


5800 MultiTechnique ESCA Operator's Guide

Part No. 647268 Rev. A

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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 volts,
- Has read and understood the PHI technician's manual for the equipment,
- Observes and understands all safety notices on PHI equipment.

The safety symbols that PHI uses are defined in the following pages.* 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: (1) the hazard, (2) the possible result of ignoring the notice, and (3) how to avoid the hazard.

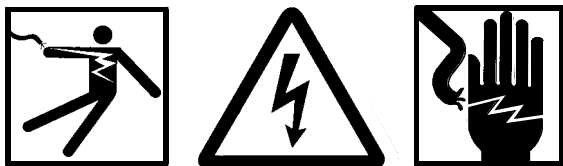
The three signal words are defined as follows:

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

* 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.

PHI Safety Notices



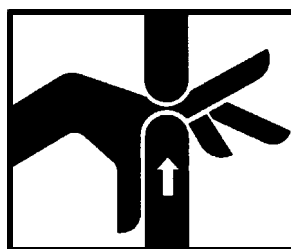
High voltages may be present that could cause personal injury or death.



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



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



A pinching point is present that could cause personal injury.



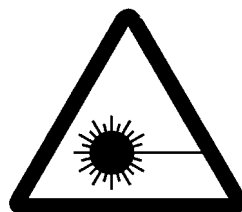
Extremely low temperatures may be present that could cause personal injury.



A potentially dangerous magnetic field may be present.



Hot surfaces may be present that could cause personal injury.

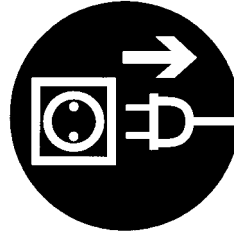


Visible or invisible radiation may be present that could cause personal injury.

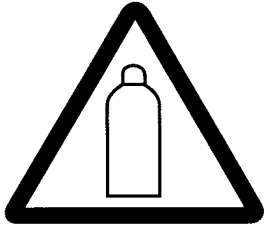
PHI Safety Notices



Heavy lifting could cause personal injury.



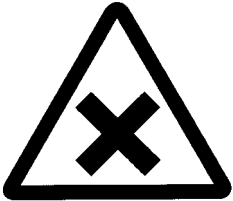
Pulling the plug from its power source before servicing is mandatory.



Contents are under pressure.



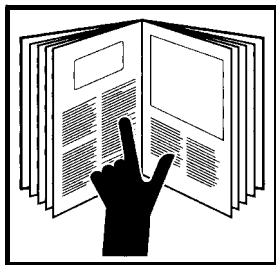
Turning off the power switch before servicing is mandatory.



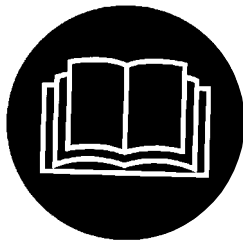
A harmful or irritant material may be present that could cause personal injury.



Wearing protective gloves is mandatory.



Refer to the manual(s) before proceeding.

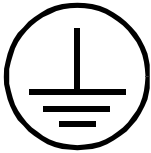


Wearing eye protection is mandatory.

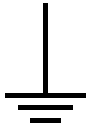
PHI Safety Notices



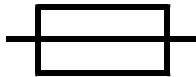
Wearing foot protection is mandatory.



This is the location of the protective grounding conductor terminal.



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



This is the location of the fuse.

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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

The manual is divided into three sections. Section 1 describes the 5800 system, how to turn the system on and off, and how to start the software. Sections 2 explains how to introduce and setup a sample for analysis. Section 3 describes the different types of data acquisition and some aspect of the associated data reduction.

The 5800 MultiTechnique XPS system is available in two physical configurations known as the 5801 and 5802. The 5801 is a single console system. The 5802 is a dual console system that is used when a scanning electron gun for Auger electron spectroscopy is added to the system. The separate vacuum console provides the vibration isolation devices needed for high spatial resolution scanning Auger microscopy. Drawings in the back of the manual show the front, back, and top views of both 5800 MultiTechnique systems. Below is a photograph of the 5802 system.



Photograph of the 5802 dual console MultiTechnique XPS system.

1: Introduction

The 5800 is the latest model in the 5000 series of Physical Electronics' (PHI's) MultiTechnique XPS systems. The 5800 has a new vacuum chamber layout to accommodate a new ion gun, and electron neutralizer. The new Model 06-350 ion gun has been optimized for sputter depth profiling and provides a new capability of showering samples with low energy (~5 eV) ions to facilitate charge neutralization. The new Model 04-091 XPS Neutralizer (electron flood gun) uses a low temperature cathode that provides high electron flux and a narrow energy distribution. Other improvements include new software driven control units for the ion gun (11-066), x-ray source (50-096), and ion pumps (DIGITEL MPC).

System Options

Optional accessories for the 5800 include:

- X-ray monochromator (Al anode with toroidally-shaped, quartz single crystals)
- Static SIMS (quadrupole-based optics and associated electronics)
- Scanning Auger Microscopy (1000Å electron gun and associated electronics)
- Ion Scattering Spectroscopy (dual polarity analyzer electronics kit and gas manifold for ion gun)
- Automated sample stage (X, Y, Z, rotation, and tilt)
- Vacuum/inert atmosphere transfer vessel
- Hot/cold sample stage
- Fracture attachment
- Chemical reactor vessel

Please contact your sales representative for information about these optional accessories.

Assumptions for This Operator's Guide

This discussion assumes you are using a system equipped as follows:

- A 5801 XPS system, configured with the optional Model 10-610 Monochromator X-ray Source and the 04-091 XPS Neutralizer.
- Manual (non-motorized) five axis sample stage.
- Auto(mated) Valve Control
- Differentially-pumped ion gun
- Magnetic Introduction System

If your system is configured differently, the PC-ACCESS menus displayed may occasionally exhibit options not discussed in this manual.

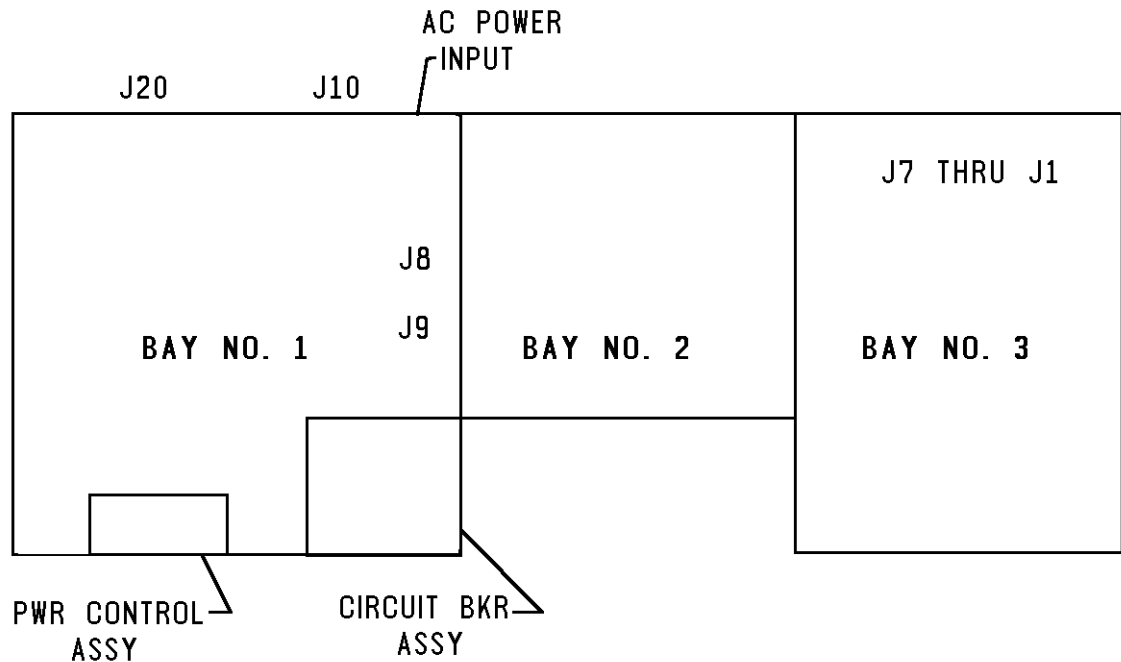
1: Introduction

Other assumptions of this Operator's Guide are

- The system is in good working order.
- The sample test chamber is under vacuum (UHV).
- The system is on, aligned, and calibrated.
- The PHI PC-ACCESS (Version 7.1A or higher) and MultiPak (Version 5.0A or higher) software packages are installed and running.

System Power and Emergency Off

The 5800 system is typically on 24 hours a day and not powered on and off daily. Each system contains a circuit breaker panel, a power control panel, and the emergency power off switch. The individual circuit breakers and power control panel allow individual segments of the instrument console to be turned on and off for servicing purposes. In the event of a hazardous occurrence or emergency, the entire system can be shut off by depressing the Emergency Off button.



Top view of 5801 single console system showing the location of the circuit breaker panel and the power control panel.

1: Introduction

Emergency
Off button—
part of Model
32-005

Circuit
breaker panel

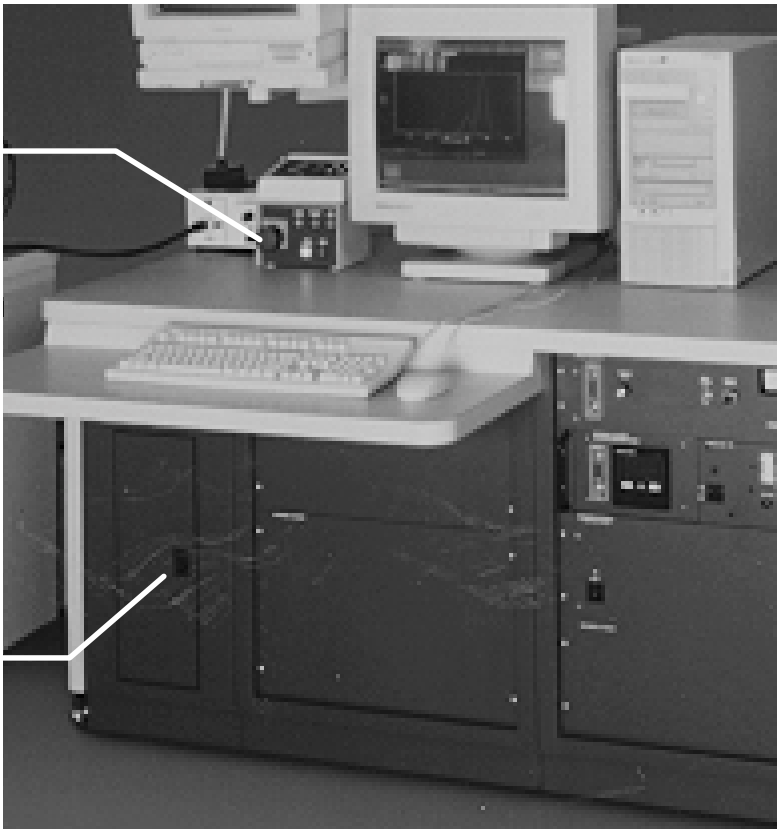


Front view of 5801 system console showing the location of the circuit breaker panel and the emergency off button.

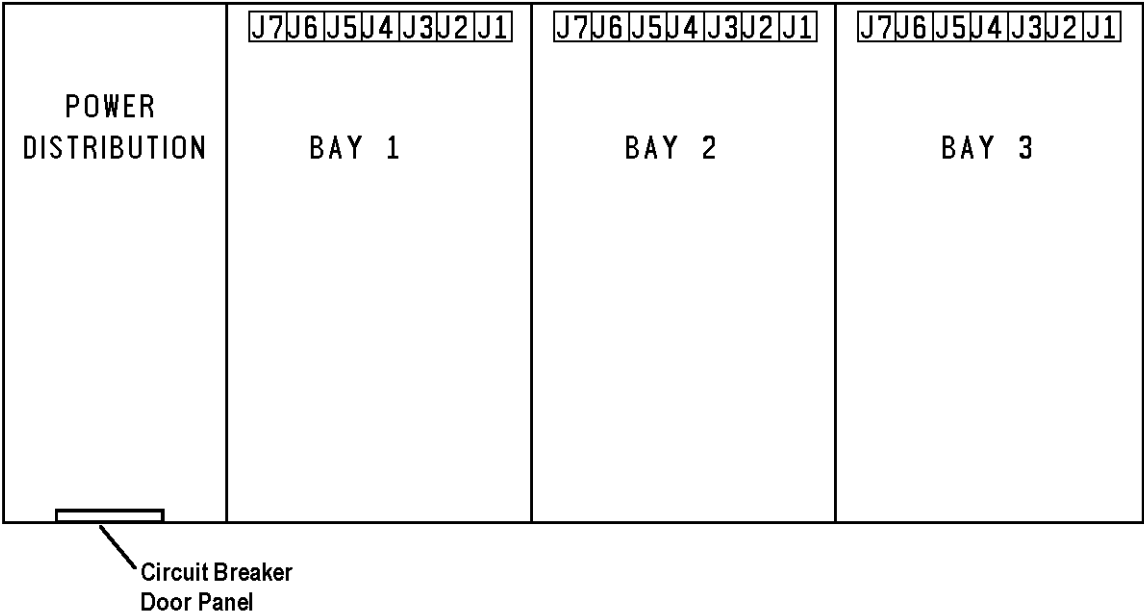
1: Introduction

Emergency Off
Button

Circuit
Breaker panel

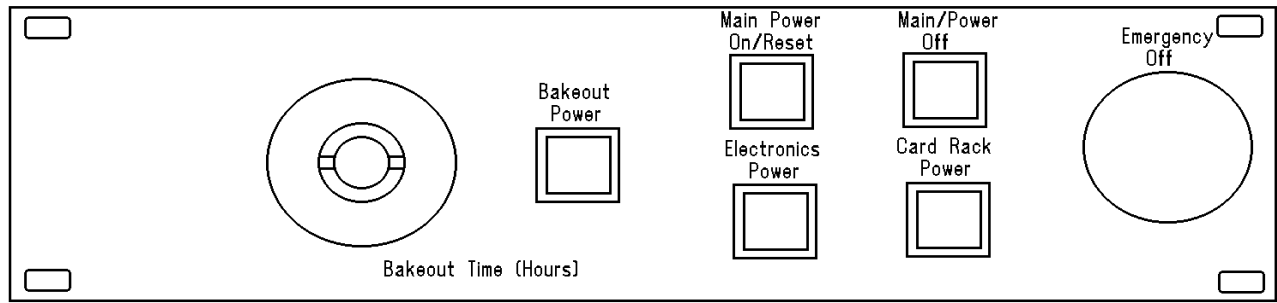


Front view of 5802 electronics console showing the location of the circuit breaker panel and the emergency off button.

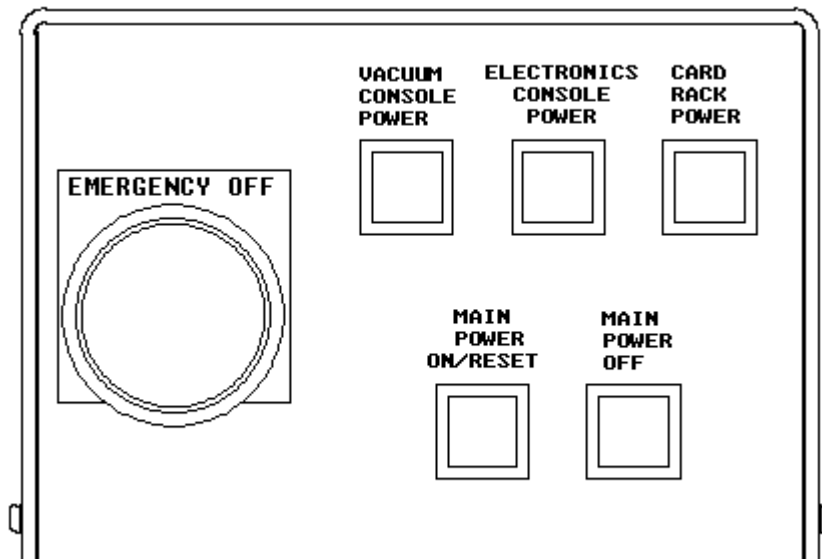


Top view of 5802 electronics console showing the location of the circuit breaker panel and the power control panel.

1: Introduction



Model 32-005 with Emergency Off switch used on the 5801 single console system.



Emergency Off Box used on the 5802 dual console system.

Power Interruption

In the event of a power interruption, the system will remain in an off state and wait for you to restore power. The following steps should be performed to restore power:

1. Press the Main Power On/Reset button on the power control panel.
2. The DIGITEL MPC should automatically restart. If the system pressure is too high for the ion pumps to restart, the control will switch to the “cool down” mode. If this occurs, there has probably been a loss of vacuum during the power interruption. It will be necessary to pump the main chamber with the turbo-molecular pump to restore vacuum.
3. Press the on switch on the Digital Gauge Control.

1: Introduction

4. Press the water pump start switch on the front panel of the 50-096 X-ray source control to start the water pump for the X-ray source.
5. Log onto the computer and start the PHI PC-ACCESS and MultiPak software packages.

Daily Shutdown of Optics

Several electron/x-ray optics devices in the 5800 systems use hot filaments: the dual anode x-ray source, the monochromator x-ray source, the ion gun, and the electron neutralizer. The x-ray sources are typically run in an automated mode and are shut off at the end of an analysis sequence. The ion gun and electron neutralizer typically idle in a standby mode, where the filament is on, but accelerating voltages are off. At the end of the day (if your analysis is complete), these devices should be turned off by selecting the off buttons in the ion gun and neutralizer software menus.

PC-ACCESS Startup

On the Microsoft Windows NT Workstation Version 4.0 desktop double-click, with the left mouse button, on the PHI PC-ACCESS icon. This will open three windows: the PC-ACCESS window, the ion gun window and the electron neutralizer window.

The PC-ACCESS window contains a series of pull-down menu buttons at the top of the window that can be operated with the left mouse button or by function keys (F1-F10) on the keyboard. Individual items from a pull-down can be selected using the left mouse button or the arrow keys on the keyboard.

