getting started:

- Operating the Ion Gun
- Selecting Parameter Values for Ion Gun Settings
- Computing Sputter Rates for Repeatability and Reliability

NOTE: A file for reporting software performance issues is available in the PHI group on the desktop. The file is named SPR.WRI or IGSPR.WRI.

Operating the Ion Gun

Operation of the ion gun falls into five major functional areas:

- Not using the ion gun (Gun State is Off),
- Warming up the ion gun or the ion gun is idle (Gun State is Standby),
- Preparing the ion gun for depth profiling (Gun State is Blanking),
- Sputtering the sample with the ion beam (Gun State is Sputter),
- Delivering a low-energy neutralization charge to the sample (Gun State is Neutralize).

NOTE: Sputter and Neutralize are the ion gun's two On states. The only difference between the two is how the operator saves the setting, as a Sputter setting or as a Neutralize setting. Once Gun State is turned to Sputter or Neutralize, the hardware operates to achieve and maintain all gun parameters.

(A sixth operational situation is the use of the ion gun the first time after installing a new ionizer assembly or after a bake. Refer to the *Ion Gun Component Manual* for this procedure.)

When the ion gun is not in use, Gun State in software should be set to off. In this state, software sets all Source, Column, and Raster Control voltages to 0. The Ion Gun Control window is open, the leak valve is closed, and the Thermovalve Control (if present) is off.

ATTENTION: The operator receives the following warning when changing Gun State to Off. "Turning off gun will result in zero emission and loss of feedback to any automated leak valves. Take necessary steps to turn off gas flow to ion gun or loss of vacuum could occur."

Gun State Is Standby

Use Standby to warm up the ion gun by adjusting Grid Supply, Emission Current, and Float parameters. The Beam, Condenser, and Objective voltages are set to minimal voltages to ensure proper gas flow with an automated leak valve. Minimal voltage operation does not adversely affect the sample.

The Extractor Pressure window should be displayed during ion source warm-up. Select Extractor Pressure from the View menu in the Ion Gun Control window. When the ion source is warmed up, use the manual leak valve on the ion gun to adjust the pressure in the source.

If an RVG 050 Thermovalve Control is used to control pressure during ion gun operation, Extractor Pressure should be brought to a level *above* the desired operating pressure before turning on the Thermovalve Control, because the Thermovalve Control can only *close* the leak valve. (For example, when 25 mPa is the desired operating pressure, reach an Extractor Pressure of 40 to 50 mPa before turning on the Thermovalve Control.)

If a Thermovalve Control is *not* used, keep track of the Extractor Pressure reading and adjust the leak valve manually as necessary to compensate for the effect of heat on the manual leak valve.

Standby is also used when the ion gun is idle. The ion gun software will automatically switch from Blanking to Standby after one hour if the ion gun is not used. In the Standby state, the beam energy is lowered and the beam is deflected off the sample.

If you decide to use the ion gun in the Sputter state after it has been in Standby, there is a 2 to 3 minute waiting period to allow the thermovalve to stabilize.

Gun State is Blanking

The ion gun should be put into the Blanking state before starting a depth profile acquisition. Blanking keeps the beam energy at the selected setting, but deflects the beam off the sample. During depth profiling, the Gun State will alternate between Sputter and Blanking.

Gun State Is Sputter or Neutralize

Once Gun State is turned to Sputter or Neutralize, hardware operates to achieve and maintain all gun parameters (except Float if disabled using the Properties–Float Supply function). Sputtering and neutralization can be timed using the Timed function.

You can choose to display extractor pressure during ion gun operation by selecting View–Extractor Pressure…. Select Close to remove the display.

NOTE: Do not leave the Extractor Pressure window open for long periods of time. Close the window when a stable operating pressure has been achieved.

You can also change operating parameters during ion gun operation by (1) loading a new setting, (2) typing a different value in any parameter field, or (3) activating a different Gun State button. New values are implemented immediately when Gun State is set to Sputter or Neutralize.

NOTE: *Changing ion gun parameters during acquisition of a depth profile changes the sputtering conditions, which will need to be taken into account when evaluating the resulting data.*

Whenever a new setting is loaded or a new value is entered in the beam voltage, raster size, raster offset, or calibration properties parameters, the potential for clipping is checked and the operator is notified if parameter changes are required.

Selecting Parameter Values for Ion Gun Settings

Settings of ion gun parameters depend primarily on beam conditions (beam and raster sizes) desired. Sputter rate varies according to the sample material and the Source and Column Control parameters used for sputtering. Different "settings" are defined to work for different situations.

Software is installed with several predefined settings that were optimized on a system with a flange-to-target distance of 30 mm. Adjusting the values in Calibration properties until the predefined setting sputters a raster area of the same size makes a predefined setting valid on the operator's unique hardware configuration. (These Calibration properties need to be defined when the ion gun control is first installed, and again only if maintenance is performed on the ion gun control's deflection control circuits.)

Computing Sputter Rates for Repeatability and Reliability

Sputter rate values vary and should be computed periodically using demonstration and calculations, as follows.

1. In the Ion Gun Control window, turn on Column Tracking so that Column Control parameters scale automatically with changes in beam voltage, which allows the ion beam to remain in focus and beam size to remain unchanged.

2. Define ion gun control parameters that match the acquisition scenario for which the sputter rates need to be calculated.

3. Acquire a depth profile through a layer of a sample that has an accurately known thickness. (A sample of tantalum oxide or silicon dioxide—or a thin film of material typically used in your application—is recommended.)

4. Compute the sputter rate (in nanometers per minute) using the time elapsed to sputter through the layer and the thickness of that layer.

5. Create a setting in the Ion Gun Control software based on the demonstration so that parameters may be used for future sputtering routines. In this way, maximum advantage is made of the hardware's repeatability and depth profile data can be compared and understood with reliability.

   When defining the setting, select a descriptive name (e.g., 5kV 1uA) and parameters that match or are based on the Source and Column Control parameters used in the demonstration.

Functions

Functions of the software are listed in alphabetical order. When the Ion Gun Control function is selected from the Help menu, a dialog box displays the version of the Ion Gun Control software. Selecting OK closes the dialog box.

Add

See Settings.

Beam

See Source Control.

Bend

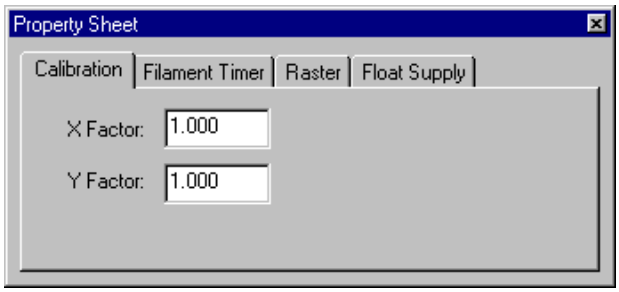
See Column Control.

Blanking

See Gun State.

Calibration

Select View–Properties…, then Calibration to open Calibration.



