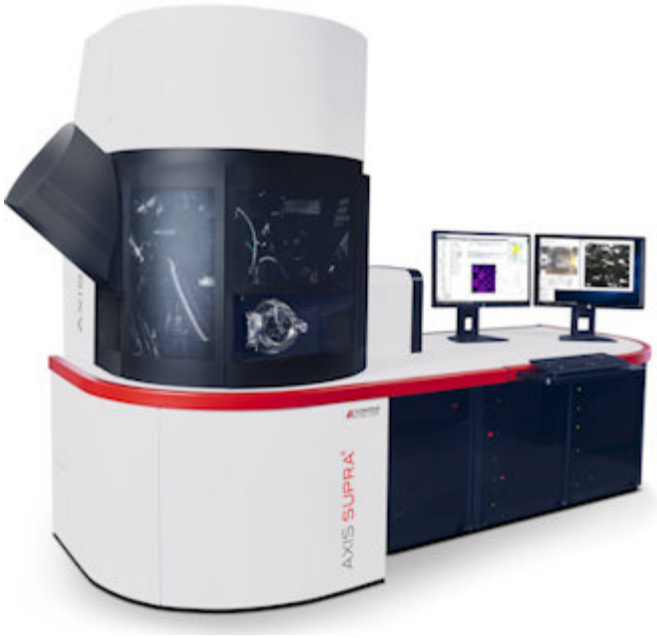
AXIS SUPRA+

and

AXIS SUPRA

Getting Started Guide

39-341



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Getting Started Guide

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This document applies to the AXIS Supra^{+} and AXIS Supra. Unless indicated, the operation of both instruments is the same.

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Contents

Chapter 1	**Warnings**	**5**
	European Union Directives Compliance	6
	FCC Compliance	6
	Electrical Supplies	6
	Maintaining Compliance with EMC and Low Voltage Directives	7
	Sample Records	7
	Ionising Radiation Generator	8
	Laser Light	8
	Handling Liquid Nitrogen	8
	Taking the Vacuum System to Atmospheric Pressure	9
	Backing Pump Exhaust	9
	Radiation Hazard	9
	Thoriated Filaments	10
	Safety Labels	11
	Instrument rating plate	12
Chapter 2	**Overview**	**13**
	Introduction	13
	Excel	14
	Samples	15
	General requirements	15
	Handling and storage	15
	Sample mounting	16
	Sample holders	17

Plain dual height sample holder 17
Plain sample holder with stub end entry 17
ESCApe 18
Introduction 18
Starting ESCApe 18
User interface 20
Status window 23
Message Log 24
ESCApe™ Information 24
Mimic diagram 25

Chapter 3 Sample Handling 27

Venting the Flexi-lock 27
Inserting and/or Removing Sample Holders 29
Pumping down the Flexi-lock 32
Obtaining an Image of a Sample Holder 34
Creating Analysis Locations on a Sample Holder 36
Transferring a Sample Holder 40
Loading a sample holder 40
Unloading a sample holder 41

Chapter 4 Acquiring Data 43

Experiment Files 43
Creating an experiment file 43
Opening an experiment file 45
Acquisition Methods 46
Kratos methods 46
Opening a method 46
Changing a method 47
Creating a new method 47
Analysing Large Areas 49
Auto Z with a survey sequence 49
Acquiring large area high resolution region spectra 55
Analysing Small Features 59
Using microscope images to set up small features 59
Acquiring selected area survey spectra from small features 64
Acquiring selected area high resolution region spectra from small features 66
Acquiring parallel XPS images 68
Acquiring selected area spectra from a parallel image 72
Setting Acquisition Preferences 77
Automatically 77
Manually 78

Chapter 5 Processing 79

Introduction 79
Quantifying survey spectra 80
Curve Fitting for Quantification 85
- Creating a region 86
- Applying a charge correction 86
- Creating components 88
- Adding fitting constraints 90
  - Applying an absolute value constraint 90
  - Constraining a component relative to another 90

Applying a Charge Correction to Other Spectra 94
Applying a Model to Other Spectra 95
Quantification from Multiple Spectra 96
Interacting with Data 98
- Energy Marker 98
- Select from library tool 99
- Charge correction tool 100
- Element Lines tools 102
- Overlaying Spectra 105
- Overlaying Images 108

Appendix A Data 111

Data Organiser 111
- Structure 111
- Opening data from an experiment file 112

Exporting Data Using the VAMAS Format 113
Copying a Displayed Data Item to the Clipboard 114
- Spectra 114
- Image 115

Exporting a Displayed Data Item 116
- Spectra 116
- Image 117

Importing Data from .Experiment Files into Excel 118
Importing Data from .Processed Files into Excel 120

Chapter 1
Warnings

Please read this section of the manual carefully as it sets out the safety issues relating to the operation of the AXIS Supra^{+} and AXIS Supra. All users must read this section and be aware of the safety aspects and safe operating procedures of the spectrometer.

European Union Directives Compliance

The instrument is compliant with the electromagnetic compatibility directive and the low voltage directive. Compliance is signified by labelling the spectrometer with the CE label.

FCC Compliance

‘This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to part 15 of the FCC rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses and can radiate radio frequency energy, and if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which the user will be required to correct the interference at his own expense’.

This instrument complies with Canadian ICES - 001 EMC requirements.

Electrical Supplies

	Europe	USA and Japan
Live	Brown	Black
Neutral	Blue	White
Earth	Green/yellow	Green

MAINS SUPPLY VOLTAGE WARNING

Although the instrument has covers fitted to protect against electrical shock it is strongly recommended that the instrument is switched off and isolated from the mains supply before removing any of the mains protection covers or attempting to replace any of the mains fuses.

WARNING - Mains voltage (230V AC) is present beneath the insulation on all **tape heaters** during baking.

HIGH VOLTAGE WARNING - High voltages (kV) are present within the circuitry of the instruments and extreme caution should be taken when making any adjustments within the instrument.

