

PHI 5600: Data Acquisition

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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. [See instructions here.](#)
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 subdirectory 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 m analysis area).
8. Select the desired x-ray source, typically the Al monochromator using the 2 mm filament.
9. Select Acquire.

When the survey acquisition has finished, you can display the data in the software provided. 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. Remove labels as needed. To label a peak or element that was missed by the software, you can add that element using the software. If you do not know the identity of an unlabeled peak, you can manually search through the database of peak labels. You can then drag the cursor across a peak and examine the peak labels in the vicinity of the peak of interest.

Quantification of elements

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. When the cursors for all the regions have been adjusted, an atomic concentration table will be displayed.

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 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.

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.

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 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 formost 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.

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.

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.

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.

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.

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 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.

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.
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 PCACCESS.

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.

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 SIN. 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.
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.
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.

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 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 m 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.

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.

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 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).

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 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.

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