Software is installed with several predefined settings optimized on a system with a nominal flange-to-target distance of 30mm. Adjusting values in Calibration properties until the predefined setting sputters a raster area of the same size makes a predefined setting valid on the operator's unique hardware configuration. These Calibration properties need to be defined when the ion gun control is first installed, and again only if maintenance is performed on the ion gun control's deflection control circuits.

The entered value is applied immediately when Gun State is set to Sputter/Neutralize.

NOTE: When a new value is entered in a calibration factor parameter, potential for clipping is checked, operator is notified if a parameter change is required.

Click on Filament Timer, Float Supply, or Raster to see a different property dialog box, or click in the upper left corner to close the dialog box.

Column Control

The Column Control area of the Ion Gun Control window contains the Tracking, Condenser, Objective, and Bend variables.

Optics parameters are divided into Source Control and Column Control. Column Control includes the Condenser, Objective, and Bend parameters. Column "Tracking," when on, shows the condenser, objective, and bend voltage values as percentages of the beam voltage. (When Tracking is *not* checked, the condenser, objective, and bend voltage parameter values are expressed in volts.) The limits of these parameters are given in Table 3-1. The values of the parameters are applied when the Gun State is changed to Sputter or Neutralize.

Table B-1. *Column Control Parameter Limits.*

Parameter	Valid Range	Step Size
Condenser	0 to 5500V, 0% to 110%	1V, 0.01%
Objective	0 to 5500V, 0% to 110%	1V, 0.01%
Bend	0 to 350V, 0% to 7%	1V, 0.01%

Condenser

See Column Control.

Delete

See Settings.

Emission Current

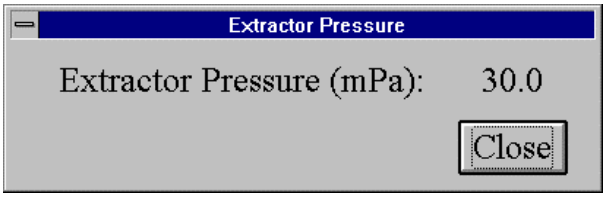
See Source Control.

Exit

Selecting Exit from the File menu displays a dialog box. The operator presses OK in the dialog box to set the gun state to Off if it is Sputter, Neutralize, Blanking or Standby, or presses Cancel to close the Exit dialog box without closing the Ion Gun Control application.

Extractor Pressure…

This function is available from the View menu. Selecting this opens the Extractor Pressure window, which has a large display of the extractor pressure (in milliPascals) for easy reading from a distance. The pressure is monitored at a rate of 4 Hz for the first minute that the window is open. After the first minute, the monitoring rate changes to once every 5 seconds.



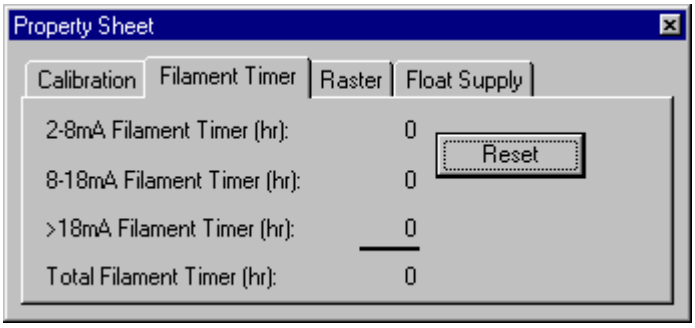
The window remains on display until the Close button (or the upper left corner) is pressed.

NOTE: Do not leave the Extractor Pressure window open for long periods of time. Close the window when a stable operating pressure has been achieved.

Filament Timer

Select View–Properties…, then Filament Timer to open the Filament Timer dialog box. This property dialog box displays the time, in hours, that the ion gun state has been in each of the emission current ranges (2-8 mA, 8-18 mA, >18 mA).

The value is updated each time the minutes counter reaches 60. (*NOTE: The minutes counter is cleared when power to the Ion Gun Control is turned off, so actual run time on the ionizer could be greater than the time displayed here.*)



The Reset button in the dialog box is used to set the timer to 0 when a new ionizer assembly is installed in the ion gun. When Reset is pressed, a dialog box is displayed. Selecting OK resets the timer to 0; pressing Cancel instead closes this dialog box and no action is taken.

Click on Calibration, Float Supply, or Raster to see a different property dialog box, or click in the upper left corner to close the dialog box.

File

The File menu is one of the main menus in the Ion Gun Control window. It is used to activate the Exit function.
See also Exit.

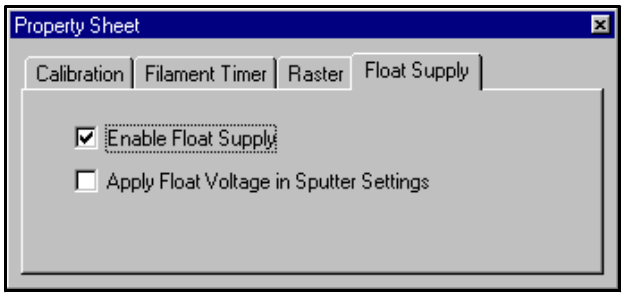
Float

See Float Supply; Source Control.

Float Supply

Select View–Properties…, then Float Supply to open the Float Supply properties dialog box (Figure 3-5). This dialog box is used to enable or disable the float supply of the ion gun control. When *Enable Float Supply* is not checked. (1) the float supply is disabled, (2) float values in all ion gun settings are ignored, and (3) the *Apply Float Voltage in Sputter Settings* field is ignored.

When the second option, Apply Float Voltage in Sputter Settings, is not checked (which is the default), any float value specified in a setting selected for the *Sputter* gun state is ignored (i.e., the float supply is set to 0 V). When this option is checked, the Sputter gun state applies any float value specified in the selected setting.
Click on Calibration, Filament Timer, or Raster to see a different property dialog box, or click in the upper left corner to close the dialog box.



Grid Supply

See Source Control.

Gun State

The Gun State area of the Ion Gun Control window defines the "state" of the ion gun, as follows.

- Sputter or Neutralize turns on the ion gun and implements all optics and raster parameters.
- Standby warms the ion gun according to the Grid Supply, Emission Current, and Float parameters. The other optics and raster parameters are set at default Standby settings to stabilize the pressure in the ion source. Standby also is used when the ion gun is idle for more than one hour.
- Blanking maintains the selected beam energy but deflects the beam off the sample. It is used during depth profiling.
- Off turns off the ion gun, changing optics and raster parameter values to 0.

When Gun State is Sputter or Neutralize, the color of the radio button is set to *green* as a visual indicator. When it is in the Blanking, Standby or Off state, the color of the radio button is gray.
See also Timed; Operating the Ion Gun with PC-*ACCESS*.

Help

The Help menu is one of the main menus in the Ion Gun Control window. It is used to activate the About Ion Gun Control function.
See also About Ion Gun Control.

Ion Current

See Sputter Conditions.

Ion Species

See Sputter Conditions.

Load

See Settings.