Maintaining Compliance with EMC and Low Voltage Directives

The following precautions must be observed to maintain conformance with the European electromagnetic compatibility (EMC) and low voltage directives:

1. Operate the instrument within the limits set out in either the technical specification or the ‘site conditions’ sheet.
2. Do not modify the instrument in any way, electrically or mechanically.
3. Ensure that all covers, fan guards, EMC gaskets and screws are fitted.
4. Ensure that all cable screens are connected.
5. Do not change component values.
6. Do not remove any labels.
7. Purchase spare parts from a recommended spare parts list.
8. Always replace parts ‘like for like’.
9. Keep a concise record of all the work carried out on the instrument and any parts changed with serial numbers where appropriate.

WARNING

Service work must be carried out by Kratos trained engineers. Kratos Analytical cannot be held responsible for the action of untrained, non-Kratos service engineers who render the instrument non-compliant with the above European directives.

Sample Records

You must keep a record of all substances analysed with the instrument and their concentration level/amounts. This record is needed by the Kratos Analytical Service Centre should any part of the instrument need servicing/replacing. Any item returned for repair/replacement must be accompanied by this sample report and a completed Equipment Return/ Repair Declaration.

Ionising Radiation Generator

This instrument is covered by Ionising Radiation Regulations 2017 (IRR17). Any company, institution and individual using this instrument must comply with IRR17 and be familiar with its contents. Note that it is the responsibility of the users' employer to ensure compliance with IRR17 and consult with a radiation protection advisor. In particular the following regulations defined in IRR17 might be relevant:

- Regulation 7 - Prior risk assessment.
- Regulation 8 - Restriction of exposure.
- Regulation 10 - Maintenance and examination of engineering controls etc.
- Regulation 12 - Contingency plans.
- Regulation 13 - Radiation Protection Advisers.
- Regulation 14 - Information, instruction and training.
- Regulation 17 - Local rules and Radiation Protection Supervisors.
- Regulation 31 - Duties of manufacturers etc. of articles for use in work with radiation.
- Regulation 33 - Misuse or interference with sources of radiation.
- Regulation 34 - Duties of employee.

The IRR17 and associated ACoP (L121) are available on-line at www.hmso.gov.uk.

Laser Light

This instrument makes provision for the use of a laser as an alignment tool. When fitted to the spectrometer, the device operates as a Class I laser. It must ***not*** be operated independently of the spectrometer, and the warnings and precautions set out in the device manual must be followed. Avoid staring at the reflected laser light for long periods. Do not continue to stare at the reflected light if it appears uncomfortably bright. Staring directly into the beam may cause damage to the eye.

Handling Liquid Nitrogen

Liquid nitrogen boils at a temperature of -195^{o}C. Surfaces cooled to this temperature, including pipes used for filling cold-traps, must never come into contact with skin which would immediately become frozen to the surface. Similarly, when pouring liquid nitrogen, care must be taken to avoid splashing the liquid onto any unprotected areas of skin or eyes.

Liquid nitrogen should only be used in a well ventilated area to avoid any possibility of the evolved nitrogen gas displacing the oxygen in the room and thereby causing suffocation.

Oxygen boils at a higher temperature (-183^{o}C) than nitrogen; any large surface area cooled to liquid nitrogen temperature and open to atmosphere can act as a condenser, producing a high concentration of oxygen locally which could be a fire hazard.

The change in volume as nitrogen changes from liquid to gas is in the approximate ratio 1:700, thus liquid nitrogen must never be introduced into a closed volume or explosive pressures could be generated.

Taking the Vacuum System to Atmospheric Pressure

The various chambers of the vacuum system are designed to operate at low pressure, hence care is needed when backfilling from compressed gas cylinders otherwise the system could be damaged. Do not pressurise the system above atmospheric pressure. It is always advisable to backfill the system with a dry, inert gas, thereby avoiding the production of an explosive mixture if, for example, an inflammable gas has been used in a preparation chamber.

Backing Pump Exhaust

You must be aware of any possible hazardous reaction, for example the emission of toxic vapour, which could take place in the specimen when subjected to the environment of the high vacuum chamber or preparation chamber. If this is a possibility then any pump exhaust gas should be vented externally.

Radiation Hazard

An intense flux of X-radiation can be generated within the instrument. Shielding is more than adequate to limit the dose rate outside the instrument to safe levels.

However, if you undertake any modifications you must bear in mind the need to maintain an adequate level of shielding. This is particularly important if the large viewport at the front of the analysis chamber is removed. The viewport is equipped with a thick lead glass shield. This ***must not be removed*** under any circumstances and ***must always*** be in place to maintain adequate shielding for the operator when X-rays are

being generated. If this viewport is changed for an alternative version, then ***adequate checks must be carried out for leakage of X-radiation*** before using the instrument. No lead glass X-ray protection windows should be removed. Other viewports also give unsafe levels of radiation.

Thoriated Filaments

Thoriated filaments are used in several components and optional extras. They are fitted to the Monochromator X-ray gun and to Minibeam ion guns.

Thorium is a naturally occurring low level alpha-emitter. Although the microscopic amount of thorium present on each filament presents no significant hazard, it is good practice to reduce any potential exposure to the minimum possible. It is suggested, therefore, that these filaments are only handled with a gloved hand and that old filaments and gloves are sealed in a plastic bag and disposed of in accordance with local regulations.

WARNING

Thorium is toxic and may cause cancer. It is harmful by inhalation, contact with skin, or if swallowed.

Wear suitable protective clothing, gloves and eye / face protection. Do not breath dust.

In case of accident, or if you feel unwell, seek medical advice immediately.

If inhaled, remove to fresh air. If breathing becomes difficult, call a physician.

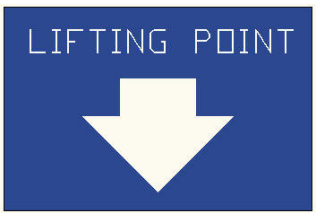
If swallowed, wash out mouth with water provided that the person is conscious. Call a physician.