1: Introduction



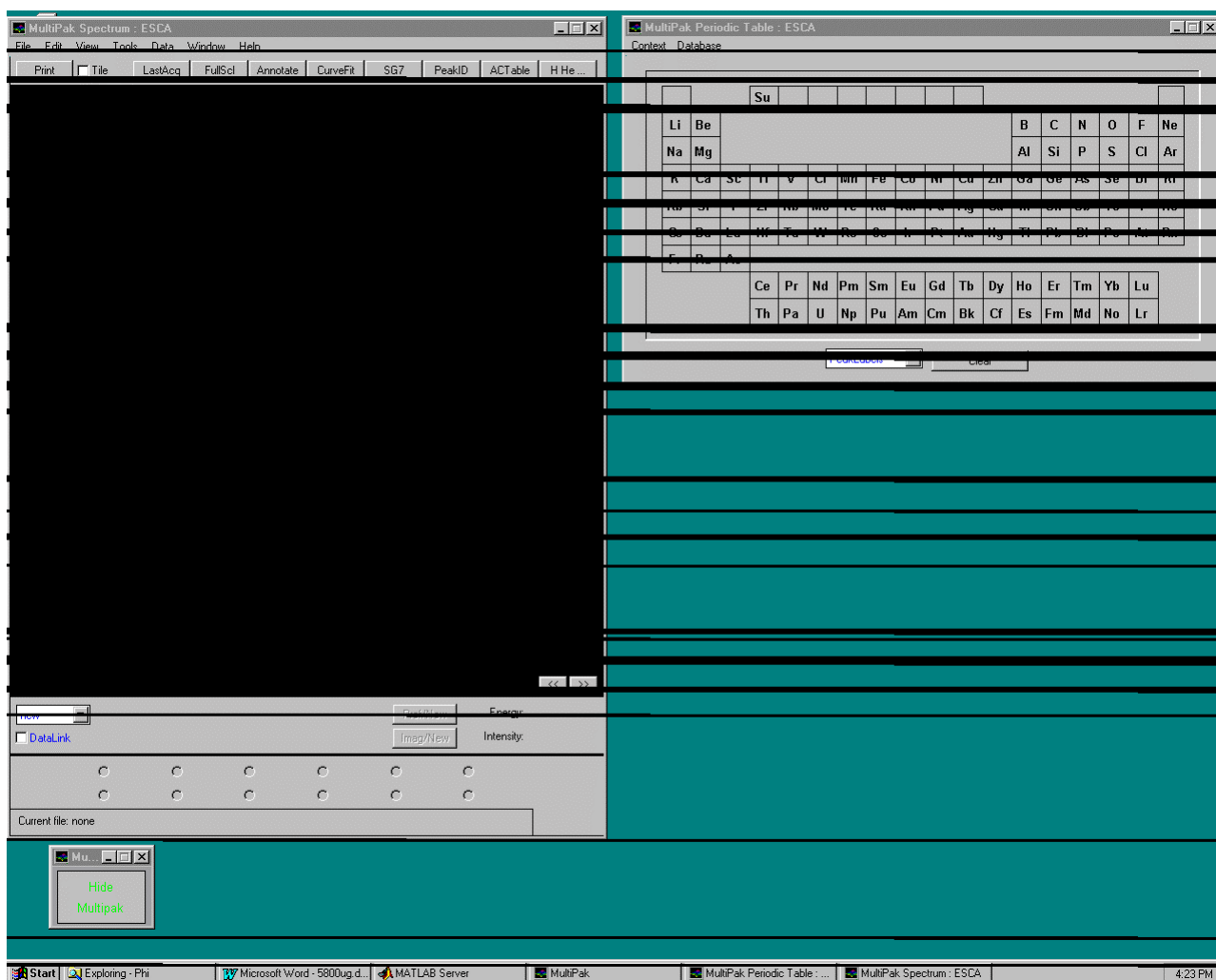
The PHl PC-ACCESS main window uses a series of pull-down buttons to select functions.

The PC-ACCESS software is shipped with a ‘standard’ layout for the functions associated with each button. However, the user can re-arrange the functions and rename the buttons, if desired, to make a custom layout, and save it. To modify the layout or “command banks”, under the System Control pull down, select the Command Settings function, make the desired changes and save them.

MultiPak Startup

On the Windows NT desktop, double-click with the left mouse button on the MultiPak icon. This will open three windows: the MultiPak Spectrum window, the MultiPak Periodic Table, and an icon that has the function of hiding or displaying the MultiPak windows. PC-ACCESS also contains a data reduction capability, but MultiPak is Physical Electronics’ electron spectroscopy data reduction platform for the development of new data reduction features and capabilities.

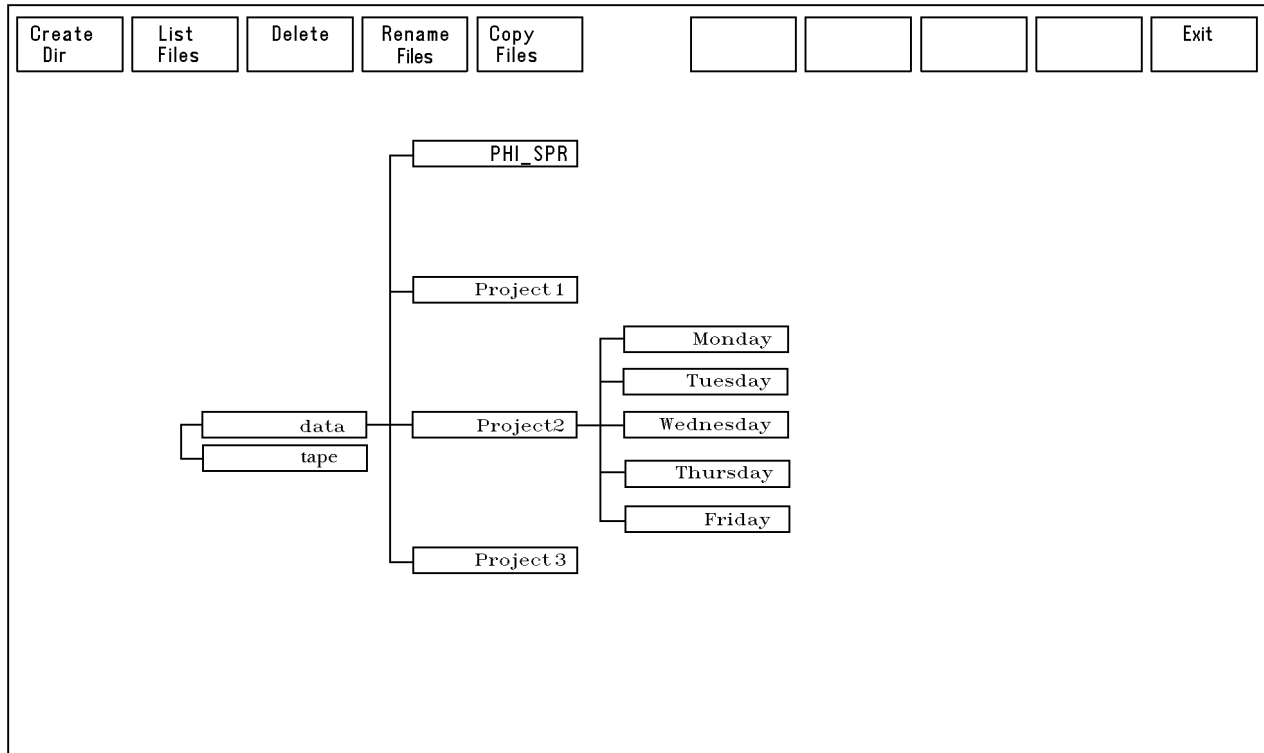
1: Introduction



PHI MultiPak windows open on the Windows NT desktop.

File Management in PC-ACCESS

PC-ACCESS has its own file manager. To create a folder for data storage select “Dir” under the Load -> Display pull down. This will open a window that contains the directory tree.

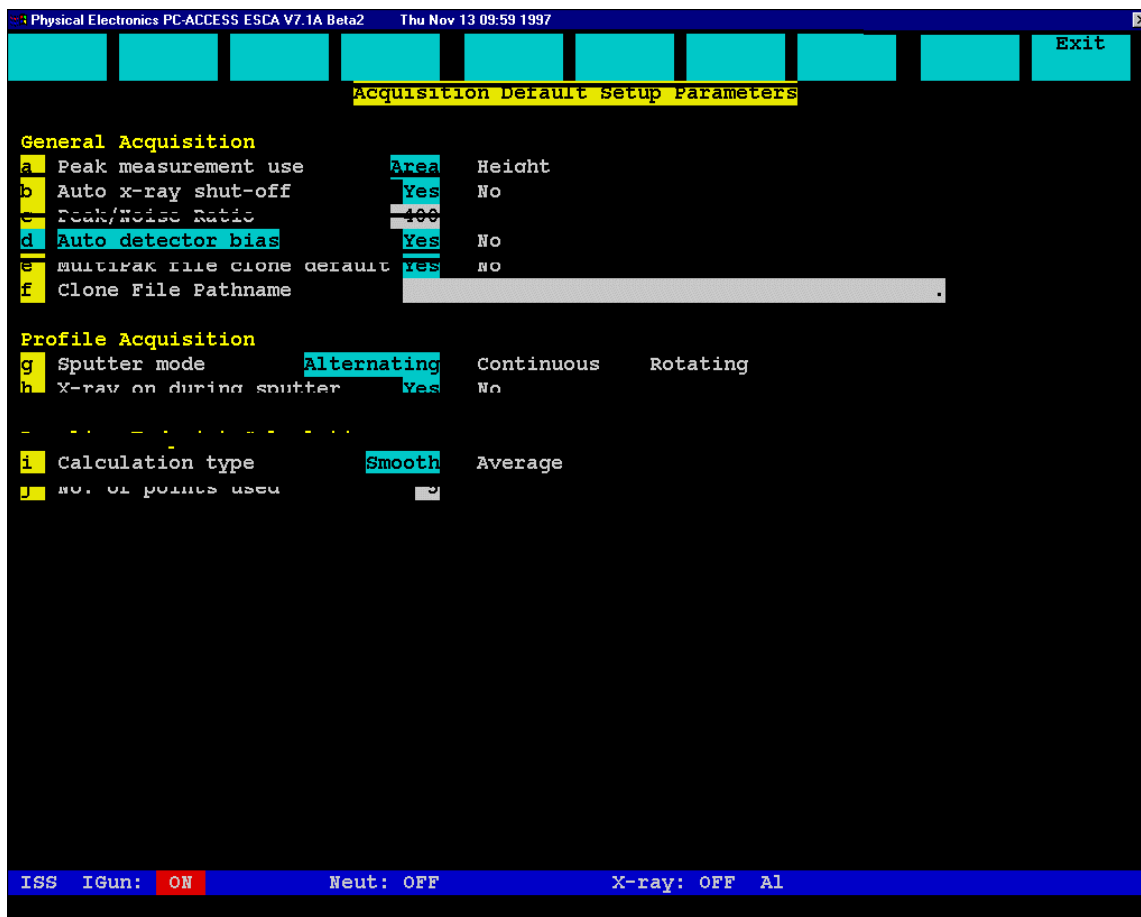


PC-ACCESS Directory window.

From the directory window you can create a folder or sub-directory to store data for a specific experiment or task. The lowest level folder is named ‘data’ which represents the internal hard disk of the system PC. To select an existing folder click on it using the left mouse key or scroll using the arrow keys on the keyboard. The currently selected folder will change its fill color to white. Other functions in this window include: a delete function, a list file function, and the ability to copy and rename files.

PC-ACCESS automatically saves data files in the ACCESS format and can also be setup to automatically save a MultiPak clone data file, which is recommended for ease-of-use of MultiPak. To activate this feature, under the Semiperm(anent) Menus pull down, select the item Acq. Params. and in the Acq. Params. menu select yes on line ‘e’.

1: Introduction

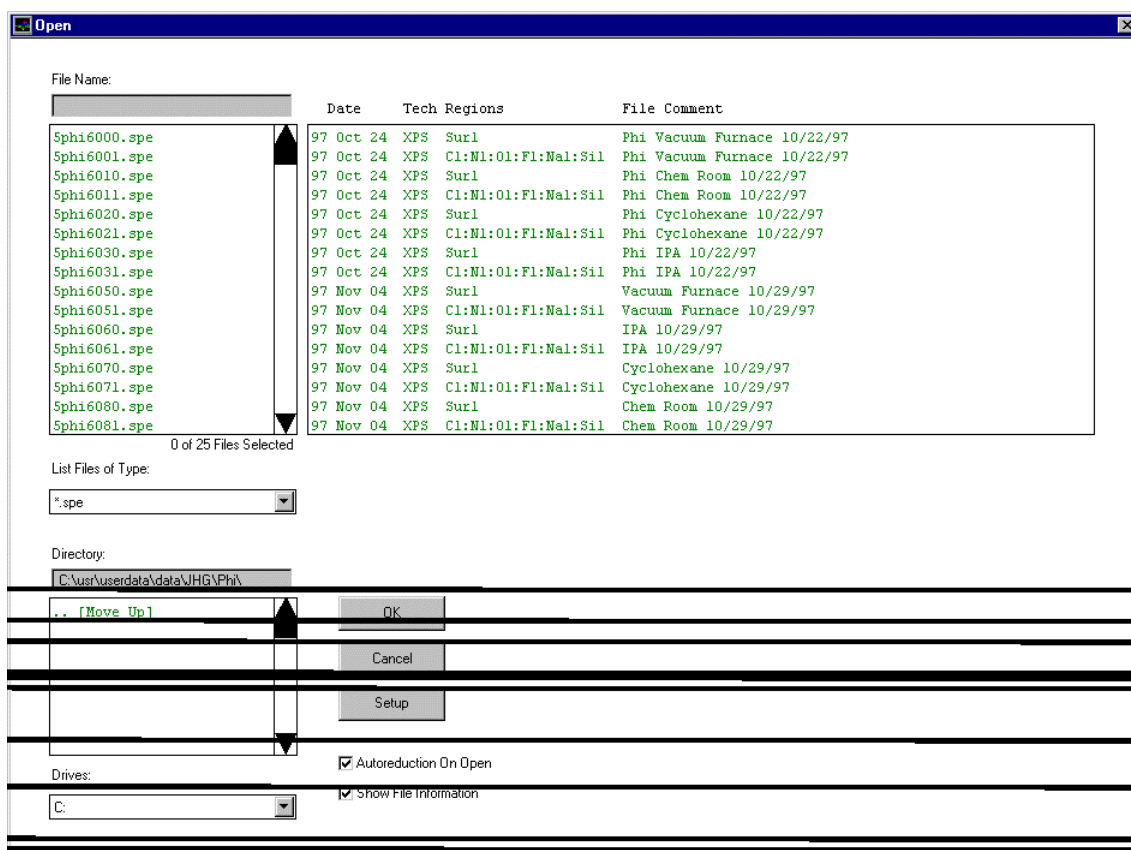


The Acq(uisition) Parm.s. menu is located under the Semiperm(anent) Menus pull down. Selecting yes in line 'e' will trigger the automatic generation of MultiPak clone files.

File Management in MultiPak

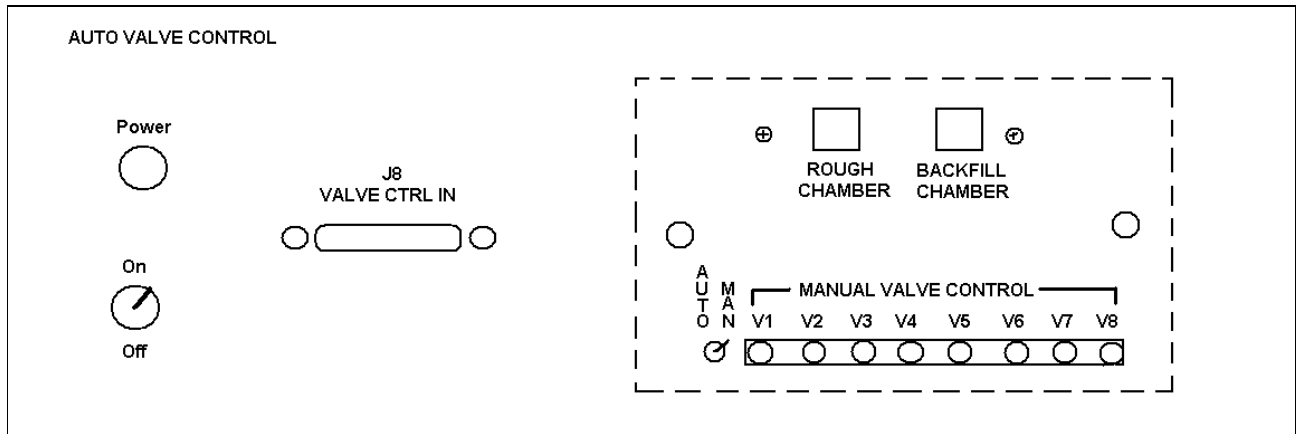
To open a data file or group of data files in MultiPak, click on 'File' in the menu bar and select 'open' from the pull down menu. This will open the MultiPak file manager. From the file manager, you can select a data directory and a data file or group of files to be opened for manipulation. The mouse interaction is very similar to that of other MS Windows-based software applications.

1: Introduction



The MultiPak file manager tool is used to select a data file or group of files for manipulation in MultiPak.

Section 2: Sample Introduction



Auto Valve Control for 5800 Systems. Note a face plate usually covers the area enclosed by the dotted line. This face plate is sometimes removed by service personnel when performing manual valve control.

Sample introduction is accomplished with a combination of software and operator driven activities:

1. Press the Backfill Intro button on the Auto Valve Control's remote keypad.
(Note: Refer to your Auto Valve Control manual for a picture of your system's Auto Valve Remote Keypad face plate.)
2. When the intro chamber is backfilled, insert the sample mount into the intro., replace the cover and press the Pump Intro button.
3. When the pump down is completed, as indicated by all five red lights being lit on the TC1 display of the Auto Valve control, press the Intro Sample button and push the intro. rod into the main chamber and transfer the sample holder onto the sample stage.
4. When the transfer is complete, withdraw the intro. rod. V1 will close automatically and isolate the main vacuum chamber from the intro. chamber.

Sample Positioning

Assuming the optical microscope has been previously aligned to the focal point of the analyzer input lens,* turn on the fiber optic light source and view the sample holder on the video monitor. Using the knobs on the sample stage, position the desired analysis area in the center of the field of view and adjust the stage so the analysis area is in focus. With the desired analysis area in focus and located at the center of the field of view, you can be relatively confident that you are analyzing the correct area of the sample. Positioning errors using this method are typically less than 100 μm . If you are using the smallest analysis area of 30 μm and higher precision is required, it is suggested that you collect an ESCA map and select the desired area from the map data. (Maps are discussed in more detail in Section 3 of this manual.)

Prior to collecting ESCA data, it is recommended that you create or select the desired directory for data storage and that you select the Sample Setup function under the Sample Setup pull down. In this screen, you can define a prefix for the data file, a starting file number, and comments to be stored with the data file.

X-Ray Source Selection

Most systems have a dual anode, non-monochromatic x-ray source and a monochromatic Al K α x-ray source. For most ESCA analysis, Physical Electronics recommends using the monochromatic x-ray source. For the following reasons:

- The 10-610 x-ray source generates x-rays very efficiently and provides good sensitivity.
- A monochromator filters out undesired high energy x-rays, x-ray satellites, and narrows the Al K α lines, greatly reducing x-ray-induced sample damage.
- The filtered Al K α beam eliminates x-ray satellites, eliminating some peak overlaps and, in general, simplifies the spectra and peak identification.
- The filtered Al K α beam provides a narrower x-ray energy distribution, increasing the energy resolution and chemical sensitivity of the instrument.

There may be situations where a peak overlap exists between a photoelectron peak and an Auger peak. Changing the x-ray energy can eliminate this overlap. This would be the most common reason to use the non-monochromatic x-ray source.

* Refer to the *5800 System Hardware Manual* for detailed procedural instructions.

2: Sample Introduction

Note: Alignment of the system and sample is critical for optimum performance when using the monochromatic x-ray source.*

The x-ray source type is chosen in the Setup X-ray menu or in the Acquisition Setup menus. The monochromatic x-ray source has two filaments: a 2 mm filament and a 7 mm filament. Physical Electronics recommends using the 2 mm filament, because this will provide the highest sensitivity. Note: the new charge neutralization system in the 5800 will effectively neutralize insulating samples under these conditions.

Note: When first turned on, the Model 50-096 X-ray source power supply needs to be programmed to operate at 15 keV. To do this, perform the following steps:

1. Press the Local button under Control Select.
2. Press the Start button for the Water Pump.
3. Press the High Voltage button above the keypad.
4. Press the Display/Enter Setpoints button above the keypad. (LED should light.)
5. Press 1 - 5 - 0 - # on the keypad.
6. Press the remote button under Control Select.

The 50-096 will retain the 15 keV setting until it is manually turned off or there is a power interruption.

Selecting the Size of the Analysis Area

The x-ray sources of the 5800 flood a relatively large area of the sample with x-rays, and the size of the analysis area is determined by the energy analyzer input lens. The user has control of two variables that effect the size of the analysis area. The first is the size of the aperture in the analyzer input lens. A rotary motion located on the side of the analyzer input lens is turned to select one of five apertures. Aperture 1 is the smallest and 5 is the largest. The aperture also must be selected in the Analyzer/Detector menu to apply the correct lens voltage for the selected analysis area.

The second variable is the relative magnification of the input lens. Three magnifications are available: large (1 X), small (1/3 X) and minimum (1/5 X).

* Refer to the *5800 System Hardware Manual* for detailed procedural instructions.