Neutralize

See Gun State.

Objective

See Column Control.

Off

See Gun State.

Properties…

The Properties… function, available from the View menu, opens the Property Sheet dialog box, which has four "pages". Calibration, Filament Timer, Float Supply, and Raster.
See Calibration; Filament Timer; Float Supply; Raster.

Raster

Select View–Properties…, then Raster to open the Raster properties dialog box. This dialog box is used to select from (1) *Off* for no raster (ion beam remains stationary during sputtering), (2) *External*, which allows an external device (a scan generator, for example) connected to J5 and J6 to drive the ion gun deflection, or (3) *Internal*, the default mode at startup, in which the ion beam sputters a sample while moving in an interlaced triangular pattern across the sample's surface.

External is typically used for ion-beam-induced imaging or Secondary Ion Mass Spectroscopy data acquisition. Internal, for sputter depth profiling in the Auger Electron Spectroscopy or X-ray Photoelectron Spectroscopy techniques.

Click on Calibration, Filament Timer, or Float Supply to see a different property dialog box, or click in the upper left corner to close the dialog box.

Raster Control

Raster Control of the Ion Gun Control window contains the X Size, Y Size, X Offset, and Y Offset parameters, which control the size and position of the raster area. Values are applied immediately when the gun is Sputter or Neutralize.

In the X Size and Y Size parameters, specify the raster beam size in millimeters in the axis. In the X Offset and Y Offset parameters, specify the offset in millimeters to position the raster pattern in the axis.

NOTE: The operator should mechanically align the ion gun with the center of the analysis area. This minimizes the amount of DC offset required, which maximizes the possible raster area.

When a new value is entered in a raster size or raster offset parameter, potential for clipping is checked and operator is notified if a parameter change is required.

See also Raster.

Sample Tilt

See Sputter Conditions.

Settings

Settings drop-down list displays defined ion gun settings. Software is installed with several predefined settings. All setting's parameters are applied when the Gun State is changed to Sputter or Neutralize, but only the Grid Supply and Emission Current parameters are applied when the Gun State is in Blanking or Standby.

Typing a name in the Setting field, then pressing the Add button creates a setting having the currently displayed Source and Column Control, Raster Control, and Sputter Conditions parameter values. Changing these values, then pressing the Update button revises the setting. Pressing the Delete button removes the currently displayed setting from the Settings list.

ATTENTION: Create back-ups of the settings files. If settings files become corrupted, delete them and the back-ups copied to the c:\phi\exe directory.

Click and hold down the mouse in the Settings field to display the Settings option menu. To load a setting, drag the mouse to highlight the desired setting, and release the mouse. Or type the setting name in the Settings field, then press the Load button. Selecting a setting loads its associated parameters automatically.

NOTE: The potential for clipping is checked every time a new setting is loaded. The operator is notified if parameter changes are required.

NOTE: For safety, Gun State is changed from Sputter or Neutralize to Standby automatically when a setting is loaded.

See also "Selecting Parameter Values for Ion Gun Settings."

Short Menu

This function is available from the View menu. Selecting Short Menu toggles the Ion Gun Control window between the short and long menus. When Short Menu is checked, it displays the short menu where it was last displayed. When Short Menu is unchecked, it displays the long menu where it was last displayed.

When the system first starts up and every time the software is restarted, the short menu is displayed in the lower right corner of the desktop. When the Short Menu function is toggled to unchecked, the long menu is displayed in the upper right hand corner of the screen.

Source Control

Optics parameters are divided into Source and Column Control. Source Control includes Beam, Grid Supply, Emission Current, and Float voltage parameters. The limits of these parameters are given below. Values of the Grid Supply and Emission Current parameters are applied when Gun State is in Standby or Blanking; the beam parameter is also applied when Gun State is changed to Sputter or Neutralize.

Table B-2. Source Control Parameter Limits.

Parameter	Valid Range	Step Size
Beam voltage	0.200 to 5.0 kV	0.1 kV
Grid supply	100 to 200 V	1 V
Emission current	0 to 50 mA	0.01 mA
Float	–500 to 0 V	1 V

In the Beam field, specify beam voltage in volts. When Column Tracking is selected, Condenser, Objective, and Bend voltages automatically scale with changes of Beam voltage.

NOTE: A change in the Beam parameter value does not change the raster size; the software adjusts the deflection voltages to maintain the specified raster size when the Beam value is changed—regardless of the state of Column Tracking.

When a new value is entered in Beam voltage parameter, potential for deflection clipping is checked and the operator is notified if a parameter change is required.

In the Grid Supply field, specify the voltage that will achieve maximum ion beam current (typically in the range 140 to 160 V).

In Emission Current field, specify the current output (in milliamperes) of the ion gun filament.

NOTE: The Filament Supply is turned off if emission current does not reach its asked-for value in 10 seconds.

In the Float field, specify the float voltage to be applied. *See also* Float Supply.

Sputter Conditions

The Sputter Conditions area of the Ion Gun Control window contains four fields that for record-keeping *only*.

- Ion Species—enter the name of the gas being used in the ion gun;
- Ion Current—enter the current incident upon a positively biased sample (measured, for example, using a Keithley picoammeter);
- Sputter Rate—enter the sputter rate (in nanometers per minute) that was calculated during calibration on a standard sample for a specified time;
- Sample Tilt—enter the tilt of the sample

Sputter Rate

See Sputter Conditions.

Standby

See Gun State.

Timed

The Timed checkbox in the Gun State part of the window enables timed sputtering or neutralizing. When Timed is checked, the Time field is active. The user can enter a time from 0 to 999.9 minutes. The next time the ion gun is set to Sputter or Neutralize, the software will start a timer for the specified time interval. The countdown is displayed in yellow until the countdown is complete, at which time the ion gun is switched back to Standby automatically.

Changes made to Timed and Time when the gun is in Sputter or Neutralize mode do not take effect the *next* time the ion gun is set to Sputter or Neutralize.

Tracking

See Column Control.

Update

See Settings.

View

The View menu is one of the main menus in the Ion Gun Control window. It contains the Short Menu, Extractor Pressure…, and Properties… functions.

See also Extractor Pressure…; Properties…; Short Menu.

X/Y Size

See Raster Control.

X/Y Offset

See Raster Control.