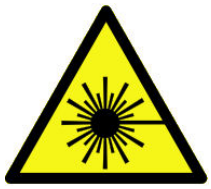
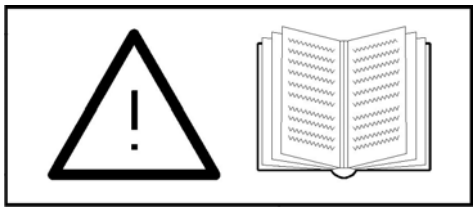
In case of skin contact, flush with copious amounts of water for at least 15 minutes. Remove contaminated clothing and shoes. Call a physician.

In case of contact with eyes, flush with copious amounts of water for at least 15 minutes. Assure adequate flushing by separating the eyelids with the fingers. Call a physician.

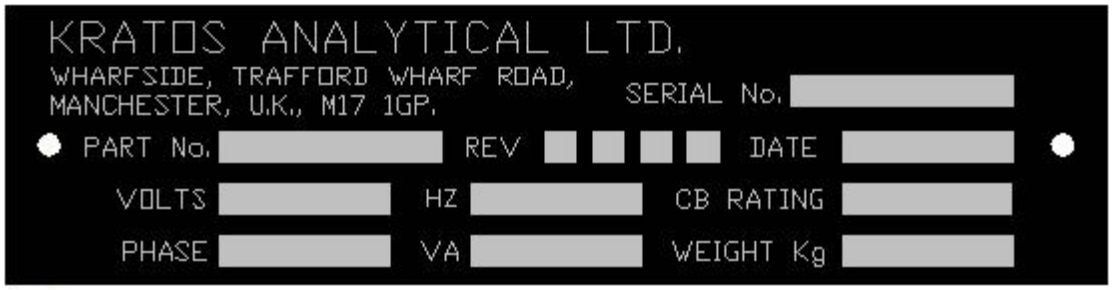
Safety Labels

The following labels are fitted to the instrument in a number of positions. Please observe the warnings given.

Label	Meaning
	Electrical safety warning label
	Electrical safety warning label
	High voltage labels - used together to indicate a hazard from high voltages
	Safety earthing point symbol - indicates the location of an earthing point
	Primary earthing point label
	Lifting point label - indicates the recommended points for lifting the instrument.

Label	Meaning
	Laser hazard symbol - indicates a specific personal injury hazard.
	Hot surface symbol - indicates a specific personal injury hazard.
	Safety alert symbol - indicates a general personal injury hazard such as rotating fans.
	Ionizing radiation symbol - indicates a specific personal injury hazard.
	Read the manual label - indicates that the user manual must be read.

Instrument rating plate



Chapter 2
Overview

Introduction

Samples are mounted on a sample holder. The sample holder is then placed on the sample magazine in the Flexi-lock and the camera is used to take an image of the sample holder. Samples can then be defined within the ESCApe software.

A sample transfer moves the sample magazine through a gate valve into the Sample Analysis Chamber (SAC). The sample holder is then picked up by the gripper on the stage and moved to the analysis zone. The microscope can then be used to define small features on the samples before setting up methods and acquiring data.

Excel

A plugin is provided to enable ESCApe data to be opened in Excel. The data can be manipulated using the tools available in Excel and then used to produce reports. It is recommended that a copy of Excel is installed on the instrument PC. This must be Excel 2007 or later and if possible should be the 64-bit version.

Samples

General requirements

In general the degree of sample cleanliness required for surface analysis is much greater than other analytical techniques.

The material under investigation must be stable in an ultra high vacuum environment. Many materials may release volatile surface species, such as water vapour or plasticisers, which may contaminate other samples. Other materials, such as polymers, foams and porous materials, may have high vapour pressures. Some elements, such as Na, K, S, P, Zn, Se, As, I, Te or Hg, also have relatively high vapour pressures.

When analysing such materials it is advisable to reduce the size of the sample to a minimum and to bear in mind the possible effects of cross contamination of other samples. Pumping the samples overnight in the Flexi-lock may also be advisable, however, the out-gassing of volatile materials can be greatly increased when the sample is subject to X-ray bombardment and/or exposure to the charge neutraliser. In extreme cases such samples can contaminate the vacuum chamber and charge neutraliser elements.

Handling and storage

Do not touch samples or sample holders with unprotected hands. Even if you do not touch the surface to be analysed, low molecular weight oils present on your skin can rapidly migrate over the area of interest. Sodium contamination is often detected on handled materials. It is essential to wear powder free gloves when handling and preparing samples.

Use clean tools made of materials which will not transfer contaminates to the samples. All tools should be demagnetised.

Take great care when cleaning samples prior to analysis. Keep cleaning to a minimum and avoid if possible. Ultrasonic cleaning in a suitable high purity solvent (for example, isopropyl alcohol) is sometimes permitted. Avoid using compressed air to remove surface particles as oil and/or water contaminates are often introduced.

When storing samples the surface of interest should not come into contact with any material. If this is unavoidable then wrapping the sample in clean Al foil may be satisfactory. Glass jars are often preferable to plastic containers due to the risk of volatile plasticisers and/or low molecular weight materials from polymers contaminating the sample surface.

Sample mounting

The best method of mounting samples is to use the screws and plates provided. You should use the BeCu screws and plates unless you are using the Heat and Cool holder. In this case you should use the Molybdenum screws and plates.

If you are unable to mount the samples using the screws and plates you can use one of the following methods:

- vacuum compatible double sided adhesive tape.

  If you use this method then surface active species (such as silicones) can migrate from these tapes. Ensure that you analyse the samples immediately and take care when reviewing the results.

  Note that conductive adhesive tape is preferred to regular double sided tape as electrical contact with the holder can be maintained thereby alleviating problems with sample charging.

  Suitable double sided conductive adhesive tape can be obtained from Kratos or other suppliers.
- fast drying silver paint.

  Ensure that the paint does not come into contact with the surface of interest and that the paint is fully dry before you place the sample in the vacuum system.

  Suitable paint is available from Agar Scientific - part number G302 (www.agarscientific.com).