2: Sample Introduction

The control of lens settings (voltages) is performed in the Analyzer/Detector menu, which is under the System Control pulldown. In this menu, you can select the lens magnification and the correct lens constants for a specific aperture. You must also manually select the correct physical aperture.

The screenshot displays the 'Analyzer/Detector Parameters' window of the Physical Electronics PC-ACCESS ESCA V7.1A Beta2 software. The window has a title bar with the software name and version, and a timestamp of 'Thu Nov 13 13:54 1997'. The main area is divided into several sections. At the top, there are buttons for 'Detector', 'Single Channel', 'Position Sensitive', and 'Multiple Channel'. Below these are buttons for 'Input lens' (Standard, Extended, Omni I, Omni II, Omni III, Omni IV, Omni V) and 'Omni Focus lens area' (Small, Large, Minimum). The 'Aperture' section shows a grid of buttons for 1, 2, 3, 4, and 5. The 'ISS scattering angle (Deg)' is set to 134.8. The 'Deflection Amplitude Gain' is set to 2090. There are two sections for calibration positions: 'First Calibration Position' and 'Second Calibration Position'. Each section has buttons for 'Kinetic energy', 'X Position', and 'Y Position'. The 'ESCA I Gun' is set to 'ON', 'Neut' is 'OFF', and 'X-ray' is 'OFF'. The 'Al' button is also visible.

Parameter	Value
Detector	Multiple Channel
Input lens	Omni II
Omni Focus lens area	Minimum
Aperture	4
ISS scattering angle (Deg)	134.8
Deflection Amplitude Gain	2090
First Calibration Position	
Kinetic energy	1
X Position	0
Y Position	0
Second Calibration Position	
Kinetic energy	1
X Position	0
Y Position	0
ESCA I Gun	ON
Neut	OFF
X-ray	OFF
Al	

The analyzer input lens magnification and lens constants for each aperture are selected in the Analyzer/Detector menu.

The size of the analysis area for a given combination of aperture and analyzer input lens magnification is shown in the following table.

2: Sample Introduction

Analysis area for a given combination of aperture and analyzer input lens magnifications.

Aperture #	Analysis Area Size		
	Minimum	Small	Large
1	30 μm diameter	-----	-----
2	120 μm diameter	180 μm diameter	0.4 mm diameter
3	400 μm diameter	600 μm diameter	2 mm diameter
4	800 μm diameter	1100 μm diameter	4 mm diameter
5	0.6 x 2.0 mm	1 x 3.5 mm	3 x 10 mm

Physical Electronics recommends using the minimum mode for most analysis and selecting as large an aperture as possible for the sample that you have, in order to maximize sensitivity and minimize data collection time. For Auger analysis, Physical Electronics recommends using either Aperture 4 or 5 along with the small or minimum lens area magnification.

Selecting a Pass Energy

Selecting an appropriate pass energy is an important part of an ESCA experiment. The pass energy determines the energy that detected photoelectrons will have as they pass through the hemispherical analyzer. The lower this energy is, the better the energy-resolving capability of the analyzer is. However, as pass energy is lowered to improve energy resolution, the intensity of the detected photoelectron peaks is reduced. This relationship is nearly linear (when the pass energy is reduced by a factor of two, so is the detected count rate). The following figure shows the pass energies and data step sizes that are available in the Survey, Utility and High Resolution modes of operation.

2: Sample Introduction

Physical Electronics PC-ACCESS ESCA V7.1A Beta2 Tue Dec 16 11:28 1997

Resolution Menu

Survey Mode

Fixed Analyzer Transmission

a	Pass Energy	46.95	93.90	187.85	
b	eV/step	0.200	0.400	0.800	1.600
c	Time/Step	20			

Fixed Retard Ratio

d	FRR Energy Resolution (%E)	0.10	0.20	0.40	0.60	0.80
---	----------------------------	------	------	------	------	------

Utility Mode

Fixed Analyzer Transmission

e	Pass Energy	29.35	58.70	117.40	
f	eV/step	0.125	0.250	0.500	1.000
g	Time/Step	50			

Fixed Retard Ratio

h	FRR Energy Resolution (%E)	0.10	0.20	0.40	0.60	0.80
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High Resolution

Fixed Analyzer Transmission

i	Pass Energy	2.95	5.85	11.75	23.50	46.95
j	eV/step	0.025	0.050	0.100	0.200	
k	Time/Step	100				

Fixed Retard Ratio

l	FRR Energy Resolution (%E)	0.10	0.20	0.40	0.60	0.80
---	----------------------------	------	------	------	------	------

Alignment Acquisition Times

m	Manual Time/Step	1000
n	Refresh Time/Step	10

5<=PARAMETER<=1000

ESCA IGun: OFF Neut: OFF X-ray: OFF Al

The Resolution Menu is accessed by selecting Resol. Select under the Acquisition Setup pulldown.

When elemental information is of primary interest, such as in survey spectra, a high pass energy and a large data step size should be used. When detailed chemical information is being sought, a lower pass energy and smaller data step sizes should be used. By selecting a pass energy that is no lower than necessary to resolve the ESCA peaks of interest, you will maintain as high a count rate as possible and improve the efficiency of your experiments.

Section 3:

Data Acquisition and Reduction

This section details the five types of data acquisition (survey, multiplex, depth profile, angle profiles, and maps) and the most commonly used data reductions for those types.

Survey Spectra

The purpose of collecting survey spectra is to determine or confirm the elemental surface composition of the area being analyzed. Since the focus of survey spectra is to obtain elemental information, the instrumental conditions are adjusted to maximize elemental sensitivity. To accomplish this, survey spectra are typically collected using the highest pass energy available (187 eV) and a minimal number of data points (typically 0.8 or 1.6 eV/step). Sensitivity increases linearly with pass energy and is also effected by the number of data points collected.

Another part of the strategy for survey spectra is to use the largest analysis area possible, while confining the analysis area to the feature of interest. Time is also a variable in the collection of a survey. However, the quality of the spectra increases relative to the square root of time. Therefore, it is more efficient to optimize the other critical parameters before adjusting the Time/step value (when focusing on sensitivity).

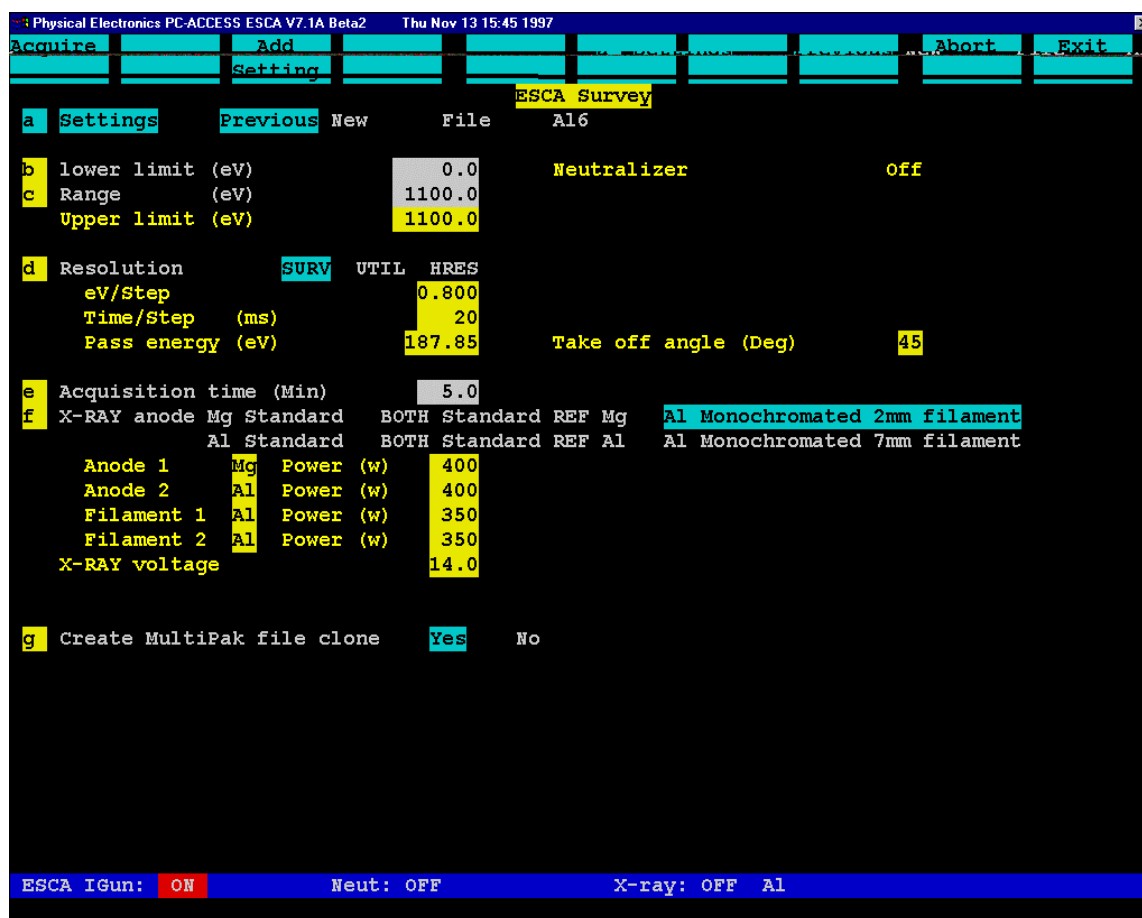
To collect a survey spectrum, perform the following steps:

1. Insert the sample into the system and position the analysis area at the focal point of the optical microscope.
2. Determine the required analysis area, select it in the Analyzer/Detector menu, and select the required aperture using the knob the on side of the analyzer input lens.
3. Choose the directory where the data should be stored (create a new sub-directory if necessary).
4. In the Sample Setup menu, enter a filename and comments about the sample.
5. Select Setup Survey from the Acquisition Setup pulldown.
6. Item 'a' in the ESCA Survey setup menu will allow the previous settings, a previous data file or new settings to be defined.
7. Enter the desired data collection time, typically five minutes or less using aperture #4 (800 μm analysis area).
8. Select the desired x-ray source, typically the Al monochromator using the 2 mm filament.

3: Data Acquisition and Reduction

9. Select Acquire.

Note: Information appearing in yellow boxes is provided for information purposes only and cannot be changed.



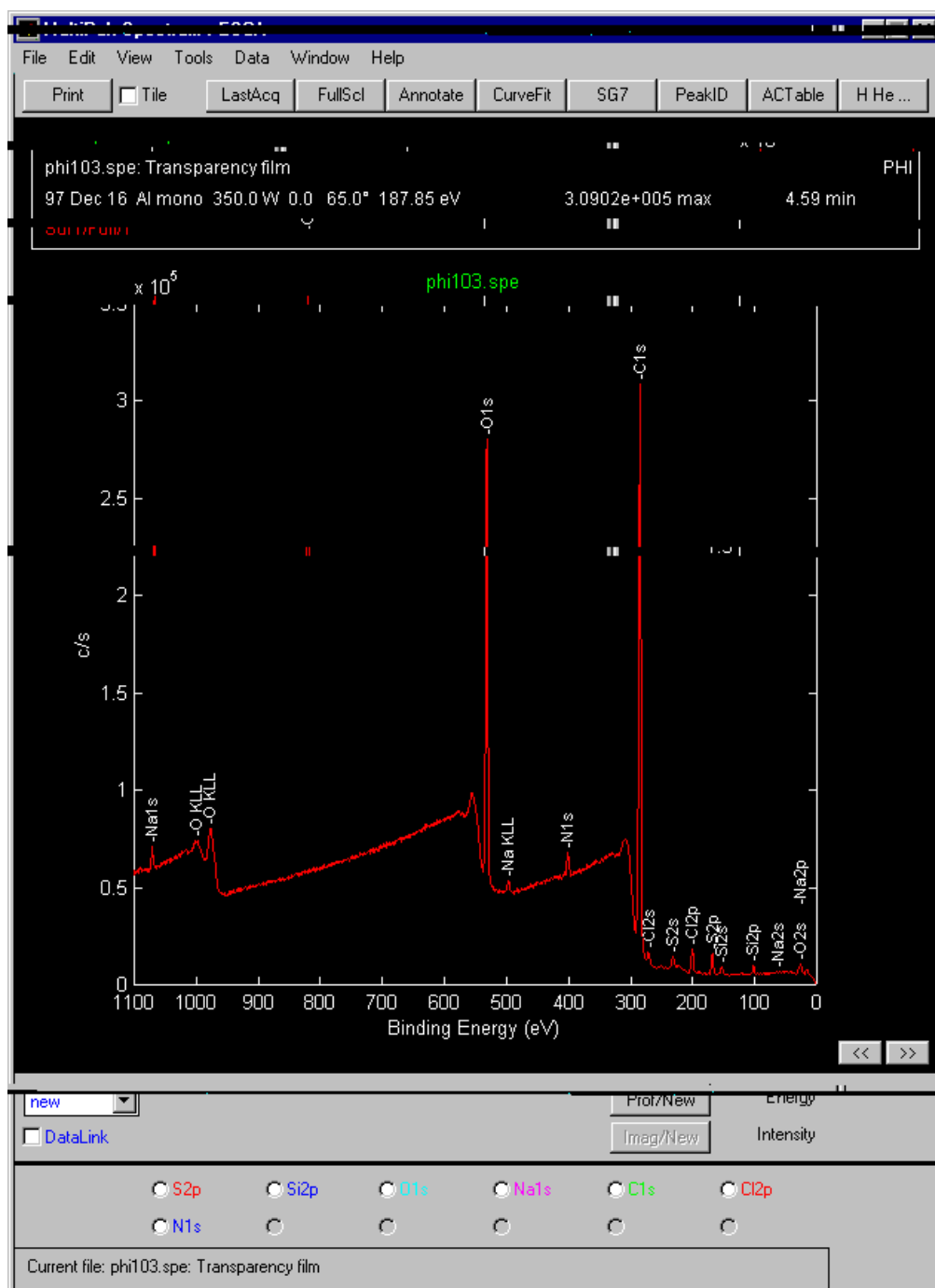
A typical survey acquisition setup with a high pass energy and coarse data step size, designed to maximize elemental sensitivity.

When the survey acquisition has finished, you can display the data in MultiPak (Physical Electronics' data reduction package) by selecting the LastAcq button or by loading the data file from MultiPak's file menu. When the file is displayed, select the PeakID button to label the peaks in the spectrum. After MultiPak has labeled the spectrum, you should verify the accuracy of the labels using your experience and the *Handbook of X-ray Photoelectron Spectroscopy*.

If mistakes or omissions were made, you can correct them with one or more of the software tools that are available for peak labeling. To remove a label, click on it and drag it outside the window using the left mouse button. Another approach to remove an unwanted peak label is to turn OFF the element in MultiPak's Periodic Table by toggling it off with the left mouse button. To label a peak or element that was missed by the Auto Peak ID routine, you can toggle

3: Data Acquisition and Reduction

that element ON in the Periodic Table using the left mouse button. When an element is selected automatically or by manually toggling it in the periodic table, a region button for that element is displayed below the spectrum in the MultiPak spectrum window.



Survey spectrum displayed in MultiPak after Auto Peak ID has been performed.

3: Data Acquisition and Reduction

If you do not know the identity of an unlabelled peak, you can manually search through the database of peak labels. Using the right mouse button, click and hold on an open area of the spectrum. This will display a cursor and a list of ESCA transitions that occur at the current cursor location. You can then drag the cursor across a peak and examine the peak labels in the vicinity of the peak of interest. When you release the mouse button, a label will be placed on the spectrum.

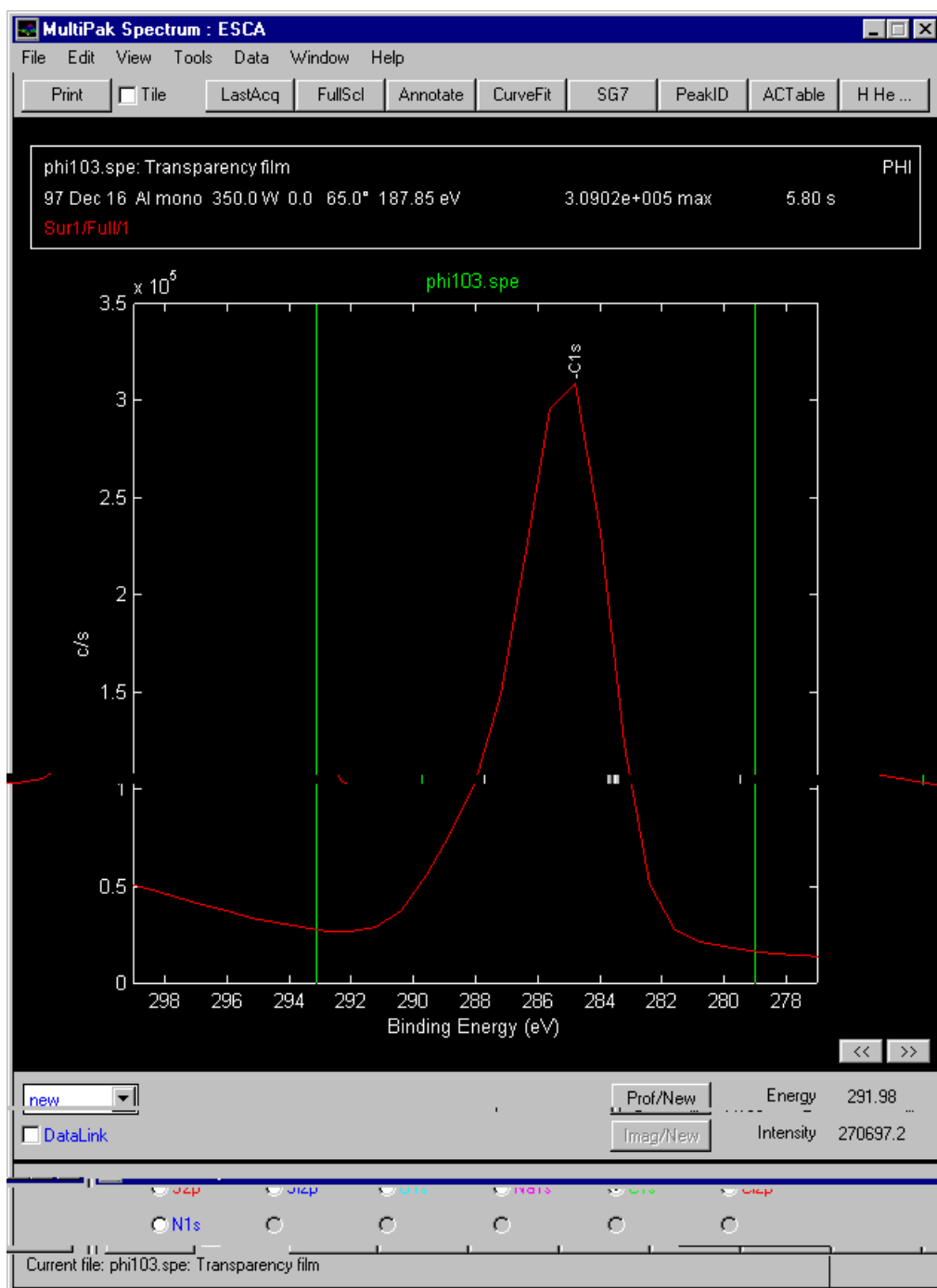
If you wish to search the peak label database and not leave any labels on the spectrum, hold down the shift key and press the left mouse button on a clear area of the spectrum to display the cursor. The search action will be the same but no labels will be placed on the spectrum when you release the mouse button. // The same search action will result, however peak labels will not display when you release the mouse button.

Note: When labeling peaks using the right mouse button, no region buttons for the newly labeled elements will be created. To create a region button for a newly identified element, toggle that element ON in the periodic table using the left mouse button.