Appendix C:
AES Element Table

Physical Electronics' (PHI's) PC-*ACCESS* software for the 680 Scanning Auger Nanoprobe contains a database called the "AES Element Table." The original settings of the relevant fields of this database, plus two additional columns (in gray), are presented here for the user's reference. The computer enters values from this table into the appropriate fields of a menu when the user enters (in the Element field) one of the Names of the elemental transitions from this Element table. The contents of the columns are defined as follows:

- **Name**—abbreviation representing the elemental transition. The asterisk (*) indicates the transition the computer enters (e.g., Au3) when the user types only the element's abbreviation (e.g., Au).
- **At.#**—atomic number of the elemental transition (not included in the database).
- **N(E) Peak**—The energy of the element's identifying peak in an undifferentiated spectrum.
- **dN(E) Peak**—The energy of the element's identifying peak in a differentiated spectrum (not included in the database).
- **S_x3/5/10**—The approximate elemental sensitivity factors, relative to that used to obtain the silver spectrum, when the electron voltage is at 3 (top value), 5 (middle value), and 10 kV (bottom value).
- **Acquisition: Lower/Upper**—The energies entered into the Lower Limit (left value) and Upper Limit (right value) menu fields for the *Acquisition* Window parameter in Setup menus and in the Narrow Acq Window parameter in the Element Table menu.
- **Analysis: Lower/Upper**—The energies entered into the Lower Limit (left value) and Upper Limit (right value) menu fields for the *Analysis* Window parameter in Setup menus and the Element Table menu.
- **B1 and B2**—B1 and B2 are energies that are used in background calculations during 2- and 3-point acquisitions of lines, maps, and profiles. B2 is a value greater than the peak energy. It is entered in the Background Energy field for 2-point acquisitions and in the E2 field for 3-point acquisitions. B1 is a value less than the peak energy. It is entered in the E1 field for 3-point acquisitions.
- **Test Width**—The value in the Test Width column is the width of the acquisition window during 2- and 3-point Test Acquire acquisitions. The midpoint of the acquisition window is the N(E) Peak energy.
- **Swps**—The value in this column is entered in the Number of Sweeps field in the Setup menu.

Name	At.#	N(E) Peak	dN(E) Peak	S_x3/5/10	Acquisition: Lower/Upper		Analysis: Lower/Upper		B1	B2	Test Width	Swps
Ag1*	47	351.5	359	1.000 0.878 0.634	331.5	371.5	334.5	368.5	336	380	80	5
Ag2	47	2573	2583	– 0.008 0.015	2553	2593	2556	2590	2525	2600	244	50
Ag3	47	2746	2755	– 0.003 0.007	2726	2766	2729	2763	2720	2762	222	50
Ag4	47	2374	2381	– 0.003 0.005	2354	2394	2357	2391	2352	2411	216	50
Al1	13	67	70	0.317 0.295 0.176	47	87	50	84	54	73	38	10
Al2*	13	1391	1396	0.075 0.121 0.105	1371	1411	1374	1408	1385	1402	115	15
Ar1*	18	216.5	219	– – –	196.5	236.5	199.5	233.5	209	226	45	20
As1*	33	1225	1229	0.111 0.124 0.110	1205	1245	1208	1242	1198	1237	128	10
As2	33	1260	1264	0.052 0.059 0.053	1240	1280	1243	1277	1253	1272	110	25
As3	33	1114.5	1118	0.062 0.067 0.029	1094.5	1134.5	1097.5	1131.5	1068	1140	154	45
As4	33	92.5	97	0.039 0.028 0.017	72.5	112.5	75.5	109.5	87	103	37	50
Au1	79	71	74	0.629 0.532 0.381	51	91	54	88	64	113	68	5
Au2	79	239.5	243	0.048 0.044 0.033	219.5	259.5	222.5	256.5	210	270	89	40
Au3*	79	2015	2022	0.020 0.041 0.049	1995	2035	1998	2032	1957	2038	217	25
Au4	79	2100	2107	0.011 0.030 0.038	2080	2120	2083	2117	2070	2128	199	35

B: AES Element Table

Name	At.#	N(E) Peak	dN(E) Peak	S_x3/5/10	Acquisition: Lower/Upper		Analysis: Lower/Upper		B1	B2	Test Width	Swps
Au5	79	1764	1771	0.006 0.013 0.015	1744	1784	1747	1781	1700	1782	203	50
Au6	79	1499	1522	0.003 0.007 0.008	1479	1519	1482	1516	1469	1531	167	50
B1*	5	178	185	0.239 0.171 0.105	158	198	161	195	156	193	63	15
Ba1	56	54	58	0.128 0.109 0.076	34	74	37	71	27	107	98	20
Ba2	56	72	76	0.124 0.097 0.065	52	92	55	89	27	107	99	20
Ba3*	56	585.5	603	0.080 0.084 0.071	565.5	605.5	568.5	602.5	519	620	151	20
Ba4	56	669	674	0.013 0.015 0.012	649	689	652	686	620	715	150	50
Be1*	4	103	108	0.270 0.188 0.108	83	123	86	120	85	114	50	10
Bi1*	83	102	105	0.348 0.304 0.212	82	122	85	119	72	136	85	5
Bi2	83	247	253	0.023 0.022 0.017	227	267	230	264	215	279	94	50
Bi3	83	2231	2243	0.004 0.014 0.017	2211	2251	2214	2248	2120	2260	289	50
Bi4	83	2340	2350	– 0.011 0.012	2320	2360	2323	2357	2307	2360	208	50
Bi5	83	1932	1960	– 0.006 0.007	1912	1952	1915	1949	1886	1973	218	50
Br1*	35	1388.5	1393	0.073 0.095 0.103	1368.5	1408.5	1371.5	1405.5	1334	1405	169	15
Br2	35	1434.5	1439	0.045 0.051 0.057	1414.5	1454.5	1417.5	1451.5	1424	1448	125	25
Br3	35	1263	1267	0.025 0.030 0.030	1243	1283	1246	1280	1216	1288	163	45

C: AES Element Table

Name	At.#	N(E) Peak	dN(E) Peak	S_x3/5/10	Acquisition: Lower/Upper		Analysis: Lower/Upper		B1	B2	Test Width	Swps
Br4	35	101	102	0.070 0.055 0.041	81	121	84	118	96	104	29	35
Br5	35	53	55	0.138 0.098 0.074	33	73	36	70	47	57	28	20
C1*	6	266	275	0.165 0.128 0.076	246	286	249	283	243	287	75	20
Ca1*	20	289	297	0.249 0.199 0.132	269	309	272	306	258	304	78	10
Cd1*	48	377	379	1.042 0.934 0.701	357	397	360	394	340	410	108	5
Cd2	48	2683	2694	– 0.006 0.011	2663	2703	2666	2700	2641	2711	246	50
Ce1	58	84.5	87	0.441 0.316 0.196	64.5	104.5	67.5	101.5	37	133	116	5
Ce2*	58	654	671	0.061 0.058 0.043	634	674	637	671	582	694	166	30
Ce3	58	753	774	0.028 0.027 0.019	733	773	736	770	694	803	169	50
Cl1*	17	181.5	184	3.199 2.182 1.724	161.5	201.5	164.5	198.5	174	193	45	5
Co1*	27	773	777	0.300 0.299 0.226	753	793	756	790	751	798	108	5
Co2	27	710	718	0.141 0.142 0.109	690	730	693	727	678	735	115	10
Co3	27	648	658	0.109 0.109 0.084	628	668	631	665	623	678	109	15
Co4	27	53	57	0.459 0.351 0.217	33	73	36	70	41	69	46	5
Cr1	24	489	491	0.344 0.308 0.226	469	509	472	506	463	502	83	5
Cr2*	24	527	531	0.404 0.359 0.265	507	547	510	544	510	542	79	5