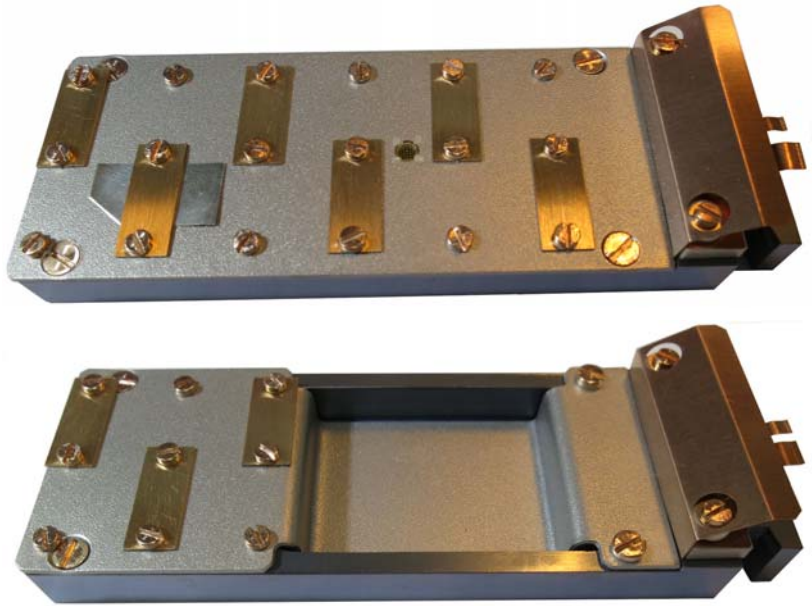
Semi conductor materials or thin insulating coatings on conductors can often present particular mounting problems. This is because vertical differential charging of the sample can sometimes be observed. In practice such problems can be alleviated by mounting the sample electrically isolated from the sample stage.

Sample holders

Samples can be mounted on a number of different sample holders. You need to choose the appropriate sample holder for the sample and for the experiment you want to perform.

Plain dual height sample holder

This sample holder comes with 2 interchangeable top plates which each have a sample mounting area of 75 x 32 mm. The flat plate is suitable for samples up to 7.5 mm in thickness. The recessed part of the other plate is suitable for samples up to 14.5 mm in thickness.



Plain sample holder with stub end entry

This sample holder comprises a flat plate with a sample mounting area of 57 x 32 mm and a fork for loading standard 15 mm diameter sample stubs.

The flat plate is suitable for samples up to 7.5 mm in thickness.

ESCApe

Introduction

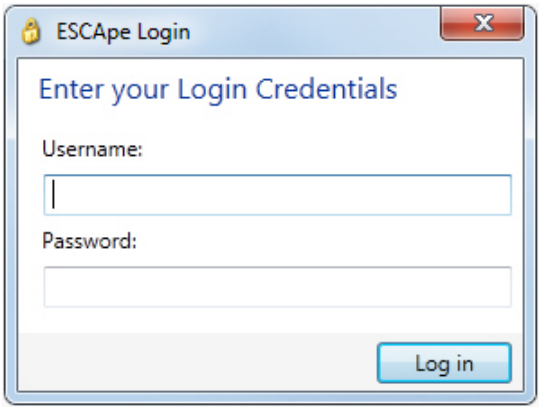
ESCApe software is used for:

- instrument control and calibration
- data acquisition
- data processing.

Starting ESCApe

1. Double click the 

icon to run ESCApe.
Depending on how ESCApe has been configured the following screen may be displayed.



2. If requested, type in your **Username** and **Password** and click **Log in**.

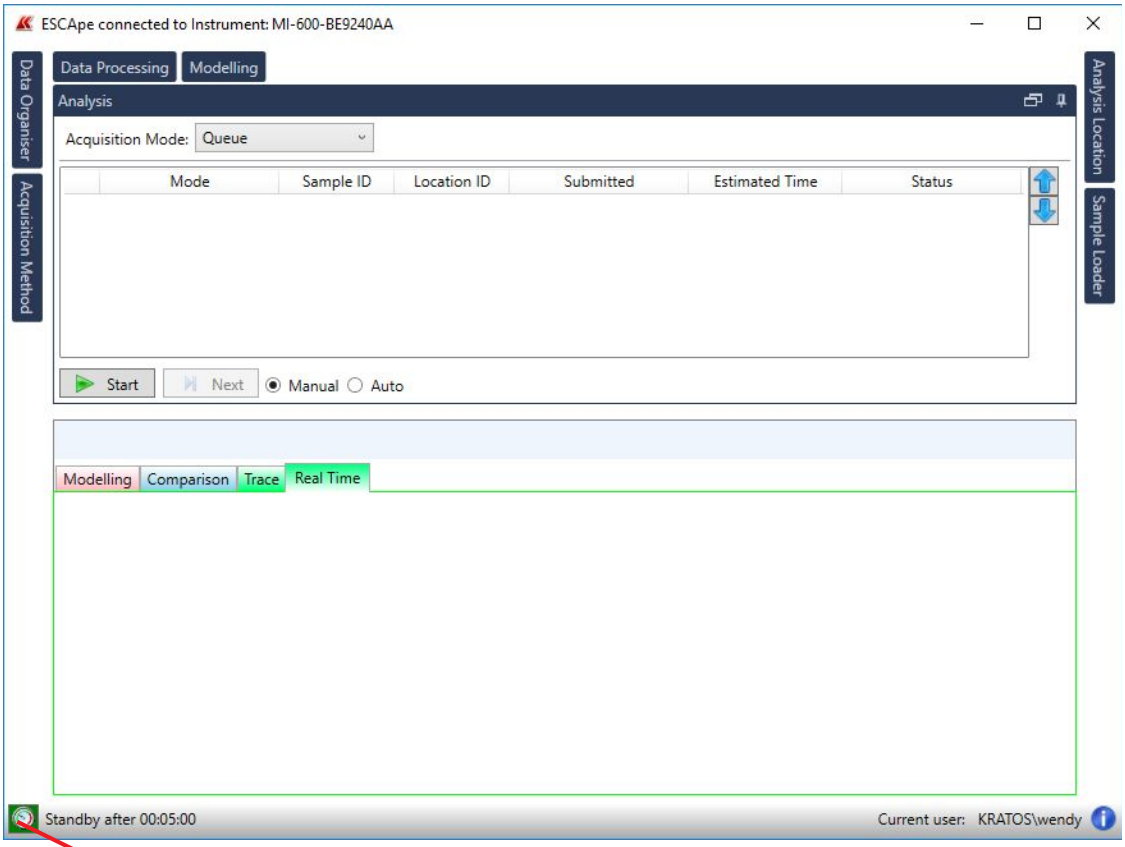
These should be obtained from your administrator.