When the peak identification process is complete, the next step in data reduction is to quantify the elements that were detected. Quantification of ESCA spectra is most accurate when peak area measurements are used. Several methods are available to remove the background and make a peak area measurement. The Shirley background is recommended in most situations. To view or change the endpoints that will be used to define the background subtraction for a specific element, press the Region button for that peak and adjust the cursors by clicking and dragging on them with the left mouse button. While the cursor is being moved, a line depicting the background will be displayed to assistance in positioning the cursors.

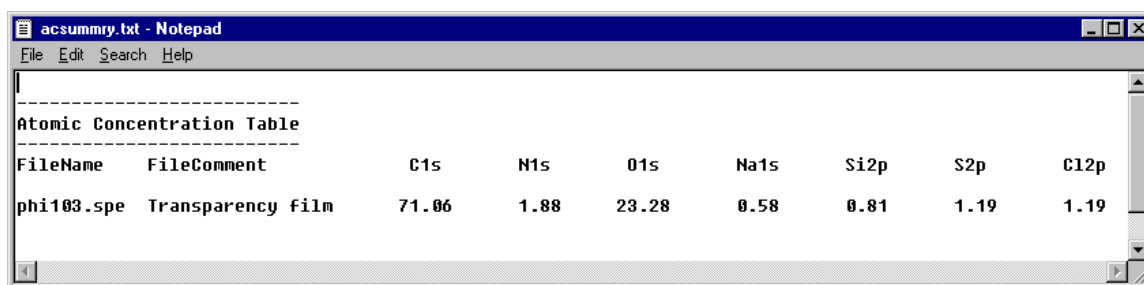
Note: The default peak for an element and the default location of the cursors is stored in the Transition Editor menu. If you wish to change the default settings you can do so in this menu. For a more detailed discussion of this subject, see the *MultiPak Software Manual*.

3: Data Acquisition and Reduction



Expanded view of the carbon 1s line showing the vertical cursors used for background subtraction. The C1s button was selected with the left mouse button to create this display.

When the cursors for all the regions have been adjusted, an atomic concentration table can be displayed by pressing the ACTable button in the upper tool bar. For detailed information about formatting, printing, and exporting AC tables, see the *MultiPak Software Manual*.



The screenshot shows a Notepad window with the title 'acsummary.txt - Notepad'. The menu bar includes 'File', 'Edit', 'Search', and 'Help'. The text content is as follows:

Atomic Concentration Table								
FileName	FileComment	C1s	N1s	O1s	Na1s	Si2p	S2p	Cl2p
phi103.spe	Transparency film	71.06	1.88	23.28	0.58	0.81	1.19	1.19

The Atomic Concentration table may be displayed, printed, or exported to other desktop software packages.

Multiplex Spectra

Multiplex spectra have a narrow energy range that encompasses a peak of interest. The narrow energy range allows you to optimize either spectral detail or sensitivity, while keeping the data collection time short.

To optimize spectral detail, a low pass energy is chosen (typically 10 - 30 eV) and the number of data points is increased (0.05 - 0.2 eV/step). There is a temptation to select a very low pass energy and a very small step size to obtain the 'best' data. In practice, a compromised choice must be made between experiment time, pass energy, and data step size. For an efficient, high-energy resolution experiment, you should use as high a pass energy as possible, while still resolving the spectral features of interest and include the fewest number of data points possible, while recording an accurate shape of the spectrum.

To optimize elemental sensitivity, a high pass energy should be chosen (typically 50 - 200 eV) and the number of data points is reduced (0.2 - 0.4 eV/step). Since sensitivity increases almost linearly with pass energy, it is very advantageous to use as high a pass energy as possible.

Whether you are trying to optimize spectral detail or sensitivity, you should select as large an analysis area as possible, while restricting the analysis to the feature of interest. This will provide the highest count rate possible and minimize the analysis time.

To collect a multiplex spectrum, perform the following steps:

1. Collect a survey spectrum to determine or confirm which elements are of interest.
2. Select Setup Multiplex from the Acquisition Setup pulldown.
3. Item 'a' in the ESCA Multiplex setup menu will allow the previous settings, a previous data file, or new settings to be defined.
4. In line 'c' enter the name of the element/transition you would like analyzed. The name for the major transition of most elements is the element symbol followed by the number '1'. If you do not know the name of a specific ESCA

3: Data Acquisition and Reduction

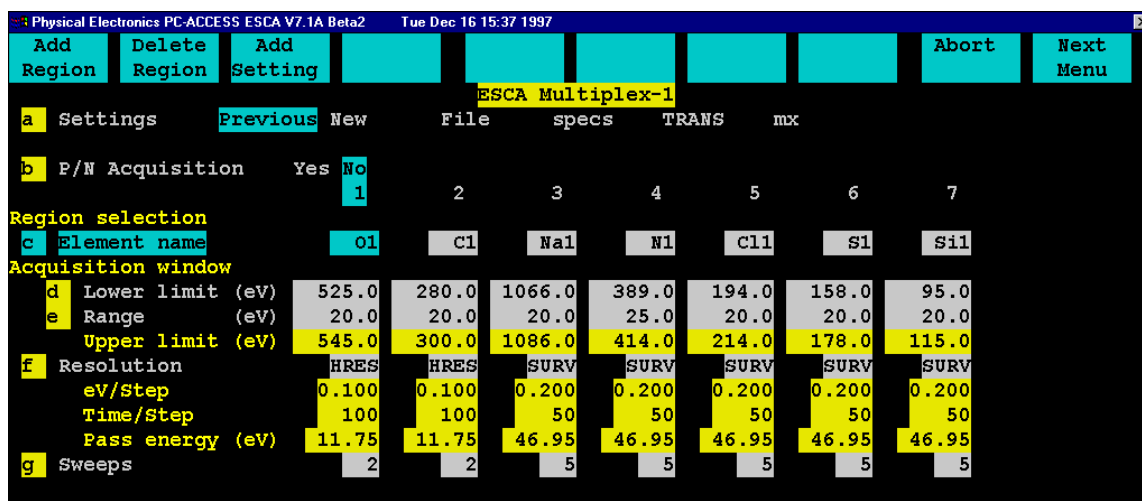
peak, enter just the element symbol and the PC-ACCESS software will display a list of valid element symbols for that element.

5. Enter the desired data collection time.
6. Select the desired x-ray source and anode, typically the Al monochromator using the 2 mm filament.
7. Select Acquire.

If you wish to customize the setup of a multiplex acquisition, you can

- add elements,
- change the selected peak for an element,
- modify the energy range of an element,
- change the pass energy of a specific element, or
- change the number of sweeps for an element.

You can use this customization capability to optimize an experiment. For example, if you would like high resolution spectra of a few major elements, select a pass energy of 11.75 eV for those elements; and for minor elements where the interest is sensitivity and quantification, you can select a higher pass energy. The MultiPak data reduction software is capable of reducing data files that have mixed pass energies and providing correct quantitative results.



The first page of the Multiplex Setup Menu showing the selected regions. Note, the use of different pass energies for various elements to optimize the efficiency of the experiment.

3: Data Acquisition and Reduction

Physical Electronics PC-ACCESS ESCA V7.1A Beta2 Tue Dec 16 15:39 1997

Buttons: Add Setting, Previous Menu, Abort, Exit

ESCA Multiplex-2

Current Previous

a Acquisition time (Min) 30.0

b X-RAY anode Mg Standard BOTH Standard REF Mg Al Monochromated 2mm filament
Al Standard BOTH Standard REF Al Al Monochromated 7mm filament

Anode 1	Mg	Power (w)	400		
Anode 2	Al	Power (w)	300	Neutralizer	off
Filament 1	Al	Power (w)	350		
Filament 2	Al	Power (w)	200		

X-RAY voltage 14.0

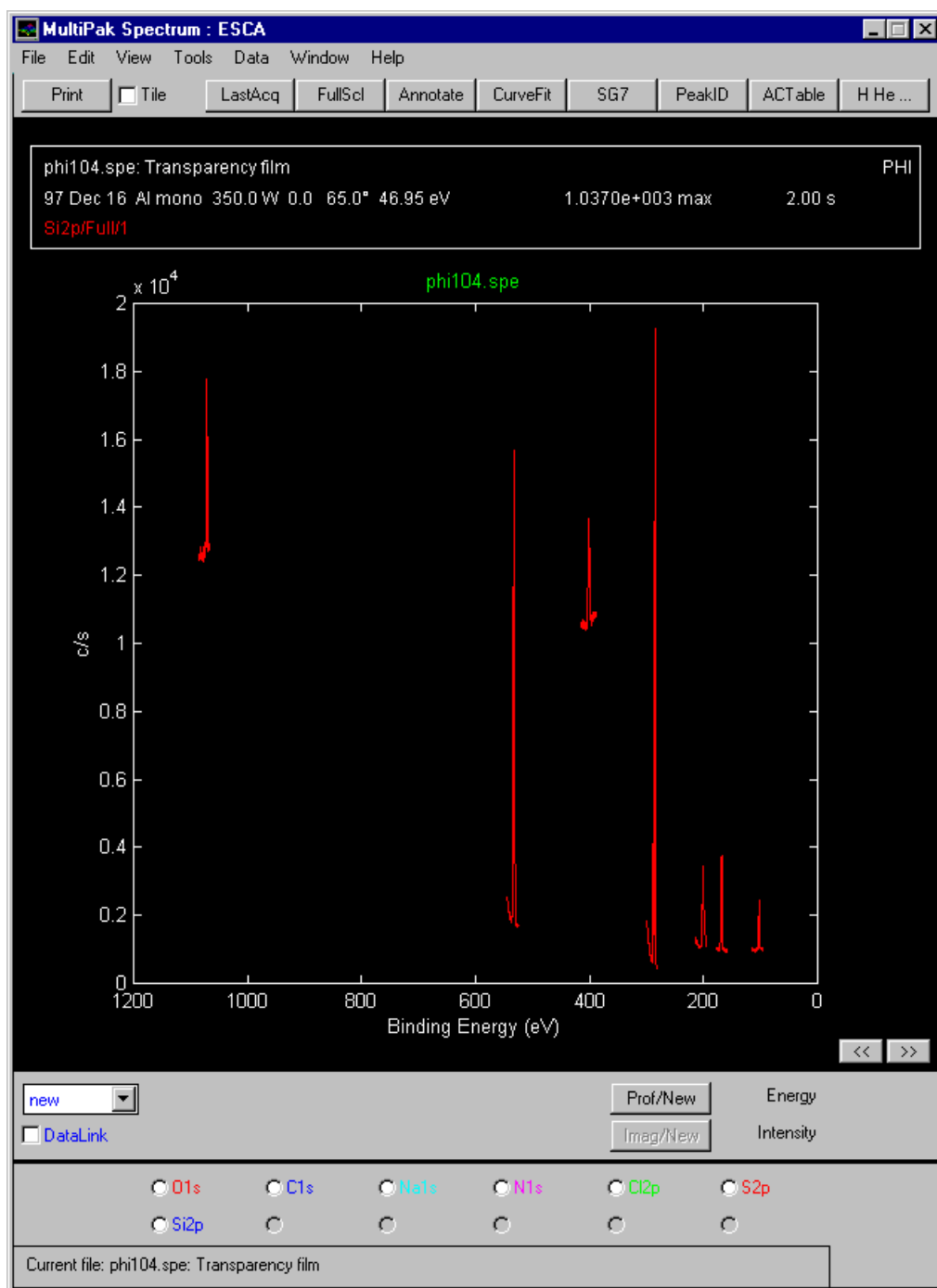
Take off angle (Deg) 65

c Create MultiPak file clone Yes No

The second page of the Multiplex Setup menu showing the acquisition time and x-ray source selection information.

When the acquisition is finished, display the data in MultiPak by selecting the LastAcq button or by loading the data file from MultiPak's file menu.

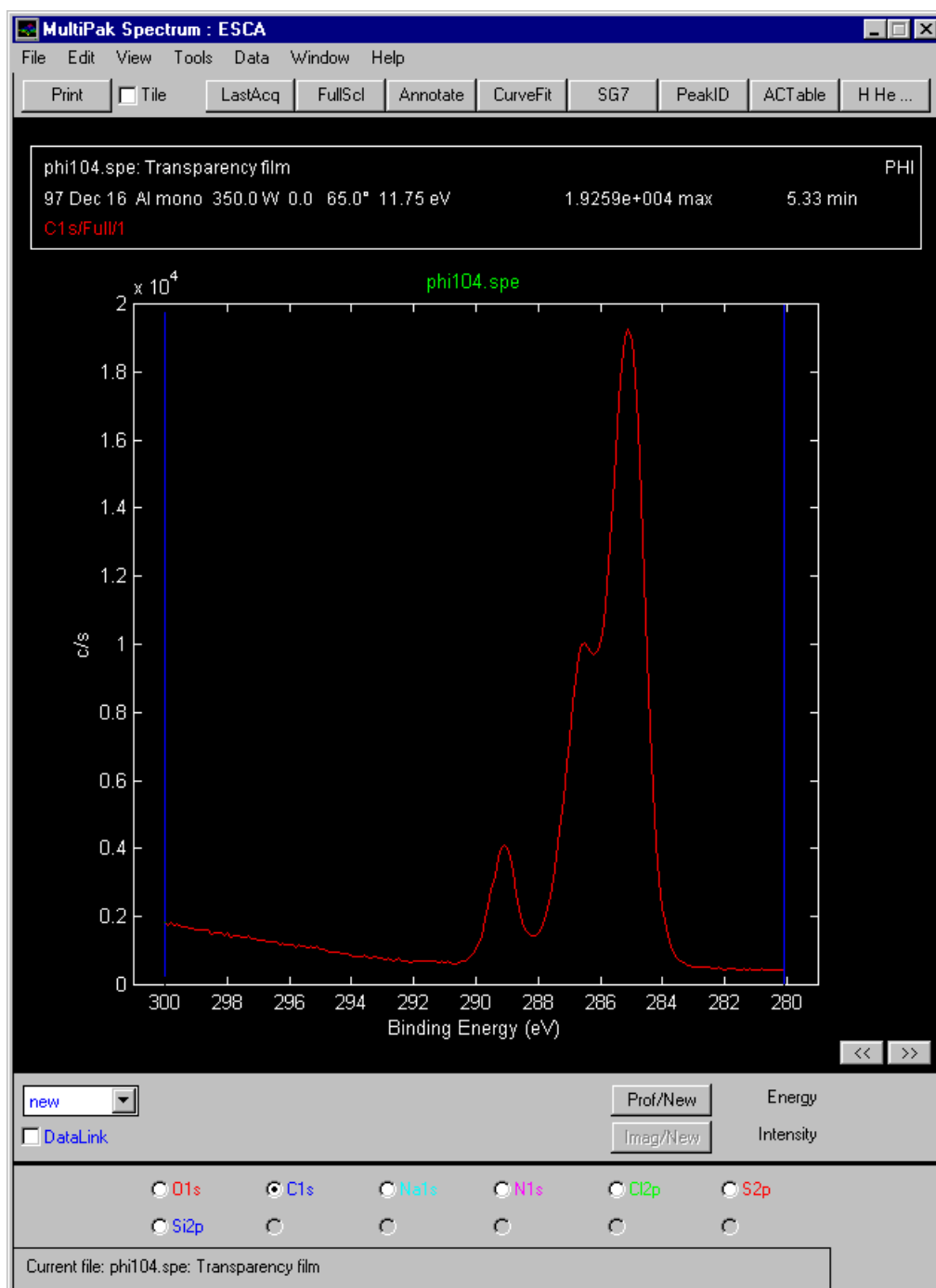
3: Data Acquisition and Reduction



Display of multiplex data file, showing all regions on one axis and a button for each region.

Initially all the individual spectra will be displayed on one energy axis, and there will be a button for each element at the bottom of the Spectrum window. To view an individual element's spectrum, press the button associated with that element.

3: Data Acquisition and Reduction



MultiPak Spectrum window showing the C1s spectrum that was selected by toggling the C1s region button with the left mouse button.

If the objective of the multiplex was focused only on sensitivity and quantification, it is probably not necessary to massage or print the spectral data. You should adjust the analysis cursors so that the background is properly subtracted (A Shirley background is recommended.) and press the ACTable button to view the results of the atomic concentration calculation.

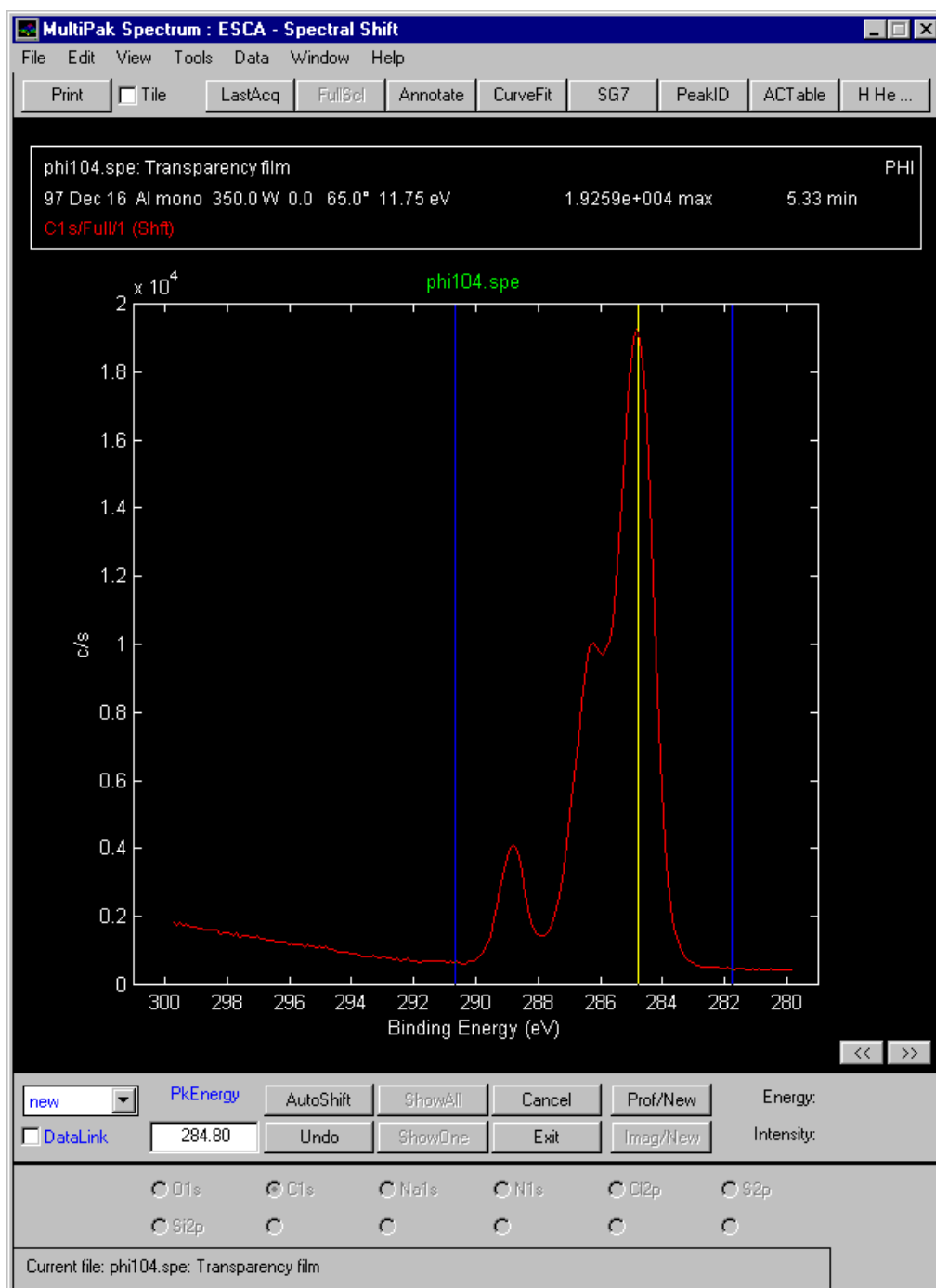
3: Data Acquisition and Reduction

If the focus of the multiplex was to examine the peak shape and position of the spectra to obtain chemical shift information, there are several software tools in MultiPak to help with that task.