B: AES Element Table

Name	At.#	N(E) Peak	dN(E) Peak	S_x3/5/10	Acquisition: Lower/Upper		Analysis: Lower/Upper		B1	B2	Test Width	Swps
Cs1*	55	554	572	0.084 0.083 0.066	534	574	537	571	492	583	139	20
Cs2	55	628	632	0.012 0.012 0.009	608	648	611	645	583	663	133	50
Cu1*	29	918.5	922	0.260 0.307 0.269	898.5	938.5	901.5	935.5	901	932	101	5
Cu2	29	839.5	842	0.080 0.093 0.081	819.5	859.5	822.5	856.5	804	873	134	15
Cu3	29	768	778	0.044 0.051 0.045	748	788	751	785	736	787	112	30
Cu4	29	62	66	0.361 0.297 0.197	42	82	45	79	49	79	49	5
Dy1	66	143.5	154	0.125 0.072 0.043	123.5	163.5	126.5	160.5	36	178	166	30
Dy2*	66	1113	1127	0.025 0.023 0.020	1093	1133	1096	1130	1043	1175	214	50
Dy3	66	954	978	0.019 0.018 0.015	934	974	937	971	877	1016	211	50
Dy4	66	1274	1284	0.012 0.011 0.009	1254	1294	1257	1291	1200	1292	183	50
Er1	68	149	168	0.089 0.067 0.043	129	169	132	166	30	186	180	30
Er2*	68	1211	1228	0.018 0.023 0.021	1191	1231	1194	1228	1129	1278	237	50
Er3	68	1040	1060	0.012 0.015 0.014	1020	1060	1023	1057	954	1104	227	50
Er4	68	1386	1395	0.017 0.022 0.021	1366	1406	1369	1403	1306	1447	239	50
Eu1	63	105	107	0.250 0.169 0.109	85	125	88	122	25	165	161	10
Eu2*	63	844	860	0.037 0.036 0.032	824	864	827	861	765	892	193	40

C: AES Element Table

Name	At.#	N(E) Peak	dN(E) Peak	S_x3/5/10	Acquisition: Lower/Upper		Analysis: Lower/Upper		B1	B2	Test Width	Swps
Eu3	63	137	140	0.145 0.098 0.063	117	157	120	154	117	165	71	20
Eu4	63	974	988	0.037 0.036 0.032	954	994	957	991	916	1025	182	45
F1*	9	655	659	0.499 0.717 0.514	635	675	638	672	642	675	87	5
Fe1*	26	591	600	0.144 0.139 0.103	571	611	574	608	568	616	98	15
Fe2	26	647.5	654	0.212 0.205 0.151	627.5	667.5	630.5	664.5	616	669	107	10
Fe3	26	702.5	705	0.255 0.246 0.178	682.5	722.5	685.5	719.5	685	725	97	10
Fe4	26	48	50	0.670 0.515 0.317	28	68	31	65	35	60	43	5
Ga1*	31	1066	1069	0.246 0.274 0.225	1046	1086	1049	1083	1055	1080	104	5
Ga2	31	1093	1096	0.130 0.137 0.115	1073	1113	1076	1110	1083	1105	103	10
Ga3	31	971	974	0.063 0.073 0.060	951	991	954	988	931	991	133	25
Ga4	31	55	58	0.171 0.130 0.077	35	75	38	72	45	63	36	15
Gd1	64	109	112	0.157 0.131 0.077	89	129	92	126	32	170	160	15
Gd2*	64	1019	1030	0.025 0.031 0.026	999	1039	1002	1036	950	1078	204	50
Gd3	64	139.5	143	0.190 0.153 0.088	119.5	159.5	122.5	156.5	120	170	73	15
Gd4	64	882	896	0.023 0.029 0.024	862	902	865	899	800	933	201	50
Ge1*	32	1146.5	1150	0.140 0.172 0.149	1126.5	1166.5	1129.5	1163.5	1118	1157	123	10

B: AES Element Table

Name	At.#	N(E) Peak	dN(E) Peak	S_x3/5/10	Acquisition: Lower/Upper		Analysis: Lower/Upper		B1	B2	Test Width	Swps
Ge2	32	1177	1181	0.063 0.080 0.070	1157	1197	1160	1194	1171	1190	105	20
Ge3	32	1043.5	1047	0.038 0.046 0.040	1023.5	1063.5	1026.5	1060.5	1000	1068	146	35
Ge4	32	53	55	0.077 0.063 0.040	33	73	36	70	45	60	33	35
Hf1	72	170	184	0.135 0.117 0.072	150	190	153	187	48.5	200	177	20
Hf2*	72	1619	1625	0.047 0.075 0.070	1599	1639	1602	1636	1517	1691	286	20
Hf3	72	1408	1427	0.015 0.024 0.023	1388	1428	1391	1425	1315	1484	268	50
Hf4	72	1216	1231	0.007 0.012 0.012	1196	1236	1199	1233	1115	1286	259	50
Hg1*	80	78	81	0.518 0.427 0.287	58	98	61	95	73.5	95	41	5
Hg2	80	243	246	0.037 0.032 0.023	223	263	226	260	236	249	43	50
Hg3	80	2068	2078	0.018 0.028 0.032	2048	2088	2051	2085	2028	2090	201	40
Hg4	80	2159	2166	0.013 0.019 0.024	2139	2179	2142	2176	2133	2175	187	50
Hg5	80	1811	1818	0.006 0.011 0.012	1791	1831	1794	1828	1760	1825	189	50
Ho1	67	150	162	0.098 0.077 0.046	130	170	133	167	33	183	174	30
Ho2*	67	1161	1177	0.025 0.031 0.026	1141	1181	1144	1178	1085	1227	227	50
Ho3	67	995	1016	0.015 0.019 0.016	975	1015	978	1012	913	1061	223	50
Ho4	67	1329	1339	0.015 0.018 0.015	1309	1349	1312	1346	1254	1386	227	50

C: AES Element Table

Name	At.#	N(E) Peak	dN(E) Peak	S_x3/5/10	Acquisition: Lower/Upper		Analysis: Lower/Upper		B1	B2	Test Width	Swps
I1*	53	506	510	0.269 0.256 0.287	486	526	489	523	448	530	127	5
I2	53	516	519	0.269 0.242 0.256	496	536	499	533	448	530	128	5
I3	53	559.5	562	0.033 0.037 0.037	539.5	579.5	542.5	576.5	535	579	93	35
In1*	49	403	405	0.715 0.586 0.432	383	423	386	420	360	443	122	5
In2	49	2793	2806	– 0.004 0.007	2773	2813	2776	2810	2757	2824	250	50
Ir1	77	163	175	0.082 0.069 0.045	143	183	146	180	100	205	130	30
Ir2*	77	1901	1909	0.028 0.053 0.055	1881	1921	1884	1918	1798	1923	254	25
Ir3	77	1975	1982	0.018 0.040 0.043	1955	1995	1958	1992	1963	2000	171	30
Ir4	77	229	233	0.051 0.044 0.028	209	249	212	246	210	257	76	50
Ir5	77	1665	1672	0.010 0.016 0.016	1645	1685	1648	1682	1542	1760	333	50
Ir6	77	1418	1439	0.004 0.009 0.009	1398	1438	1401	1435	1360	1509	249	50
K1*	19	247	252	0.300 0.255 0.192	227	267	230	264	224	261	67	5
Kr1*	36	1474	1478	– – –	1454	1494	1457	1491	1463	1480	120	20
Kr2	36	1528	1531	– – –	1508	1548	1511	1545	1515	1535	127	20
Kr3	36	1340	1343	– – –	1320	1360	1323	1357	1333	1360	122	20
La1	57	80	83	0.389 0.271 0.168	60	100	63	97	35	119	104	10