For your information

If you are asked to enter a username and password, your administrator may have restricted user access to some components in ESCApe. If you are unable to follow any procedures described in this manual, see your administrator for details.

3. The following popup message is displayed:

Wait until initialisation is complete and the Status icon  in the bottom left hand corner of the main window has turned green.



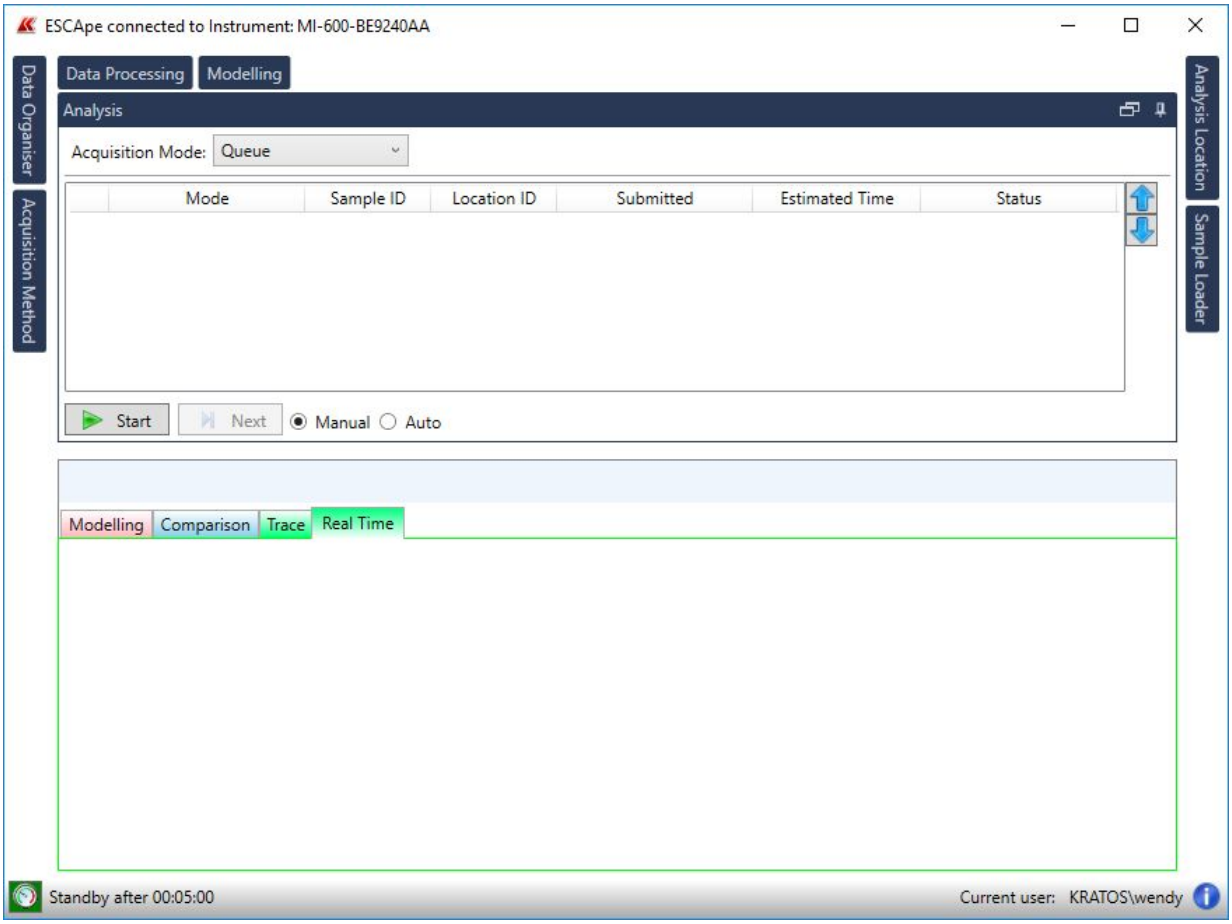
Status icon

User interface

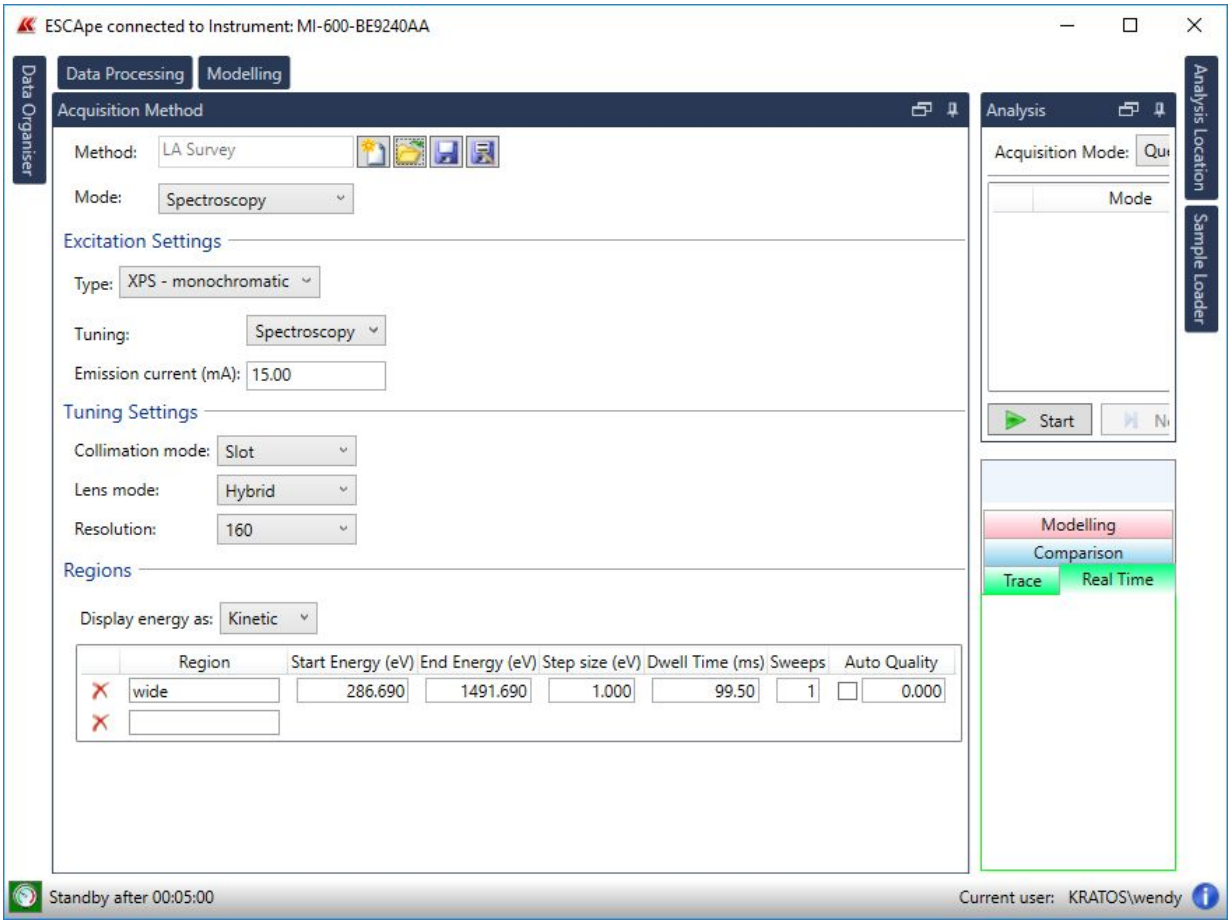
When you log in the main screen comprises an **Analysis** window and a display area with a number of tabs along the top and down the sides. Each tab represents a window.