Before reducing the data in a multiplex, a smoothing routine may be applied to the data set to improve the accuracy of peak position and peak area measurements. A Savitzky-Golay smoothing routine is recommended, because it does an excellent job of retaining the original shape of the data curve during the smoothing process. Depending upon the size of the data steps and the width of the peaks, a 5-, 7-, or 9-point smoothing operation is typically used. To set up the Smooth button, select the Data item at the top of the spectrum window and then select Smooth/Derivative Setup from the pulldown. This will open a window for setting up a smoothing functions that will be associated with the Smooth button in the Spectrum window. Using the left mouse button, select the Savitzky-Golay smooth and the desired number of points to be used for smoothing. If you selected 9 points, the Smooth button will read 'SG9'. The smoothing functions operate on the displayed data. If you press the smooth button a second time, the data will be smoothed twice.

The first step in extracting chemical information from a peak is to correct the energy scale for any shifts that are due to charging. This is typically done by looking at the position of the C 1s peak from the hydrocarbon that is present on most sample surfaces. If the C 1s peak is not at approximately 284.8 eV, it should be adjusted using the Shift routine. To open the Shift routine, select the Data item at the top of the Spectrum window and select Shift Setup. A yellow cursor will appear on the screen that you should position with the left mouse button on the center of the hydrocarbon portion of the C 1s peak. When you release the mouse button, the C 1s will shift to 284.8eV (or the position for C 1s defined in your Periodic Table database). All other peaks in the data file will also be shifted by the same amount. If no C is present on the sample, a peak from a matrix element with known chemistry and binding energy may be used in place of the C 1s to correct the energy scale for charge induced shifts.

3: Data Acquisition and Reduction



Charge correcting the data using the Shift Tool on the C1s peak. The C1s peak for CH has been shifted to 284.80 eV.

Note: All other regions in the data file will be shifted by the same value.

The next step in extracting chemical information is to determine the binding energy of the peaks for each element. This can be done in several ways. With the peak displayed in the Spectrum window, position the mouse pointer at the

3: Data Acquisition and Reduction

center of the peak. The location of the pointer is displayed in terms of binding energy and intensity at the lower right corner of the Spectrum window.

The FWHM/Area function may also be used to determine the binding energy of a peak. With the peak displayed in the Spectrum window, select the Tools item at the top of the MultiPak spectrum window and select FWHM/Area from the pulldown. The FWHM/Area routine will automatically search for the most intense peak in the displayed data region and report a list of information that describes the peak including its binding energy. A yellow cursor is displayed where the software has determined the location of the peak to be. If you want to change this position, grab the cursor using the left mouse button and drag it to the desired location. When you have finished moving the cursor, the displayed information will be updated.

If the peak of interest contains more than one chemical state, it may be necessary to use the Curve Fit routine to determine the binding energy of each of the chemical states. To perform a curve fit, use the following steps:

1. Make any necessary corrections to the energy scale using the Shift routine.
2. Adjust the analysis window cursors so that an appropriate background subtraction will be used in conjunction with the curve fit.
3. Press the Curve Fit button in the upper tool bar.
4. Select the Setup mode, if it is not already selected.
5. Select a Gaussian-Lorentzian peak shape. (This peak shape is appropriate for most ESCA peaks.)
6. Define initial peaks (bands) by pointing and clicking at the desired peak position with the middle mouse button.
7. Readjust the initial peak estimates by entering numerical values in the Curve Fit Setup menu or drag the flags attached to the currently selected peak using the mouse.
8. Press the Fit button to start the fitting routine.
9. Evaluate the resulting peak positions as shapes using your experience, reference spectra, and database information.
10. Make any necessary adjustments to the band parameters and fit again.
11. When you are satisfied with the results, print the Summary information and the curve fit spectrum.
12. Press Exit to leave the curve fit routine.

3: Data Acquisition and Reduction

Curve Fit - Setup

File

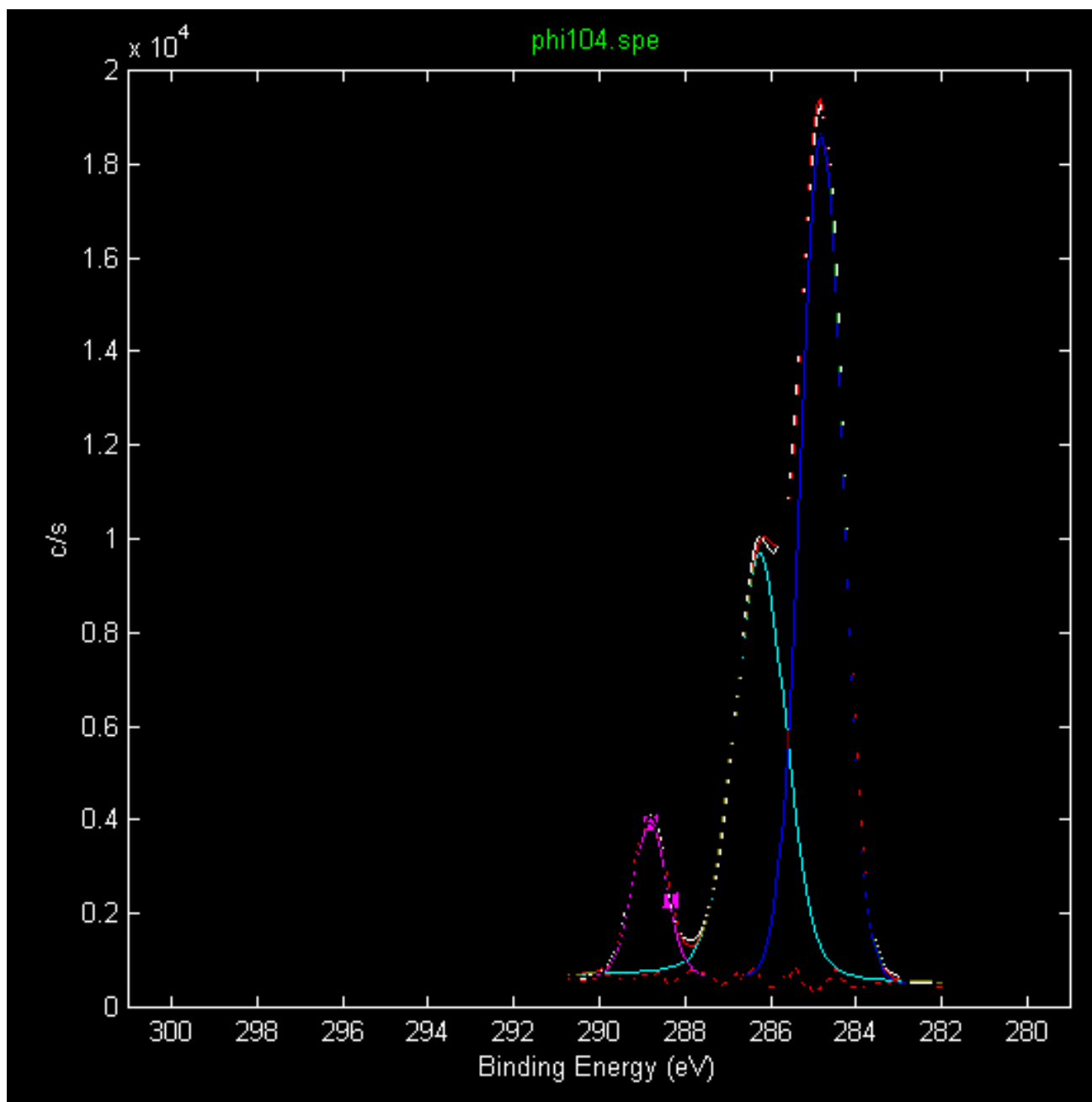
Setup File Gauss-Lorentz

Max Iterations Use Previous

ChiSquared 4.579

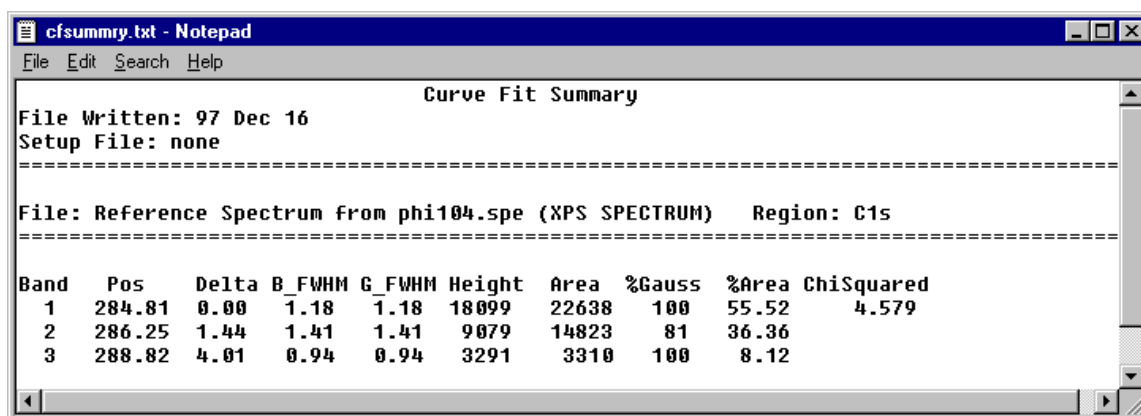
	<input type="radio"/> Band1	<input type="radio"/> Band2	<input checked="" type="radio"/> Band3
Area	22638	14823	3310
Band FWHM	1.18	1.41	0.94
Position	<input type="text" value="284.81"/>	<input type="text" value="286.25"/>	<input type="text" value="288.82"/>
Height	<input type="text" value="18099"/>	<input type="text" value="9079"/>	<input type="text" value="3291"/>
Gauss FWHM	<input type="text" value="1.18"/>	<input type="text" value="1.41"/>	<input type="text" value="0.94"/>
% Gauss	<input type="text" value="100"/>	<input type="text" value="81"/>	<input type="text" value="100"/>
Tail Length	<input type="text"/>	<input type="text"/>	<input type="text"/>
Tail Scale	<input type="text"/>	<input type="text"/>	<input type="text"/>
Lock Area	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>
Area Ratio	<input type="text" value="0.00"/>	<input type="text" value="0.00"/>	<input type="text" value="0.00"/>
Lock Pos	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>
Separation	<input type="text" value="0.00"/>	<input type="text" value="0.00"/>	<input type="text" value="0.00"/>

The curve fit setup menu shows that three bands have been setup to fit the C1s peak.



A curve fit with three bands was applied to the C1s peak. The fitting error is shown in red along the baseline of the peak.

The Curve Fit Summary table contains peak position information that can be used to determine chemical state identity and peak area information that can be used to quantify those chemical states. For a more detailed discussion of the Curve Fit software, see the *MultiPak Software Manual*.



cfsummary.txt - Notepad

File Edit Search Help

Curve Fit Summary

File Written: 97 Dec 16
Setup File: none

=====

File: Reference Spectrum from phi104.spe (XPS SPECTRUM) Region: C1s

=====

Band	Pos	Delta	B_FWHM	G_FWHM	Height	Area	%Gauss	%Area	ChiSquared
1	284.81	0.00	1.18	1.18	18099	22638	100	55.52	4.579
2	286.25	1.44	1.41	1.41	9079	14823	81	36.36	
3	288.82	4.01	0.94	0.94	3291	3310	100	8.12	

The curve fit summary table.

Peak areas are typically used to quantify ESCA spectra. To define the background line necessary for a peak area measurement, push the button for that element and adjust the analysis cursors, if necessary. When each element has been evaluated, press the AC Table button to view the results.

To print multiplex spectra, select the element for display in the Spectrum window and press the Print button. To tile several of the spectrum on a single sheet, you can use the Publish mode that is found on the Tools pulldown menu.

Sputter Depth Profiles

A sputter depth profile consists of a series of multiplex type spectra that have been collected at different depths as material was removed from the sample. The purpose of a depth profile is usually to verify or determine the construction of a thin film structure. Other applications include studying inter-diffusion of layers, looking for impurities in layers or at interfaces, and general characterization of surfaces.

The setup of the data acquisition parameters for a depth profile is very similar to that used for a multiplex. There are tradeoffs to make with time, pass energy and step size. You should remember that you will be sputtering the sample prior to collecting spectra. The sputtering process may cause some chemical damage and broaden the peaks. For this reason, depth profiles are usually collected with medium to high pass energies (50 - 100 eV). With these pass energies, there is sufficient energy resolution to observe most chemical shifts and the higher pass energy sensitivity is improved.

Whether you are trying to optimize spectral detail or sensitivity, you should select as large an analysis area as possible, while restricting the analysis to the feature of interest. This will provide the highest possible count rates and shorten the analysis time.

3: Data Acquisition and Reduction

In addition to defining the data acquisition parameters for a sputter depth profile, you must also determine the sputter interval that is necessary to detect the layers you expect to see. The parameters you can control on the sputter ion gun include: accelerating voltage (beam voltage), ion current, the size of the sputtered area, and the option of rotating the sample while sputtering (Zalar rotation). The sputter etching rate of the ion gun can be calibrated by measuring ion current with the Faraday cup and sputtering through a reference film of known thickness. Reference films of SiO₂ on Si and Ta₂O₅ on Ta are available from Physical Electronics and other suppliers of surface analysis accessories.

To collect a sputter depth profile, perform the following steps:

1. Collect a survey spectrum to determine or confirm which elements are of interest.
2. In the Ion Gun Control window, select the Standby mode. (For improved stability of etch rate and reproducibility of the location of the sputter crater, enter the Standby mode 30 minutes before using the ion gun.)
3. Turn on the differential ion pumping for the ion gun using the Auto Valve Control.
4. Adjust the manual or regulated leak valve for the desired gas pressure, typically 15 mPa.
5. In the Ion Gun Control menu, select the desired sputter setting.
6. Select Setup Profile from the Acquisition Setup pulldown.
7. Item 'a' in the ESCA Profile - Region Parameters setup menu will allow the previous settings, a previous data file, or new settings to be defined.
8. In line 'b,' enter the name of the peak(s) you would like analyzed. The name for the major transition of most elements is the element symbol followed by the number '1'. If you do not know the name of a specific ESCA peak, enter just the elemental symbol, and the PC-ACCESS software will display a list of valid choices for that element.
9. Move to the ESCA Profile - Profile Parameters menu and enter the total sputter time in line 'a.'
10. In line 'b,' select the Alternating mode.
11. In line 'c,' enter the desired sputter interval that will determine how often data is collected.
12. Select the desired x-ray source and anode, typically the Al monochromator using the 2 mm filament.
13. If the sample is insulating and the X-ray monochromator will be used, select a valid ion neutralizer setting and select Neutralize in the Ion Gun Control menu. In the Neutralizer control menu, select a valid setting and turn on the electron neutralizer.
14. Select Acquire.

3: Data Acquisition and Reduction

Physical Electronics PC-ACCESS ESCA V7.1A Beta2 Wed Dec 17 09:29 1997

Add Region Delete Region Add Setting Profile Parm's Abort

ESCA Profile: Region Parameters

a Settings Previous New File dp

Region selection 1 2 3 4 5

b Element name Cr1 Ni1 O1 Si1 Cl

Acquisition window

c	Lower limit (eV)	568.0	844.0	525.0	95.0	278.0
d	Range (eV)	25.0	50.0	20.0	20.0	20.0
	Upper limit (eV)	593.0	894.0	545.0	115.0	298.0

Analysis window

e	Lower Limit (eV)	568.0	844.0	525.0	95.0	280.0
f	Range (eV)	25.0	50.0	20.0	20.0	15.0
	Upper limit (eV)	593.0	894.0	545.0	115.0	295.0
g	Resolution	SURV	SURV	SURV	SURV	SURV
	eV/Step	0.200	0.200	0.200	0.200	0.200
	Time/Step (ms)	50	50	50	50	50
	Pass energy (eV)	93.90	93.90	93.90	93.90	93.90
h	Sweeps	4	4	4	5	5

The Profile Region Parameters menu.

Physical Electronics PC-ACCESS ESCA V7.1A Beta2 Wed Dec 17 09:31 1997

Acquire Add Setting Region Parm's Image Area Abort Exit

ESCA Profile: Profile Parameters

Current Previous

Periodic data

a Total Sputter time (Min) 30.0

b Sputter type Alternating Continuous

c Sputter interval (Min) 0.5

d X-RAY on during sputter Yes No e X-RAY retracted during sputter Yes No

X-RAY source

f X-RAY anode Mg Standard BOTH Standard REF Mg Al Monochromated 2mm filament

Al Standard BOTH Standard REF Al Al Monochromated 7mm filament

Anode 1	Mg	Power (w)	400
Anode 2	Al	Power (w)	300
Filament 1	Al	Power (w)	350
Filament 2	Al	Power (w)	200
X-RAY voltage			14.0

g Peak measurement use Area Height

h Delay after sputter (sec) 0

i Create MultiPak file clone Yes No

Neutralizer Off

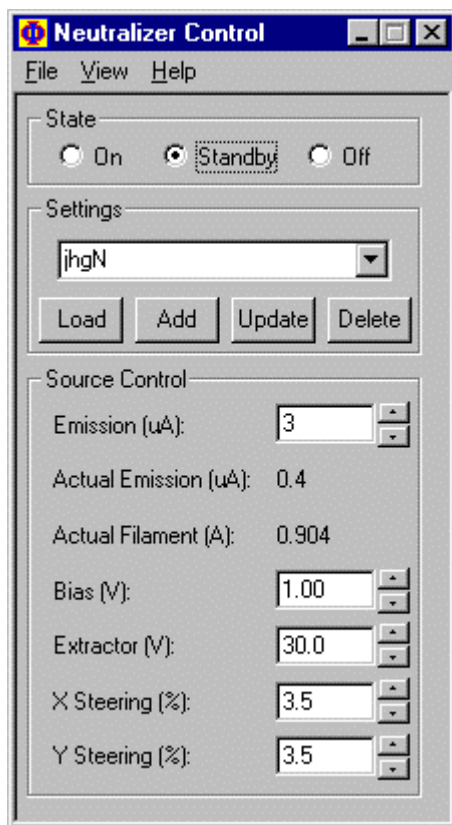
Take off angle (Deg) 65

The Profile Sputter Parameters menu.

3: Data Acquisition and Reduction



The short version of the Ion Gun Control menu



The Neutralizer Control menu for the electron flood gun.

When the acquisition is complete, open MultiPak. To open the sputter depth profile file, press the LastAcq button (which will open the file most recently acquired) or press the File button in the upper tool bar to open the desired file from a File selection menu.

The depth profile will be displayed in the Profile Viewer, and the individual spectra will be displayed on one energy axis in the Spectrum viewer. There will

3: Data Acquisition and Reduction

be a button for each element at the bottom of the Spectrum window. To view an individual element's spectra, press the button associated with that element.