B: AES Element Table

Name	At.#	N(E) Peak	dN(E) Peak	S_x3/5/10	Acquisition: Lower/Upper		Analysis: Lower/Upper		B1	B2	Test Width	Swps
La2*	57	619	634	0.081 0.076 0.059	599	639	602	636	550	656	158	25
La3	57	711.5	732	0.027 0.025 0.019	691.5	731.5	694.5	728.5	656	759	161	50
Li1*	3	47	57	0.120 0.070 0.024	27	67	30	64	43	53	28	50
Lu1	71	169	182	0.107 0.086 0.052	149	189	152	186	36	193	182	25
Lu2*	71	1561	1568	0.040 0.057 0.050	1541	1581	1544	1578	1465	1632	276	25
Lu3	71	1359	1378	0.014 0.020 0.017	1339	1379	1342	1376	1268	1434	263	50
Lu4	71	1173	1188	0.009 0.012 0.010	1153	1193	1156	1190	1080	1240	245	50
Mg1	12	46	48	0.381 0.283 0.174	26	66	29	63	25	50	43	10
Mg2*	12	1174	1188	0.098 0.121 0.109	1154	1194	1157	1191	1147	1194	132	10
Mn1*	25	536	545	0.189 0.173 0.122	516	556	519	553	514	558	91	10
Mn2	25	586	592	0.246 0.222 0.161	566	606	569	603	558	605	97	10
Mn3	25	635	638	0.189 0.169 0.123	615	655	618	652	620	655	88	10
Mn4	25	42	45	0.508 0.369 0.226	22	62	25	59	30	53	41	5
Mo1*	42	187	190	0.343 0.271 0.170	167	207	170	204	171	197	52	10
Mo2	42	2037	2044	0.008 0.028 0.031	2017	2057	2020	2054	2000	2058	195	45
Mo3	42	222	225	0.302 0.241 0.149	202	242	205	239	214	232	46	10

C: AES Element Table

Name	At.#	N(E) Peak	dN(E) Peak	S_x3/5/10	Acquisition: Lower/Upper		Analysis: Lower/Upper		B1	B2	Test Width	Swps
Mo4	42	2142	2149	0.003 0.013 0.015	2122	2162	2125	2159	2103	2160	201	50
Mo5	42	1874	1881	0.003 0.009 0.010	1854	1894	1857	1891	1810	1892	209	50
N1*	7	382	389	0.327 0.246 0.161	362	402	365	399	376	398	60	10
Na1*	11	986	996	0.081 0.087 0.076	966	1006	969	1003	959	1005	120	20
Nb1*	41	167.5	170	0.357 0.290 0.190	147.5	187.5	150.5	184.5	158	176	43	5
Nb2	41	1937	1944	0.011 0.026 0.031	1917	1957	1920	1954	1871	1960	220	45
Nb3	41	198.5	201	0.191 0.153 0.100	178.5	218.5	181.5	215.5	186	207	48	15
Nb4	41	2031	2039	0.007 0.012 0.015	2011	2051	2014	2048	1997	2048	188	50
Nb5	41	1762	1787	0.004 0.010 0.011	1742	1782	1745	1779	1711	1799	209	50
Nd1	60	93	96	0.357 0.257 0.166	73	113	76	110	39	159	141	10
Nd2*	60	726	734	0.057 0.056 0.048	706	746	709	743	652	770	177	30
Nd3	60	838	861	0.022 0.022 0.018	818	858	821	855	787	891	169	50
Ne1*	10	818	821	– – –	798	838	801	835	803	824	85	20
Ni1*	28	846	849	0.269 0.281 0.227	826	866	829	863	827	873	112	5
Ni2	28	774	785	0.103 0.107 0.086	754	794	757	791	742	803	122	15
Ni3	28	708	718	0.063 0.066 0.053	688	728	691	725	680	730	107	25

B: AES Element Table

Name	At.#	N(E) Peak	dN(E) Peak	S_x3/5/10	Acquisition: Lower/Upper		Analysis: Lower/Upper		B1	B2	Test Width	Swps
Ni4	28	59	64	0.410 0.321 0.208	39	79	42	76	43	74	50	5
O1*	8	507	510	0.338 0.296 0.212	487	527	490	524	496	525	74	5
Os1	76	166	180	0.102 0.071 0.046	146	186	149	183	92	201	134	30
Os2*	76	1844	1850	0.037 0.054 0.055	1824	1864	1827	1861	1741	1866	251	25
Os3	76	1913	1920	0.024 0.037 0.038	1893	1933	1896	1930	1904	1935	161	35
Os4	76	222	226	0.045 0.031 0.020	202	242	205	239	202	250	76	50
Os5	76	1615	1622	0.010 0.014 0.014	1595	1635	1598	1632	1494	1704	322	50
Os6	76	1377	1396	0.006 0.008 0.008	1357	1397	1360	1394	1269	1462	291	50
P1*	15	120.5	123	0.828 0.613 0.371	100.5	140.5	103.5	137.5	112	128	38	5
P2	15	1855	1862	0.026 0.046 0.049	1835	1875	1838	1872	1848	1867	145	25
Pb1*	82	94	97	0.551 0.473 0.311	74	114	77	111	70	124	75	5
Pb2	82	247	251	0.033 0.032 0.022	227	267	230	264	211	276	95	50
Pb3	82	2176	2188	0.005 0.020 0.023	2156	2196	2159	2193	2057	2205	294	50
Pb4	82	2278.5	2228	– 0.001 0.016	2258.5	2298.5	2261.5	2295.5	2250	2299	201	50
Pb5	82	1885	1914	0.003 0.008 0.009	1865	1905	1868	1902	1850	1925	203	50
Pb6	82	1613	1641	– 0.003 0.004	1593	1633	1596	1630	1569	1650	193	50