Windows can be:

- **docked** to fixed positions at the side or at the top of the main window with only the tab visible.



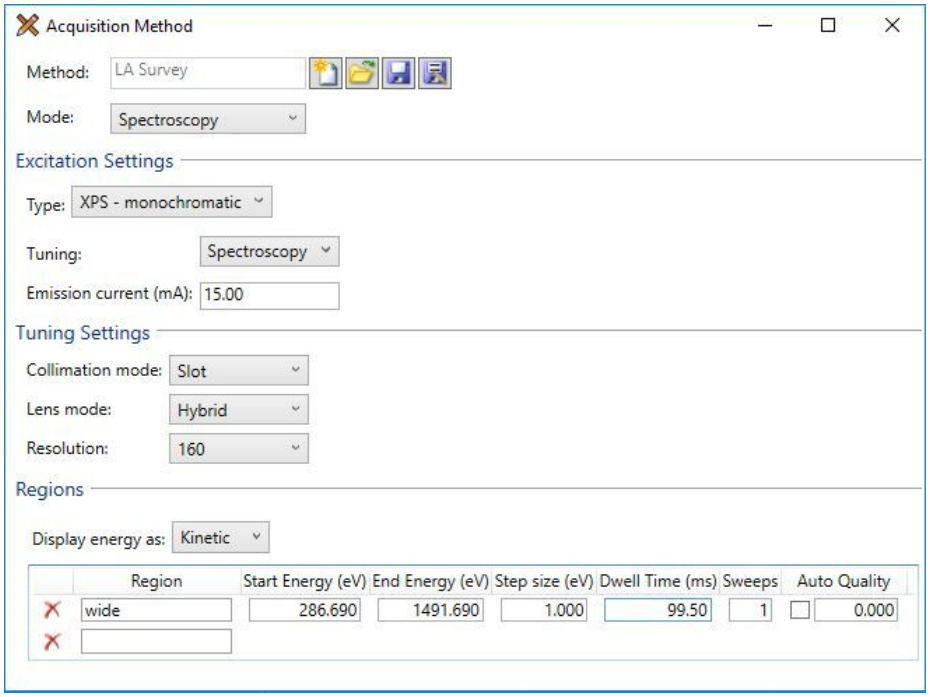
- **pinned** to the main window



To pin a window, click on the appropriate tab.
When a window is pinned it can be:

- detached and made to float .
- docked

- **detached** and made to float.



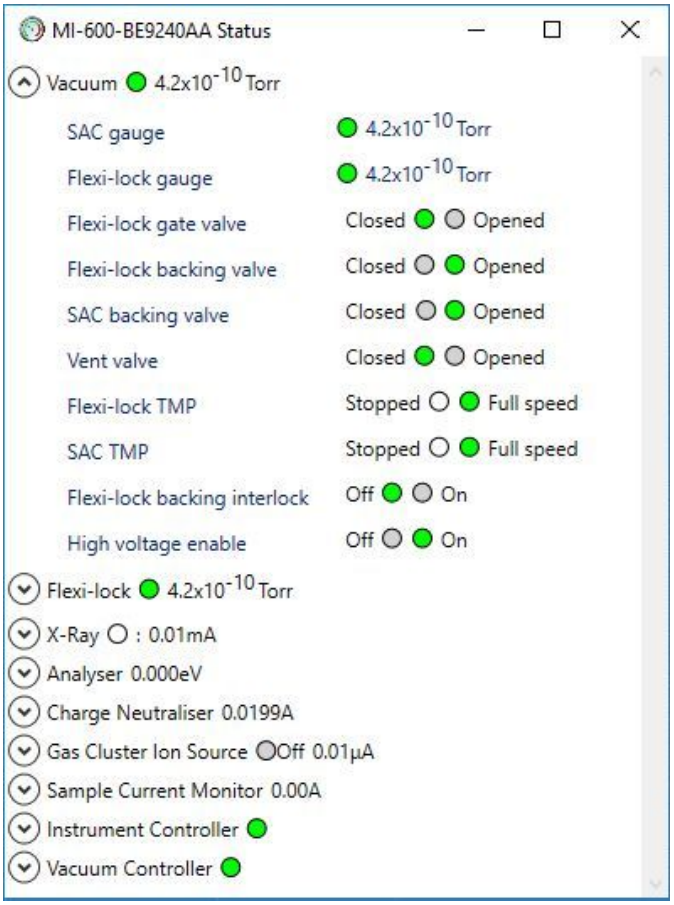
To float a window, click on the **Float** button  in the title bar. The window can then be moved by clicking on the title bar at the top of the window and dragging in the direction you want to move.