Depth profiles are usually massaged and displayed in terms of atomic concentration versus sputter time. In preparing to quantify a depth profile, Physical Electronics recommends the following steps be performed:

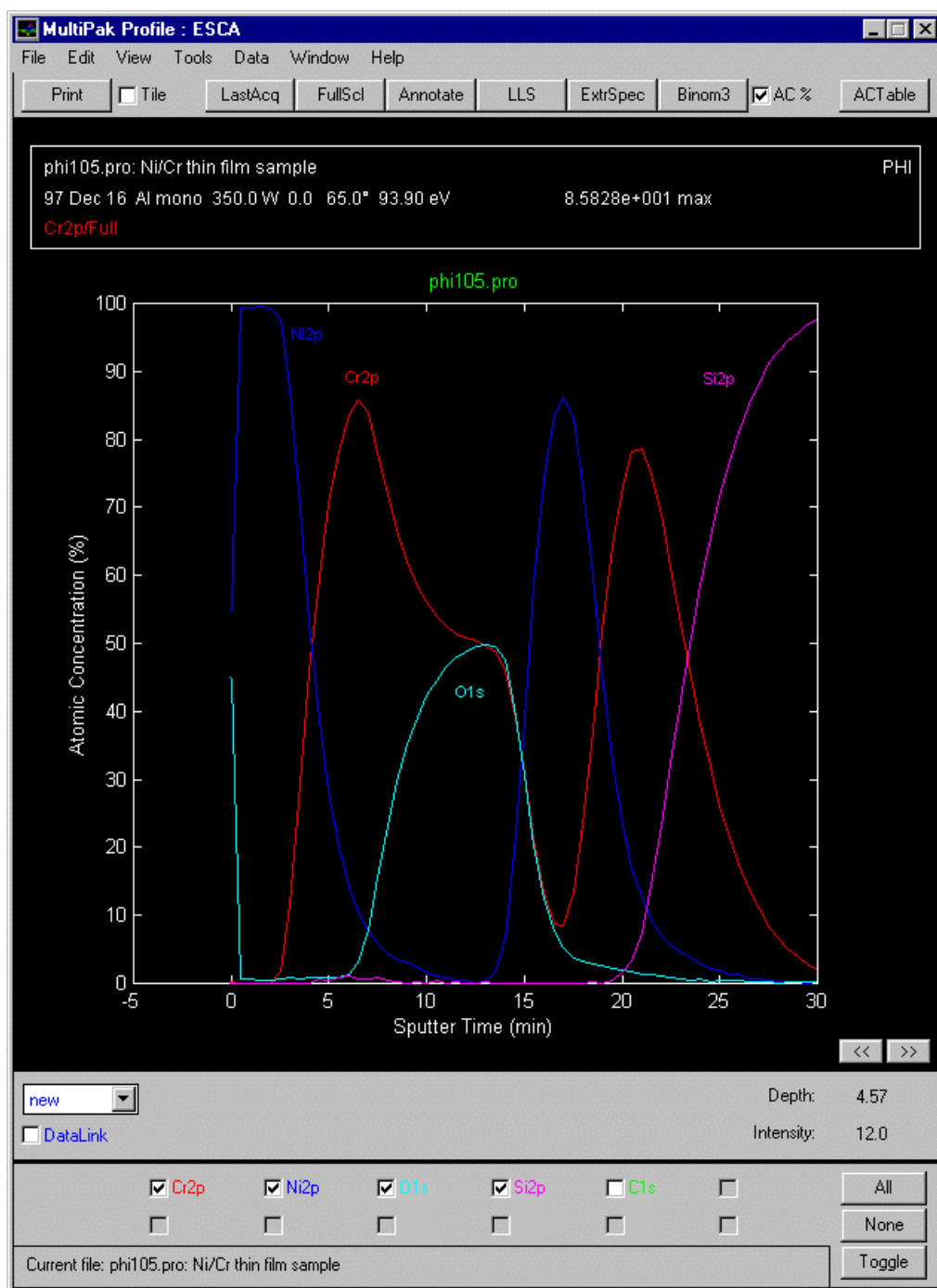
1. With all spectra displayed in the Spectrum Viewer (no individual region selected), press the Smooth button (SG5 - SG9) to smooth all the individual spectra from the profile. Smoothing will improve the accuracy of the peak area measurements by reducing spectral noise and improving the placement of the endpoints used to define the background subtraction.
2. Select each element individually by pressing its button at the bottom of the Spectrum Viewer and adjust the analysis endpoints for background subtraction. When finished adjusting the endpoints for all the elements, press the Update Profile button. This will update the profile display with the newly calculated peak area information.
3. Press the AC button in the Profile viewer to convert the display's relative intensity to atomic concentration.

The previous steps generate a quantitative elemental depth profile that may often meet all your experimental needs. MultiPak contains additional software tools that may be used to improve signal-to-noise ratios, remove peak overlaps, and extract chemical state information from the spectra in a depth profile.

LLS (Linear Least Squares) fitting is a powerful tool that has the ability to improve signal-to-noise ratio, remove peak overlaps and extract chemical state information. It does this by examining the spectral shapes for an element at user selected regions of interest in the profile and fitting extracted peak shapes to the data set using a linear least squares fit.

To demonstrate LLS, we will use profile data from a Ni/Cr/CrO/Ni/Cr/Si multilayer thin film structure. The spectra were smoothed and the analysis endpoints were adjusted prior to displaying the peak area profile shown next.

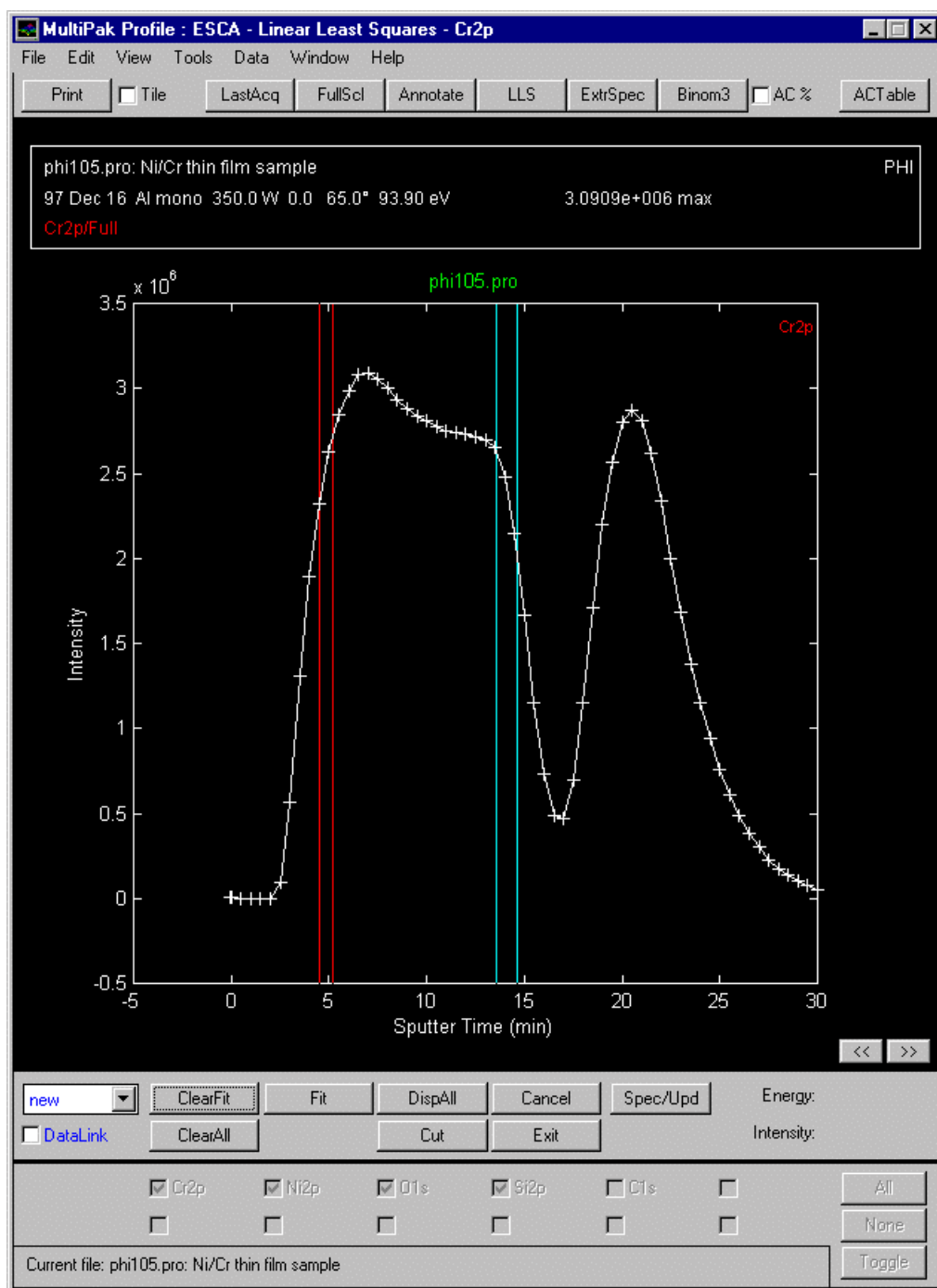
3: Data Acquisition and Reduction



Elemental, peak area-based sputter depth profile

Chromium is present in the depth profile in both the oxide and metal states. LLS can be used very successfully to separate the chemical states and create a chemical depth profile. To begin, press the LLS button and select Chromium by clicking on the 'Cr' button or the Cr data with the left mouse button. Then select the regions of interest using the left mouse button as shown in the following figure.

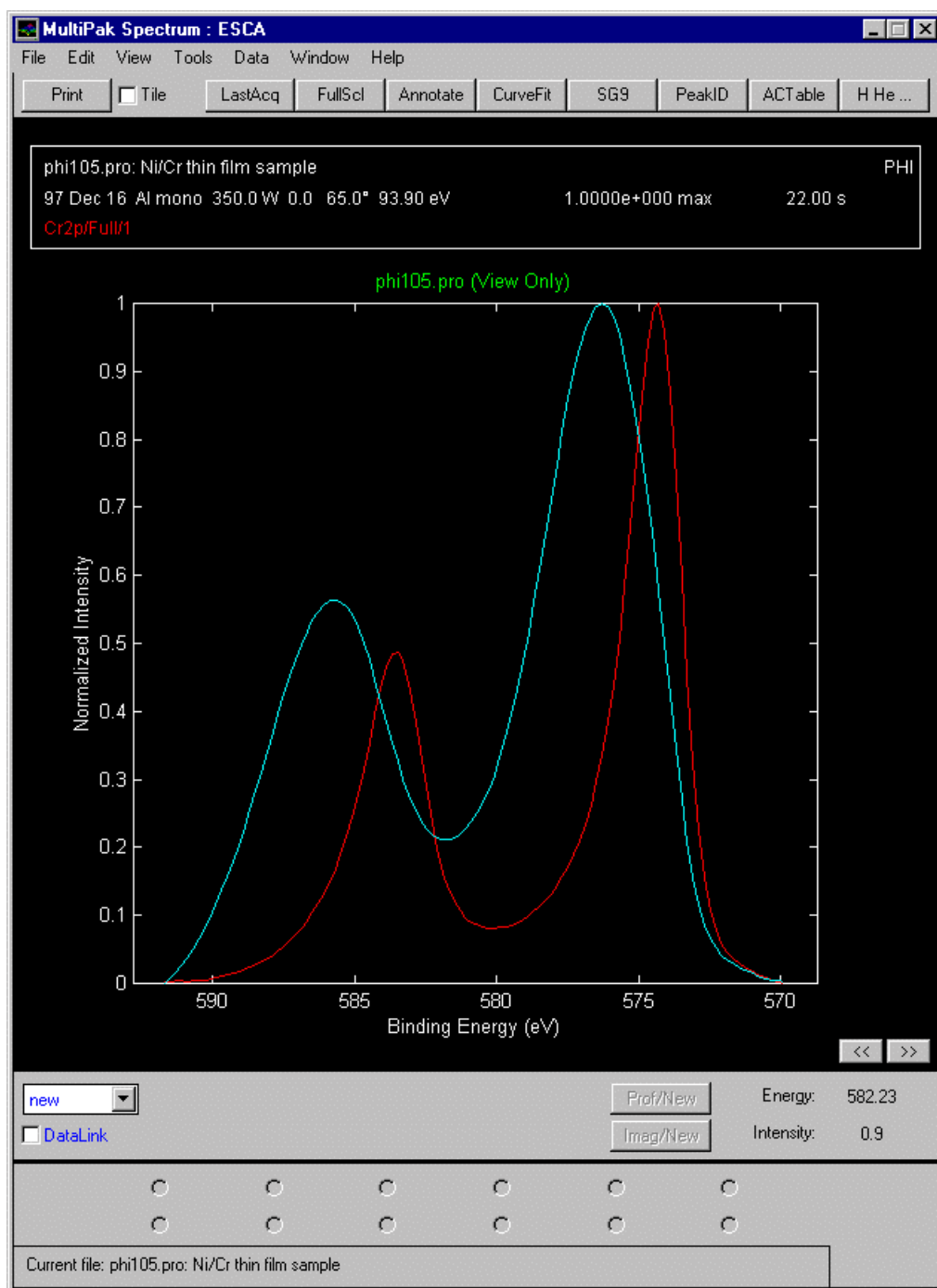
3: Data Acquisition and Reduction



Once the regions are defined, you can view the average spectrum from these regions by pressing the Spec/New button.

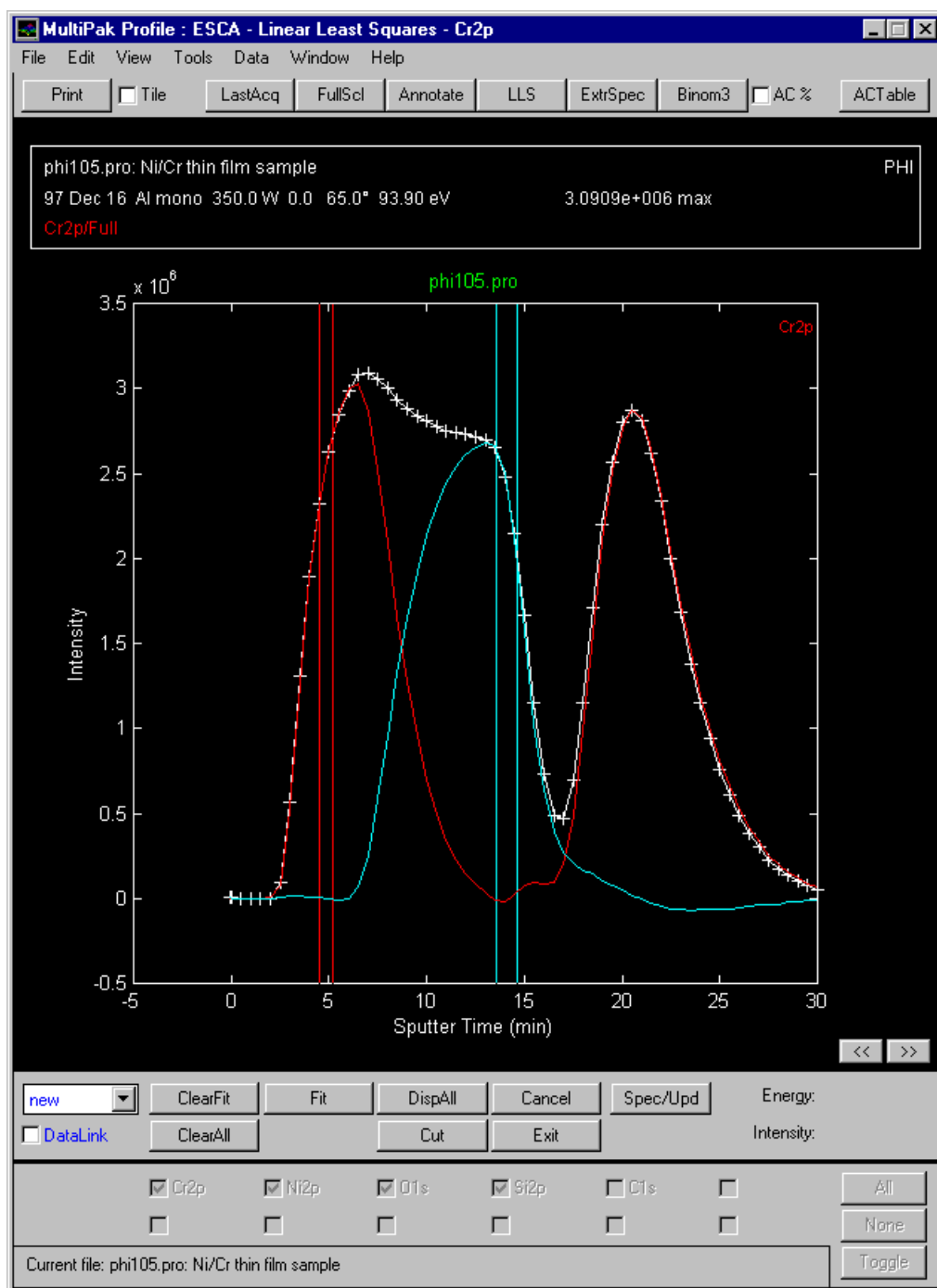
If the spectra are of interest, the entire data set can be fit with the extracted component spectra by pressing the Fit button. The resulting LLS profiles are shown next.

3: Data Acquisition and Reduction



Cr spectra extracted from the depth profile data set showing two different chemical states, Cr and Cr oxide.

3: Data Acquisition and Reduction



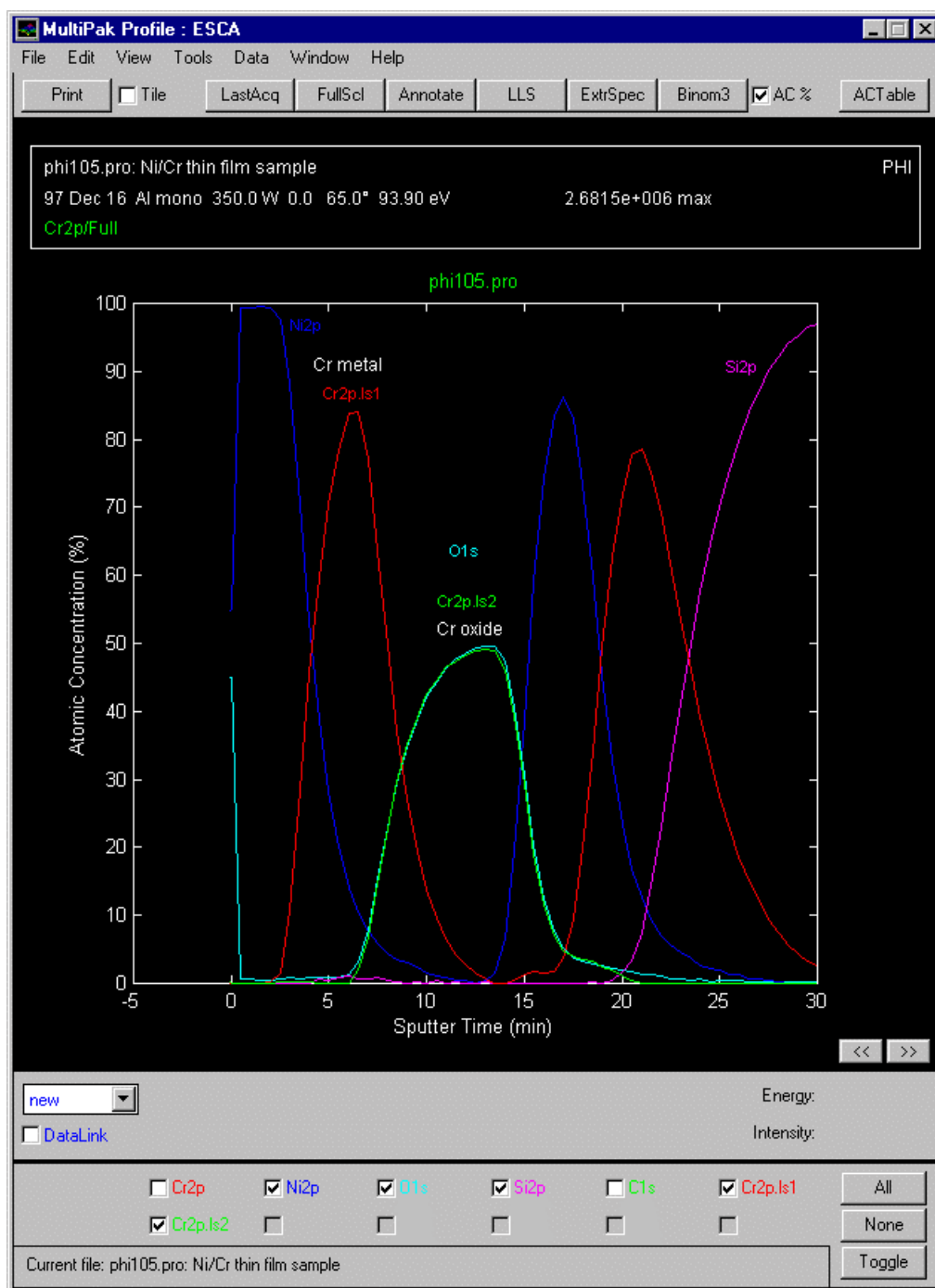
Results of the LLS fitting show that the Cr and Cr oxide are present in distinct, separate layers.