C: AES Element Table

Name	At.#	N(E) Peak	dN(E) Peak	S_x3/5/10	Acquisition: Lower/Upper		Analysis: Lower/Upper		B1	B2	Test Width	Swps
Pd1*	46	327.5	333	0.882 0.768 0.547	307.5	347.5	310.5	344.5	310	343	68	5
Pd2	46	275	283	0.152 0.132 0.094	255	295	258	292	254	296	74	15
Pd3	46	2467	2476	– 0.012 0.017	2447	2487	2450	2484	2415	2550	298	50
Pd4	46	2625	2633	– 0.005 0.008	2605	2645	2608	2642	2590	2645	228	50
Pd5	46	2275	2282	– 0.004 0.005	2255	2295	2258	2292	2226	2303	229	50
Pr1	59	88.5	91	0.361 0.266 0.165	68.5	108.5	71.5	105.5	38	148	130	10
Pr2*	59	689	696	0.049 0.049 0.038	669	709	672	706	615	730	171	35
Pr3	59	795	817	0.021 0.020 0.015	775	815	778	812	730	845	178	50
Pt1*	78	64	70	0.513 0.393 0.296	44	84	47	81	56	107	70	5
Pt2	78	170	173	0.043 0.035 0.028	150	190	153	187	107	209	127	50
Pt3	78	236.5	241	0.042 0.034 0.026	216.5	256.5	219.5	253.5	214	266	81	50
Pt4*	78	1962	1969	0.022 0.042 0.051	1942	1982	1945	1979	1859	1984	258	25
Pt5	78	2042	2048	0.017 0.035 0.043	2022	2062	2025	2059	2025	2060	173	30
Pt6	78	1718	1725	0.008 0.012 0.015	1698	1738	1701	1735	1615	1820	323	50
Pt7	78	1462	1484	0.003 0.007 0.008	1442	1482	1445	1479	1433	1492	162	50
Rb1*	37	1554	1561	0.012 0.017 0.022	1534	1574	1537	1571	1495	1574	187	50

B: AES Element Table

Name	At.#	N(E) Peak	dN(E) Peak	S_x3/5/10	Acquisition: Lower/Upper		Analysis: Lower/Upper		B1	B2	Test Width	Swps
Rb2	37	1614	1620	0.006 0.008 0.011	1594	1634	1597	1631	1598	1630	144	50
Rb3	37	1413	1433	0.003 0.005 0.006	1393	1433	1396	1430	1366	1442	176	50
Rb4	37	103.5	107	0.025 0.018 0.013	83.5	123.5	86.5	120.5	82	118	57	50
Rb5	37	73.5	78	0.053 0.040 0.029	53.5	93.5	56.5	90.5	64	82	37	45
Re1	75	163	179	0.156 0.126 0.080	143	183	146	180	81	198	142	15
Re2*	75	1787	1793	0.042 0.072 0.070	1767	1807	1770	1804	1686	1806	242	20
Re3	75	1852	1858	0.025 0.045 0.044	1832	1872	1835	1869	1843	1874	157	30
Re4	75	215	218	0.058 0.046 0.029	195	235	198	232	198	243	73	45
Re5	75	1565	1572	0.012 0.019 0.019	1545	1585	1548	1582	1448	1650	311	50
Re6	75	1336	1354	0.007 0.012 0.011	1316	1356	1319	1353	1228	1417	284	50
Rh1*	45	301	305	0.848 0.725 0.530	281	321	284	318	284	314	63	5
Rh2	45	253	259	0.222 0.191 0.140	233	273	236	270	233	268	65	10
Rh3	45	2356	2366	– 0.011 0.019	2336	2376	2339	2373	2303	2383	236	50
Rh4	45	2500	2507	– 0.005 0.010	2480	2520	2483	2517	2471	2520	214	50
Rh5	45	2172	2180	– 0.004 0.006	2152	2192	2155	2189	2120	2191	216	50
Ru1*	44	274	277	0.574 0.495 0.302	254	294	257	291	256	287	62	5

C: AES Element Table

Name	At.#	N(E) Peak	dN(E) Peak	S_x3/5/10	Acquisition: Lower/Upper		Analysis: Lower/Upper		B1	B2	Test Width	Swps
Ru2	44	230	235	0.216 0.186 0.114	210	250	213	247	211	245	63	10
Ru3	44	2248	2256	– 0.019 0.023	2228	2268	2231	2265	2191	2272	231	50
Ru4	44	2376	2385	– 0.009 0.011	2356	2396	2359	2393	2356	2395	197	50
Ru5	44	2038	2078	– 0.006 0.006	2018	2058	2021	2055	2024	2088	201	50
S1*	16	149.5	153	1.277 1.042 0.652	129.5	169.5	132.5	166.5	145	158	37	5
S2	16	2111	2119	0.013 0.023 0.030	2091	2131	2094	2128	2082	2125	185	45
Sb1*	51	455	458	0.738 0.704 0.525	435	475	438	472	405	513	150	5
Sb2	51	3022	3035	– 0.003 0.007	3002	3042	3005	3039	2968	3054	282	50
Sc1*	21	337	343	0.305 0.250 0.168	317	357	320	354	300	349	84	10
Sc2	21	368	370	0.322 0.264 0.176	348	388	351	385	359	380	58	10
Se1*	34	1306	1311	0.074 0.097 0.032	1286	1326	1289	1323	1272	1320	141	40
Se2	34	1347	1352	0.038 0.045 0.016	1327	1367	1330	1364	1338	1360	118	50
Se3	34	1188	1192	0.020 0.027 0.008	1168	1208	1171	1205	1140	1213	159	50
Se4	34	97	104	0.031 0.024 0.010	77	117	80	114	88	110	43	50
Se5	34	44	47	0.027 0.022 0.024	24	64	27	61	38	56	36	50
Si1	14	93	96	0.414 0.403 0.393	73	113	76	110	77	101	45	5

B: AES Element Table

Name	At.#	N(E) Peak	dN(E) Peak	S_x3/5/10	Acquisition: Lower/Upper		Analysis: Lower/Upper		B1	B2	Test Width	Swps
Si2*	14	1615	1621	0.034 0.069 0.071	1595	1635	1598	1632	1609	1628	131	20
Sm1	62	100.5	103	0.214 0.160 0.104	80.5	120.5	83.5	117.5	25	164	160	15
Sm2*	62	801	814	0.037 0.038 0.031	781	821	784	818	724	848	187	45
Sm3	62	120.5	135	0.109 0.078 0.047	100.5	140.5	103.5	137.5	110	164	76	30
Sm4	62	927	940	0.026 0.028 0.022	907	947	910	944	864	984	191	50
Sn1*	50	429	432	0.688 0.643 0.465	409	449	412	446	382	474	133	5
Sn2	50	2908	2919	– 0.003 0.006	2888	2928	2891	2925	2860	2939	268	50
Sr1*	38	1640	1651	0.016 0.023 0.027	1620	1660	1623	1657	1579	1663	197	50
Sr2	38	1706	1718	0.007 0.009 0.011	1686	1726	1689	1723	1665	1728	180	50
Sr3	38	1495	1516	0.005 0.006 0.007	1475	1515	1478	1512	1440	1523	188	50
Sr4	38	110	114	0.057 0.040 0.029	90	130	93	127	95	122	49	45
Ta1	73	168	183	0.175 0.145 0.100	148	188	151	185	56	193	162	15
Ta2*	73	1674	1680	0.046 0.075 0.080	1654	1694	1657	1691	1573	1750	292	15
Ta3	73	1454	1475	0.015 0.024 0.026	1434	1474	1437	1471	1361	1539	280	50
Ta4	73	1255	1271	0.008 0.012 0.013	1235	1275	1238	1272	1152	1331	269	50
Tb1	65	114	116	0.120 0.067 0.040	94	134	97	131	31	175	166	35