When a window is floating it can be:

- resized
- maximised to fill the whole screen
- if already maximised, restored to the previous size
- minimised to the Task bar
- closed which returns the window back to the docked state.

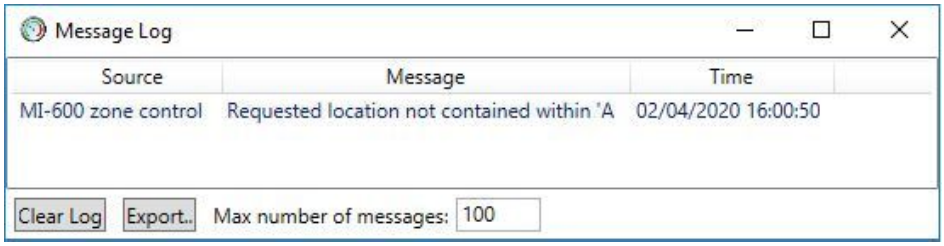
Status window

The Status window is displayed by clicking on the Status icon  in the bottom left hand corner of the main window. This window is not required for any procedures described in this manual but provides instrument readback information such as chamber pressures and whether the ion guns, X-ray guns, analyser or charge neutraliser are on. Further information can be found by expanding a section.



Message Log

The Message Log is displayed by right-clicking on the Status icon  in the bottom left hand corner of the main window and selecting **Instrument > Message Log**.



The maximum number of messages that are retained before the oldest message gets overwritten is set at the bottom of the Message Log.

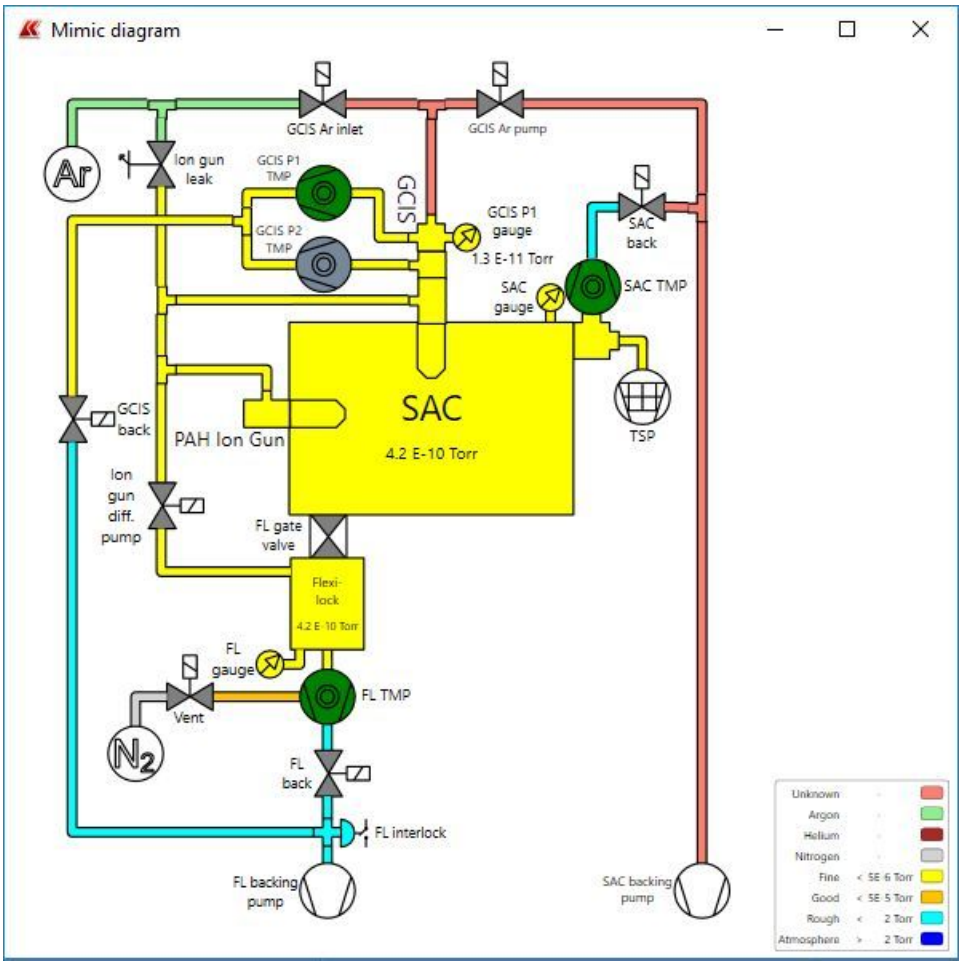
ESCApe™ Information

The ESCApe™ Information window is displayed by clicking on the **ESCApe™ Information** icon  in the bottom right hand corner of the main window. This window includes:

- the version of ESCApe
- a link to the Contact Us page on the Kratos website
- links to all ESCApe documentation located in the C:\User documents folder.

Mimic diagram

The **Mimic Diagram** window is displayed by right-clicking on the Status icon  in the bottom left hand corner of the main window and selecting **Instrument > Mimic diagram**.This comprises a real-time mimic diagram which can be used to monitor the vacuum level in various parts of the instrument.