For additional detail about the LLS routine, see the *MultiPak Software Manual*.

The LLS routine is very effective at isolating chemical species or peak overlaps when relatively pure component spectra exist within a sub-region of the depth profile. If pure component spectra do not exist within the data set, curve fitting may be a good alternative for extracting chemical information from the profile.

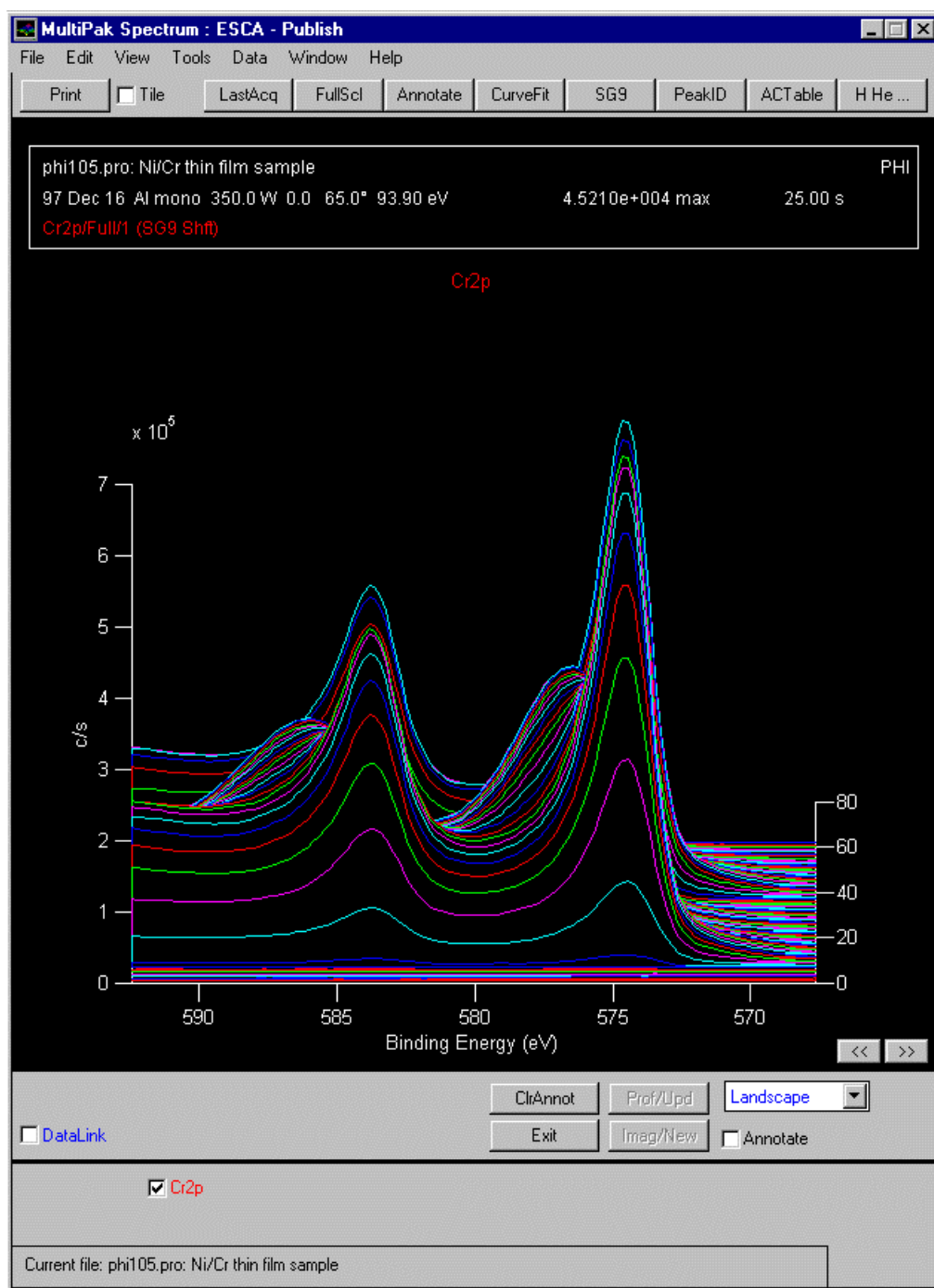
3: Data Acquisition and Reduction

The curve fit routine in MultiPak allows an entire region of a profile to be curve fit, and the results to be brought back into the profile for display and quantification. For detailed information about curve fitting depth profiles, see the *MultiPak Software Manual*.



The results of the LLS fitting show that the Cr and Cr oxide are present in distinct, separate layers.

3: Data Acquisition and Reduction



Montage plot of the Cr spectra generated with the Publish tool.

There are several options for outputting spectra from a depth profile. The buttons at the bottom of the Spectrum Viewer will display all the spectra from a specific element. You can use the Select Spectra function to display a subset of all the spectra from a specific element. The Publish mode will display a montage plot of the spectra from an element or group of elements. See the *MultiPak Software Manual* for additional information about the Publish mode and its options.

Angle Profiles

Angle resolved (angle dependent) profiles are used to probe the near surface region of a sample in a non-destructive manner. Angle profiles are possible, because the input lens of the analyzer (SCA) operates with a small acceptance angle, and it is possible to tilt the sample in front of the analyzer. The analysis depth at a given angle is defined by the equation:

$$d = \lambda \sin \theta$$

where

- d is the effective analysis depth,
- λ is the inelastic mean free path (escape depth), and
- θ is the angle between the sample surface and the analyzer input lens.

Angle profiles are typically used to measure thickness or explore the chemistry of very thin layers. Applications include the study of chemically modified surfaces, lubrication, cleaning processes, etc.

The setup of the data acquisition parameters for an angle profile is very similar to that used for a multiplex. There are concessions to make with time, pass energy and step size. Angle profiles are usually collected with low to medium pass energies (10 - 50 eV), because of the interest in obtaining chemical state information. If elemental information is sufficient for a specific experiment, the pass energy could be increased to 100 eV or more.

Whether you are trying to optimize spectral detail or sensitivity (count rate), you should select as large an analysis area as possible, while restricting the analysis to the feature of interest. This will provide the possible count rates from the analysis area and minimize the analysis time.

For an angle resolved acquisition, the sample should be mounted on the flat 1 inch diameter holders.

In the Acquisition Setup menu, select the Angle Profile option. Select a pass energy and data step size combination that meets the needs of the experiment. Select the largest analysis area that will comfortably fit inside the features of interest.

To collect an angle profile, perform the following steps:

1. Collect a survey spectrum to determine or confirm which elements are of interest.
2. Select Setup A.R. from the Acquisition Setup pulldown.
3. Item 'a' in the ESCA Angle Resolved - 1 menu will allow the previous settings, a previous data file, or new settings to be defined.

3: Data Acquisition and Reduction

4. In line 'b,' enter the name of the peak(s) you would like analyzed. The name for the major transition of most elements is the element symbol followed by the number '1'. If you do not know the name of a specific ESCA peak, enter just the elemental symbol and the PC-*ACCESS* software will display a list of valid element choices for that element.
5. Move to the 'Next Menu', enter the first angle and the number of sweeps. At 10 degrees, the signal intensity will be reduced by a factor of five when compared to high angles. For this reason, more sweeps will be required at lower angles.
6. Select 'Add Angle' and define the second angle and its number of sweeps. Continue until all the desired angles have been defined. Because the analysis depth is changing in relationship to the sine of the angle, you should have more small increments at lower angles. 10, 20, 30, 50, and 80 degrees would be a typical set of angles for an A.R. profile.
7. Select the desired x-ray source and anode, typically the Al monochromator using the 2 mm filament.
8. If the sample is insulating and the X-ray monochromator will be used, select a valid ion neutralizer setting and select Neutralize in the Ion Gun Control menu. In the Neutralizer Control menu, select a valid setting and turn on the electron neutralizer.
9. Select Acquire.
10. Unless the stage is motorized, you will have to manually change the angles and reposition the sample for each angle when prompted to do so by PC-*ACCESS*.

When the acquisition is complete, open MultiPak. To open an angle profile file, press the LastAcq button (which will open the file most recently acquired), or press the File button in the upper tool bar to open the desired file from a File selection menu.

3: Data Acquisition and Reduction

Physical Electronics PC-ACCESS ESCA V7.1A Beta2 Wed Dec 17 16:19 1997

Add Region Delete Region Add Setting Abort Next Menu

ESCA Angle Resolved-1

a Settings Previous New File

Region selection 1 2 3

b Element name 01 C1 Si1

Acquisition window

c	Lower limit (eV)	525.0	280.0	95.0
d	Range (eV)	20.0	20.0	20.0
	Upper limit (eV)	545.0	300.0	115.0

Analysis window

e	Lower Limit (eV)	525.0	280.0	95.0
f	Range (eV)	20.0	20.0	20.0
	Upper limit (eV)	545.0	300.0	115.0
g	Resolution	HRES	HRES	HRES
	eV/Step	0.100	0.100	0.100
	Time/Step (ms)	100	100	100
	Pass energy (eV)	11.75	11.75	11.75
h	Sweeps	5	5	5

The Angle-Resolved region setup menu.

Physical Electronics PC-ACCESS ESCA V7.1A Beta2 Wed Dec 17 16:25 1997

Add Setting Previous Add Angle Delete Angle Abort Exit

ESCA Angle Resolved-2

Current Previous 1 2 3 4 5

Angle Selection

a	Angle (Deg)	10	20	30	50	80
b	Cycles	6	5	4	4	4
	Acq. time (Min)	30	25	20	20	20
	Est. acq. time/cycle (Min)				5.0	
	Est. total acq. time (Min)				115.6	

X-RAY source

c	X-RAY anode	Mg	Standard	BOTH	Standard	REF	Mg	Al Monochromated 2mm filament
		Al	Standard	BOTH	Standard	REF	Al	Al Monochromated 7mm filament
	Anode 1	Mg	Power (w)				400	
	Anode 2	Al	Power (w)				300	
	Filament 1	Al	Power (w)				350	
	Filament 2	Al	Power (w)				200	
	X-RAY voltage						14.0	

d Peak measurement use Area Height

e Create MultiPak file clone Yes No

Neutralizer Off

Take off angle (Deg) 10

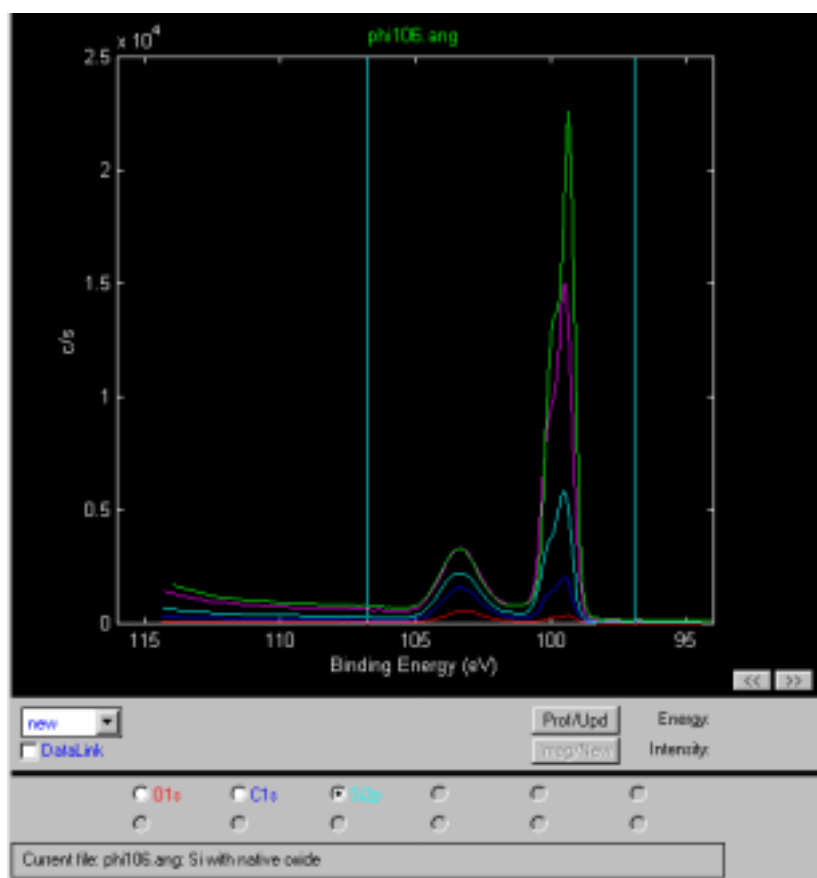
The Angle-Resolved angle setup menu.

3: Data Acquisition and Reduction

The angle profile will be displayed in the Profile Viewer, and the individual spectra will be displayed on one energy axis in the Spectrum Viewer. There is a button for each element at the bottom of the Spectrum window. To view an individual element's spectra, press the button associated with that element.

Angle profiles are usually massaged and displayed in terms of atomic concentration versus $\text{SIN}\theta$. In preparing to quantify an angle profile, Physical Electronics recommends the following steps be performed:

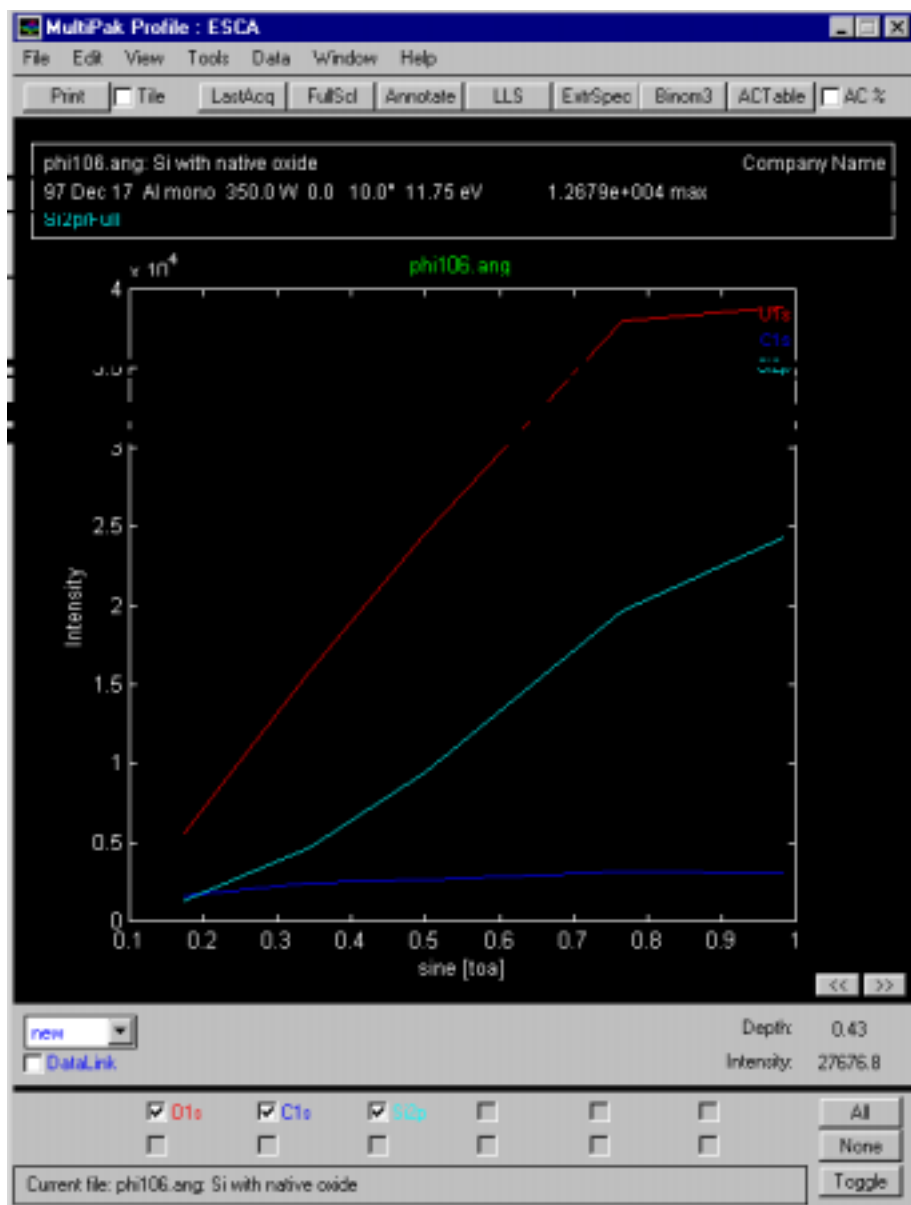
1. With all spectra displayed in the Spectrum Viewer (no individual region selected), press the smooth button (SG5 - SG9) to smooth all the individual spectra from the profile. Smoothing will improve the accuracy of the peak area measurements by reducing spectral noise and improving the placement of the endpoints used to define the background subtraction.
2. Select each element individually by pressing its region button at the bottom of the Spectrum Viewer and adjust the analysis endpoints for proper background subtraction.



The MultiPak Spectrum window showing the selection of endpoints for background subtraction of the Si spectra.

3: Data Acquisition and Reduction

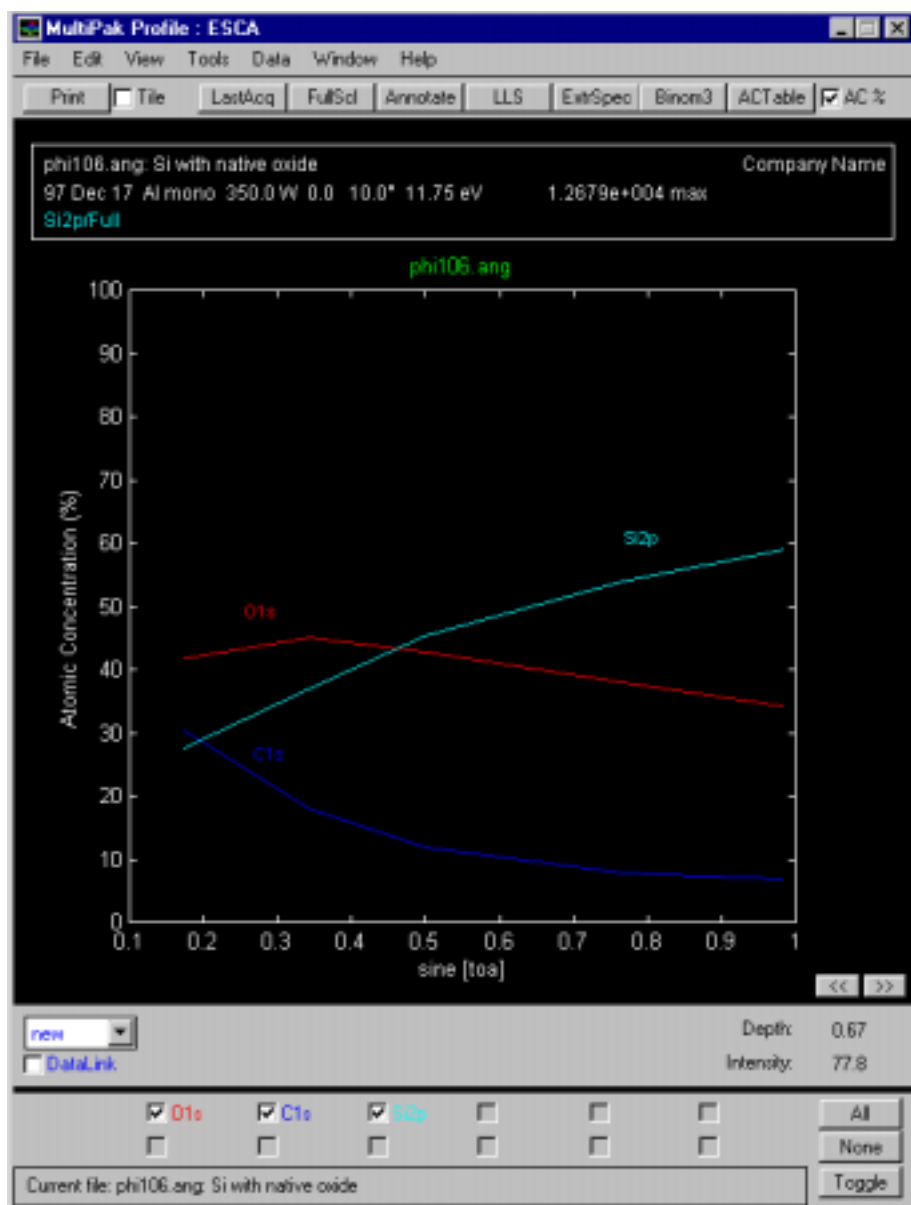
3. After adjusting the cursors for all elements, press the Prof/Upd button to update the profile display with the newly defined peak area information.