C: AES Element Table

Name	At.#	N(E) Peak	dN(E) Peak	S_x3/5/10	Acquisition: Lower/Upper		Analysis: Lower/Upper		B1	B2	Test Width	Swps
Tb2*	65	1066	1078	0.030 0.027 0.021	1046	1086	1049	1083	992	1125	212	50
Tb3	65	142	150	0.147 0.079 0.045	122	162	125	159	31	175	168	30
Tb4	65	918	937	0.028 0.023 0.018	898	938	901	935	836	974	208	50
Tb5	65	1222	1230	0.012 0.009 0.007	1202	1242	1205	1239	1150	1273	211	50
Te1*	52	482	486	0.477 0.437 0.337	462	502	465	499	429	503	118	5
Th1*	90	66.5	69	0.537 0.435 0.286	46.5	86.5	49.5	83.5	53	107	73	5
Th2	90	246.5	249	0.069 0.066 0.049	226.5	266.5	229.5	263.5	242	258	46	25
Th3	90	2620	2634	– 0.005 0.007	2600	2640	2603	2637	2542	2654	284	50
Th4	90	2778	2789	– 0.003 0.005	2758	2798	2761	2795	2710	2800	272	50
Th5	90	2286	2296	– 0.002 0.003	2266	2306	2269	2303	2210	2307	249	50
Ti1	22	383	390	0.326 0.274 0.188	363	403	366	400	358	397	77	5
Ti2*	22	419	421	0.518 0.438 0.296	399	439	402	436	410	430	60	5
Tl1*	81	86.5	90	0.584 0.531 0.332	66.5	106.5	69.5	103.5	70	110	60	5
Tl2	81	245	250	0.032 0.031 0.020	225	265	228	262	211	273	92	50
Tl3	81	2122	2132	0.009 0.024 0.026	2102	2142	2105	2139	2000	2148	290	50
Tl4	81	2218	2227	0.006 0.017 0.018	2198	2238	2201	2235	2193	2260	215	50

B: AES Element Table

Name	At.#	N(E) Peak	dN(E) Peak	S_x3/5/10	Acquisition: Lower/Upper		Analysis: Lower/Upper		B1	B2	Test Width	Swps
Tl5	81	1838	1865	0.005 0.009 0.009	1818	1858	1821	1855	1721	1877	281	50
Tl6	81	1575	1601	– 0.004 0.004	1555	1595	1558	1592	1508	1611	213	50
Tm1*	69	154	170	0.119 0.085 0.057	134	174	137	171	35	192	181	25
Tm2*	69	1444	1452	0.033 0.040 0.038	1424	1464	1427	1461	1360	1508	250	35
Tm3	69	1260	1278	0.022 0.024 0.024	1240	1280	1243	1277	1175	1331	247	50
Tm4	69	1083	1100	0.015 0.017 0.016	1063	1103	1066	1100	995	1149	234	50
U1*	92	74.5	77	0.888 0.654 0.452	54.5	94.5	57.5	91.5	61	130	88	5
U2	92	282	284	0.191 0.166 0.132	262	302	265	299	275.5	300	56	10
U3	92	2755	2764	– 0.004 0.008	2735	2775	2738	2772	2685	2789	284	50
U4	92	2930	2940	– 0.004 0.007	2910	2950	2913	2947	2952	2952	191	50
U5	92	2404	2414	– 0.003 0.004	2384	2424	2387	2421	2378	2426	207	50
V1	23	432	440	0.281 0.247 0.177	412	452	415	449	404	449	86	10
V2*	23	472	475	0.445 0.387 0.272	452	492	455	489	459	486	70	5
W1	74	168	182	0.175 0.142 0.091	148	188	151	185	74	195	146	15
W2*	74	1730	1737	0.050 0.081 0.080	1710	1750	1713	1747	1628	1810	301	15
W3	74	1502	1524	0.017 0.024 0.023	1482	1522	1485	1519	1403	1593	295	50

C: AES Element Table

Name	At.#	N(E) Peak	dN(E) Peak	S_x3/5/10	Acquisition: Lower/Upper		Analysis: Lower/Upper		B1	B2	Test Width	Swps
W4	74	1295	1312	0.010 0.014 0.014	1275	1315	1278	1312	1190	1373	276	50
W5	74	207	210	0.036 0.028 0.019	187	227	190	224	196	235	66	50
Xe1*	54	534	537	– – –	514	554	517	551	518	552	81	20
Y1	39	130.5	132	0.157 0.127 0.075	110.5	150.5	113.5	147.5	120	136	39	20
Y2*	39	1739	1748	0.014 0.029 0.029	1719	1759	1722	1756	1676	1762	205	45
Y3	39	1815	1823	0.007 0.013 0.013	1795	1835	1798	1832	1790	1833	167	50
Y4	39	1583	1606	0.004 0.008 0.008	1563	1603	1566	1600	1535	1616	191	50
Yb1	70	162	174	0.094 0.068 0.052	142	182	145	179	28.5	190	186	25
Yb2*	70	1501	1511	0.033 0.042 0.046	1481	1521	1484	1518	1411	1570	264	30
Yb3	70	1309	1329	0.013 0.016 0.018	1289	1329	1292	1326	1224	1381	251	50
Yb4	70	1130	1141	0.009 0.011 0.012	1110	1150	1113	1147	1038	1197	242	50
Zn1*	30	993	997	0.257 0.296 0.278	973	1013	976	1010	945	1008	138	5
Zn2	30	905.5	908	0.078 0.088 0.083	885.5	925.5	888.5	922.5	868	943	144	15
Zn3	30	828	839	0.037 0.042 0.038	808	848	811	845	793	845	117	35
Zn4	30	61	64	0.328 0.261 0.183	41	81	44	78	55	70	34	5
Zr1*	40	148.5	151	0.369 0.298 0.204	128.5	168.5	131.5	165.5	136	155	43	5

B: AES Element Table

Name	At.#	N(E) Peak	dN(E) Peak	S_x3/5/10	Acquisition: Lower/Upper		Analysis: Lower/Upper		B1	B2	Test Width	Swps
Zr2	40	1836	1844	0.018 0.036 0.043	1816	1856	1819	1853	1809	1857	173	30
Zr3	40	1921	1929	0.006 0.016 0.020	1901	1941	1904	1938	1897	1940	173	50
Zr4	40	1672	1695	0.007 0.012 0.015	1652	1692	1655	1689	1621	1706	200	50