The MultiPak profile window showing the peak area display of the Angle Profile that is dominated by the angular response function of the instrument.

4. Press the AC button in the Profile Viewer to convert the display's relative intensity to atomic concentration. In addition to providing quantitative information, this will remove the angular response function from the display.

3: Data Acquisition and Reduction



The MultiPak profile window showing the Atomic Concentration angle profile.

The sample was a Si wafer with a native oxide. The data shows a thin C layer on the surface, an oxide layer below it and the Si wafer substrate as the third layer.

The previous steps generate a quantitative elemental angle profile. MultiPak contains software tools that may be used to improve signal-to-noise, remove peak overlaps, and extract chemical state information from an angle profile.

LLS (Linear Least Squares) fitting is a powerful tool that has the ability to improve signal-to-noise ratio, remove peak overlaps and extract chemical state information. It does this by examining the spectral shapes for an element at user selected regions of interest in the profile and applying extracted peak shapes to

3: Data Acquisition and Reduction

the data set using a linear least squares fit. The LLS software is applied to angle profiles in the same manner it is used for sputter profiles. For a detailed discussion about LLS, see the Sputter Profile portion of this manual or the *MultiPak Software Manual*.

The LLS routine is very effective at isolating chemical species or peak overlaps when relatively pure component spectra exist within a sub-region of the angle profile. If pure component spectra do not exist within the data set, curve fitting may be a good alternative for extracting chemical information from the profile. The curve fit routine in MultiPak allows an entire region of a profile to be curve fit and the results to be brought into the profile for display and quantification. For detailed information about curve fitting, see the *MultiPak Software Manual*.

There are several options for outputting spectra from an angle profile. Use buttons at the bottom of the Spectrum viewer to display all the spectra from a specific element. In addition, you can use the Select Spectra tool to display a subset of spectra from a specific element. You can use the Publish tool to display a montage plot of the spectra from an element or group of elements. See the *MultiPak Software Manual* for additional information about the Publish mode and its options.

Maps

Maps provide a two dimensional display of elemental or chemical state information. Maps may provide information that directly leads to the solution of a problem, but often act as a guide, showing where additional spectra should be taken.

Maps consist of an array of individual pixels. With the 5800, a complete spectrum is collected at each pixel. These spectra are collected in an 'unscanned' mode, where the entire spectrum is collected in one step as a 'snapshot' that greatly reduces the time required to collect a map. The unscanned spectrum contains 16 data points, one from each detector channel. The width of the spectrum is 13.6% of the pass energy being used to collect the data. If a pass energy of 187.85 eV is used, the energy width will be 25.6 eV (1.6 eV/channel).

Maps are typically collected using the maximum pass energy of 187.85 eV, because of the large number of data points and the need to collect data with a good signal-to-noise ratio in a short period of time. The x-ray spot size should be as large as possible. Use an analysis area that matches the average size feature you expect to see in the map. When using the 30 μm analysis area, a time per step of 1 second is appropriate unless the peak for the element being mapped is very intense. With a matrix of 32 x 32 points and a dwell time of one second per point, one sweep of the map will take approximately 17 minutes. If a larger analysis area is used, the acquisition times can be shortened.

3: Data Acquisition and Reduction

Prior to setting up a map you must locate and define the analysis area. For imaging acquisitions, it is especially important that your sample be at the focal point of the x-ray and analyzer optics.

To collect a map, perform the following steps:

1. Collect a survey spectrum to determine or confirm which elements are of interest.
2. Select Setup Map from the Acquisition Setup pulldown.
3. Item 'a,' in the ESCA map menu will allow the previous settings, a previous data file, or new settings to be defined.
4. In line 'b,' select the Unscanned acquisition mode.
5. In line 'c,' enter the name of the element(s) you would to map. The name for the major transition of most elements is the elemental symbol followed by the number '1'. If you do not know the name of a specific ESCA peak, enter just the elemental symbol and the PC-ACCESS software will display a list of valid choices for that element.
6. Select Next Menu.
7. In line 'a,' select the number of pixels for the map. (Select '32' to acquire a matrix of 32 x 32 pixels).
8. Select the Image Area button and item 'd.' Using the return button, toggle to the desired map (image) area. The area can be set to a square with sides varying from 0.125 mm to 2.0 mm.
9. Using items 'b' or 'c,' move the map area to the desired location within the 2 x 2 mm field of view of the analyzer.
10. Select the desired x-ray source and anode, typically the Al monochromator using the 2 mm filament.
11. If the sample is insulating and the X-ray monochromator will be used, select a valid ion neutralizer setting and select Neutralize in the Ion Gun Control menu. In the Neutralizer Control menu, select a valid setting and turn on the electron neutralizer.
12. Select Acquire.

3: Data Acquisition and Reduction

Physical Electronics PC-ACCESS ESCA V7.1A Beta2 Fri Dec 19 14:20 1997

Add Region Delete Region Add Setting Abort Next Menu

ESCA Map-1

a Settings Previous New File

b Acquisition Mode Scanned Unscanned

Region selection

	1	2
c Element name	In1	Cr1
Peak position (eV)	0.0	0.0

Acquisition window

	1	2
d Lower limit (eV)	433.0	565.0
Range (eV)	24.0	24.0
Upper limit (eV)	457.0	589.0

Analysis window

	1	2
e Lower Limit (eV)	441.2	569.5
f Range (eV)	7.5	15.0
Upper limit (eV)	448.8	584.5
g Resolution	SURV	SURV
eV/Step	1.600	1.600
Time/Step (ms)	250	250
Pass energy (eV)	187.85	187.85
h Sweeps	1	1

ESCA Map region setup menu.

Physical Electronics PC-ACCESS ESCA V7.1A Beta2 Fri Dec 19 14:21 1997

Add Setting Previous Menu Image Area Abort Exit

ESCA Map-2

Current File

a Resolution 2 4 8 16 32 64 128

X-RAY source

	1	2
b X-RAY anode	Mg Standard	Al Standard
Anode 1	Mg	Al
Anode 2	Al	Al
Filament 1	Al	Al
Filament 2	Al	Al
X-RAY voltage	14.0	14.0

c Peak measurement use Area Height Integrated

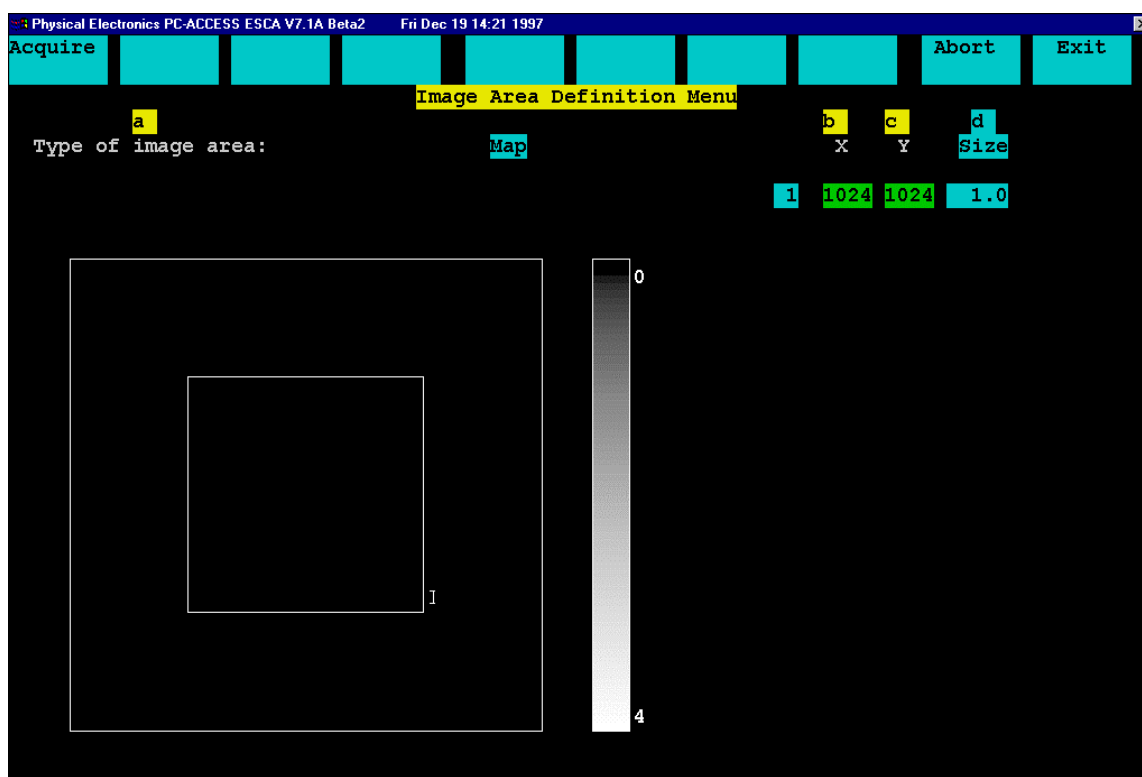
d Create MultiPak file clone Yes No

Neutralizer off

Take off angle (Deg) 45

ESCA Map hardware setup menu.

3: Data Acquisition and Reduction



ESCA Map area definition menu.

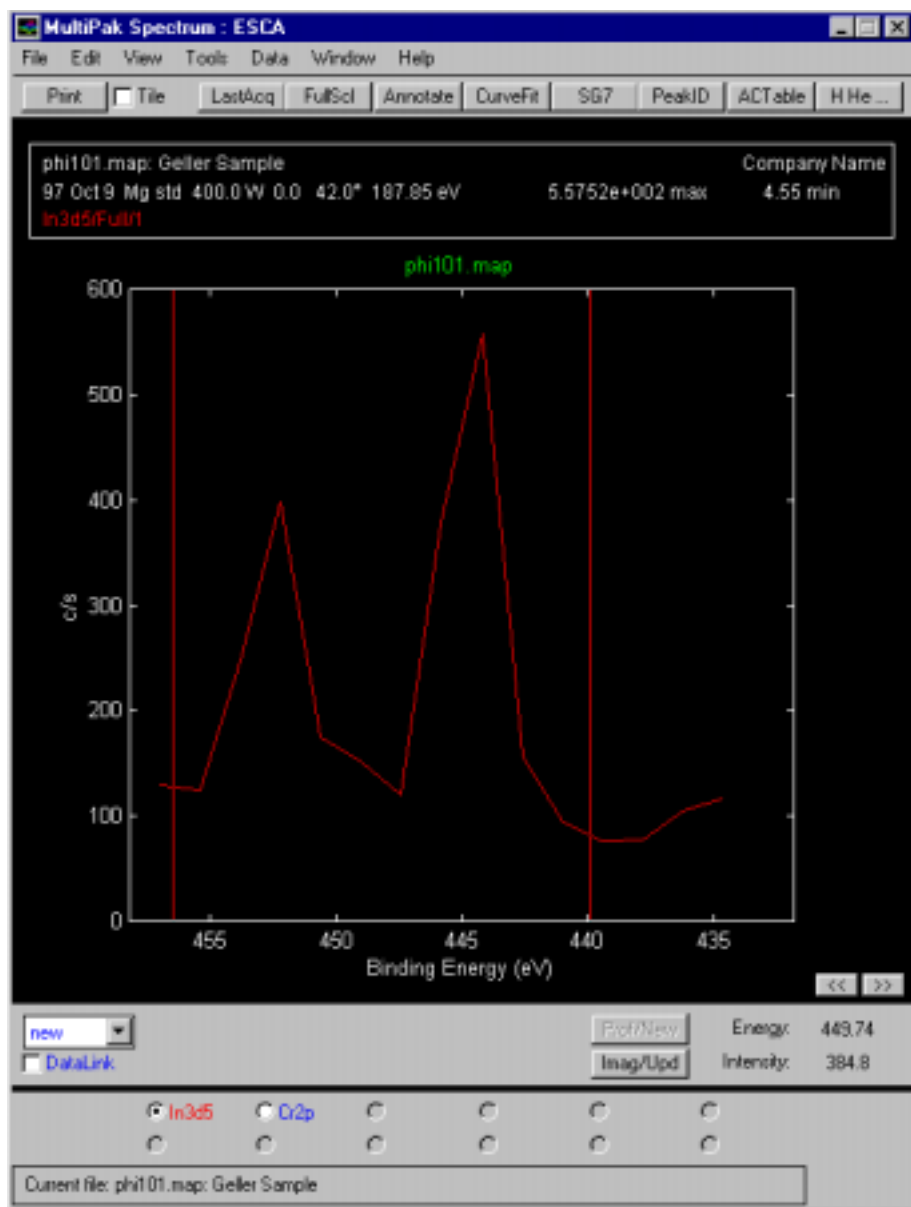
When the acquisition is complete, press the Reduce Data button in the Main Control menu to open MultiPak. To open a map file, press the LastAcq button (which will open the file most recently acquired) or press the File button in the upper tool bar to open the desired file from a File selection menu.

The unprocessed map will be displayed in the Map Viewer, and an average spectrum will be displayed in the Spectrum Viewer. Due to the large number of spectra that are stored in a map file, it may take a few moments to load the file, especially if the file contains maps for multiple elements. When all the spectra have been loaded, a background correction will be made based upon the location of the analysis cursors, and the map display will be updated with the newly calculated peak area information.

The first step in data reduction of a map is to adjust the analysis cursors in the Spectrum Viewer for a proper background subtraction. Each time you make an adjustment with the analysis cursors, the map will be redrawn using the newly calculated peak area values. The next step is to adjust the brightness and contrast using the software handles on the color bar to the right of the map. Brightness is adjusted with the left handle and contrast is adjusted using the two handles on the right side of the color bar. The left mouse button is used to click and drag on the handles. After making a contrast adjustment, the contrast handle will be displayed in an unfilled or outlined mode. When you are finished adjusting the contrast, you will need to close (resolve) the contrast operation by clicking on the

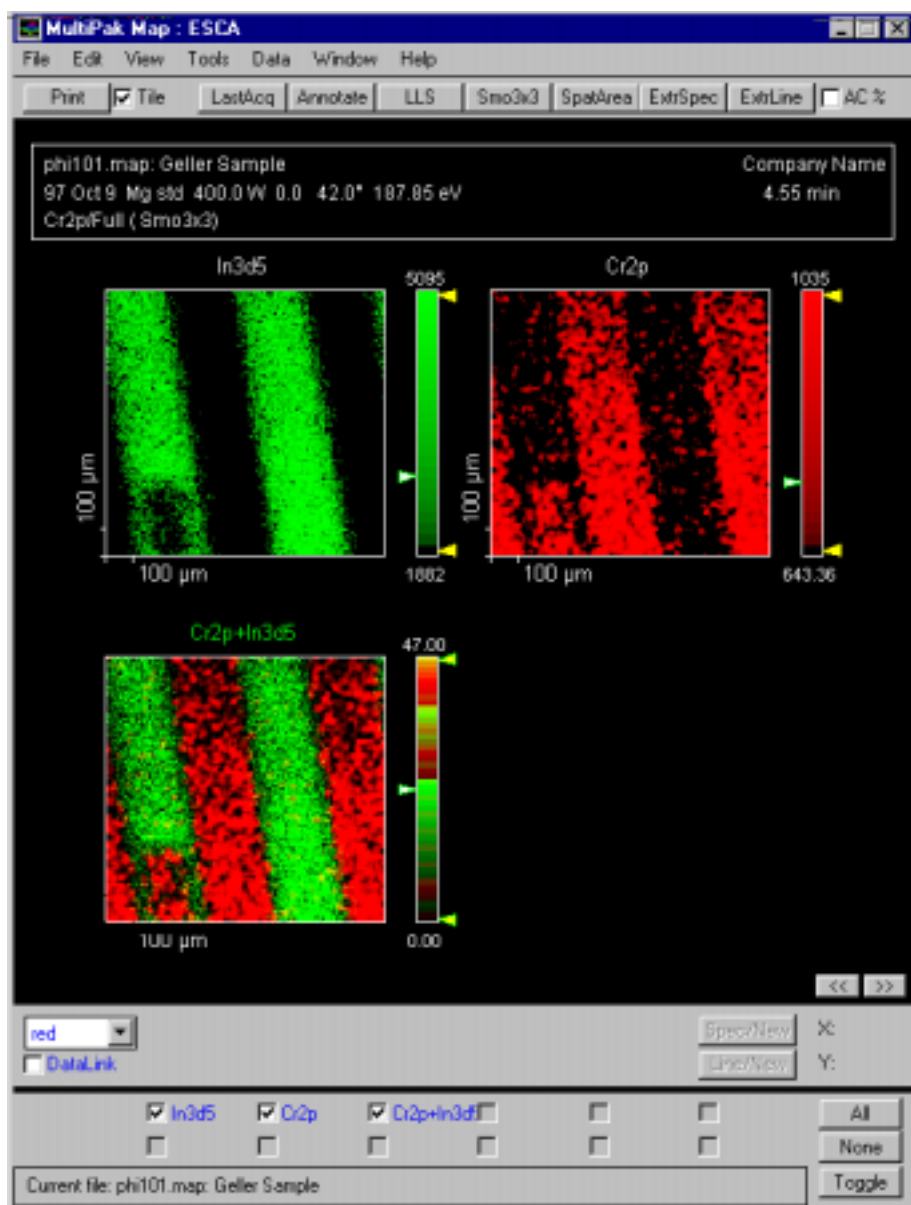
3: Data Acquisition and Reduction

handle with the middle mouse button. Performing a contrast stretch will improve the appearance of most maps. Drag the lower contrast handle up to eliminate noise pixels in black areas, then drag the upper contrast handle down until the features of interest begin to saturate as indicated by their white color during the contrast adjustment (when using a black and white display).



MultiPak spectrum window from In 3d map showing 16 channel unscanned In 3d spectrum.

3: Data Acquisition and Reduction



MultiPak map window showing In and Cr maps.

Opening the color display menu in the tool bar below the map, you can select a gray scale display mode, a color display using red, green, or blue, and one of two color contour modes that associate a color scheme with the peak intensities being displayed.

Additional data massage tools are available for improving the signal-to-noise ratio of a map and for extracting chemical information from ESCA maps. To improve signal-to-noise, there are several mathematical filters available in MultiPak. The most commonly used filter is a 3 x 3 point, map smooth (Smo3x3). A 3 x 3 smooth followed by a contrast stretch will enhance the

3: Data Acquisition and Reduction

appearance of many maps. For detailed information about the filter routines, see the *MultiPak Software Manual*.

To extract chemical shift information from a map, the data file may be treated using the LLS routines in MultiPak. For a detailed description on how to use LLS to extract chemical shift information from map data files, see the *MultiPak Software Manual*.

Maps may be printed in either color or black and white to the system printer, individually or in groups by selecting the Tile option at the top of the map window.