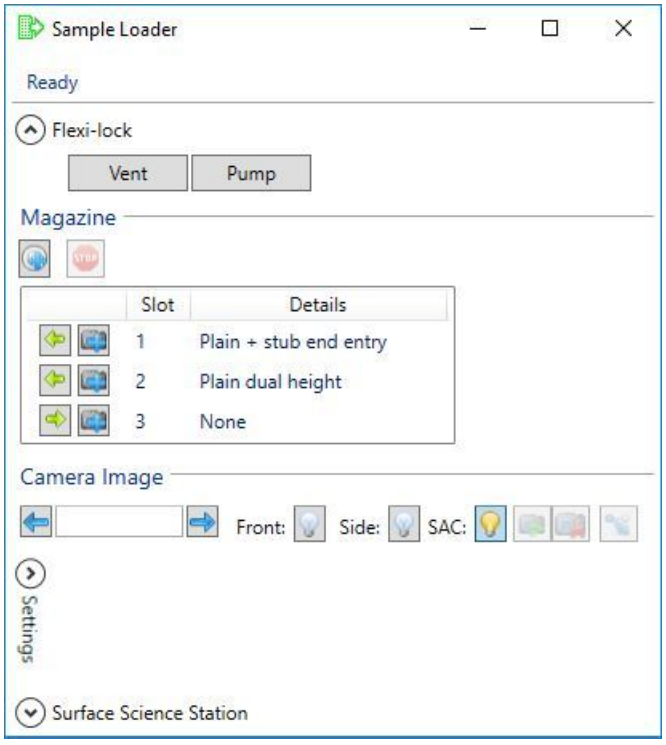


Chapter 3
Sample Handling

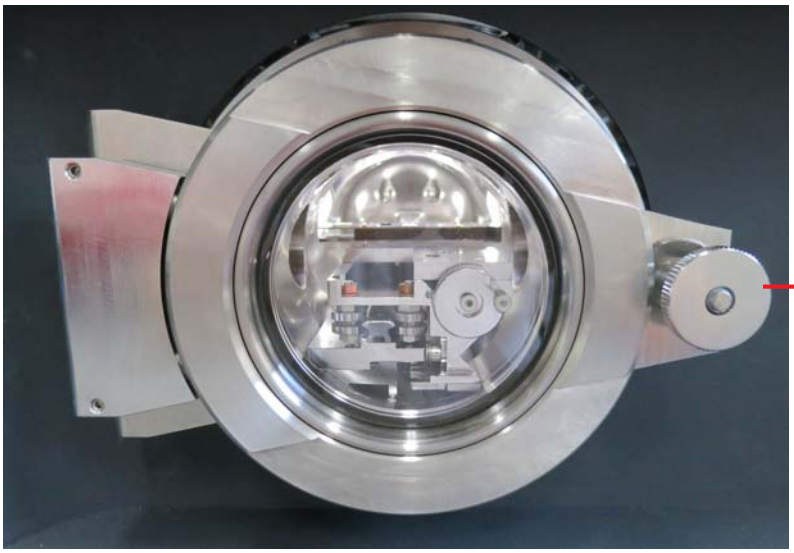
Venting the Flexi-lock

1. Open the **Sample Loader** window. This is located at the right hand side of the main window. This can be pinned to the main window or floated as shown below.

2. If necessary, click on the button to display the **Flexi-lock** controls.



3. Click on **Vent**.
4. Loosen the Flexi-lock swing bolt to prevent the Flexi-lock from over pressuring.



An automatic sequence vents the Flexi-lock and provides a flow of dry nitrogen. During the sequence a message is displayed stating **Venting Flexi-lock**. This changes to **Venting Flexi-lock completed** when the sequence is complete.

For your information

When the Flexi-lock reaches atmospheric pressure the door can be opened to enable sample transfer.

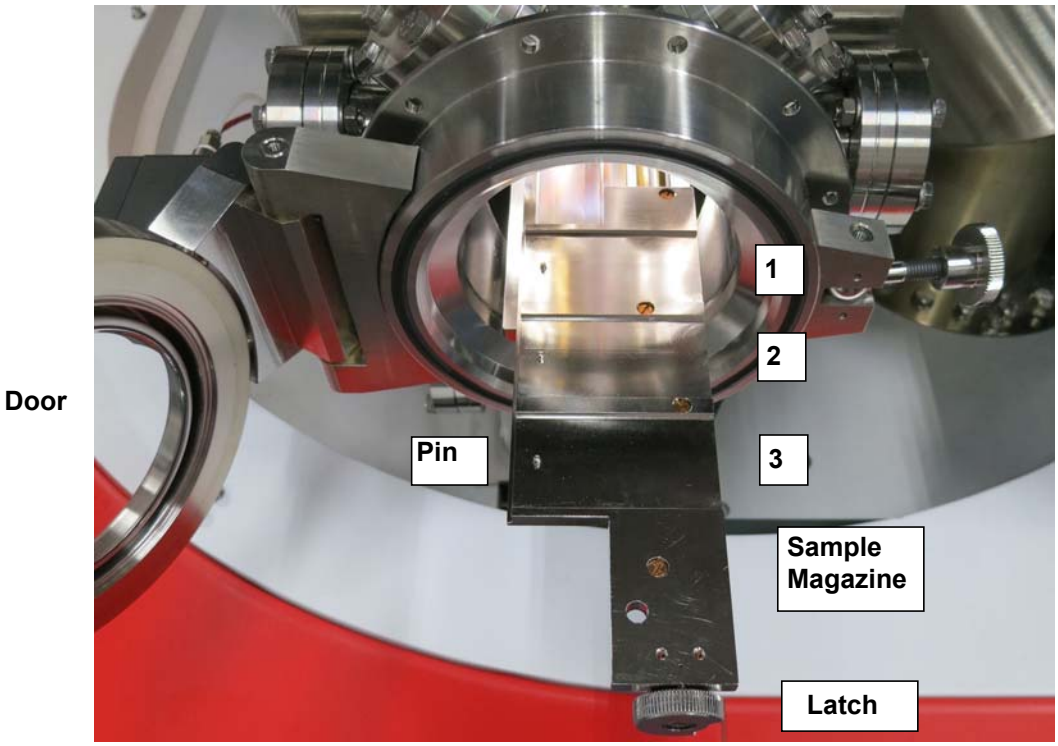
It is recommended that where possible sample holders are inserted and/or removed during the vent sequence.

Inserting and/or Removing Sample Holders

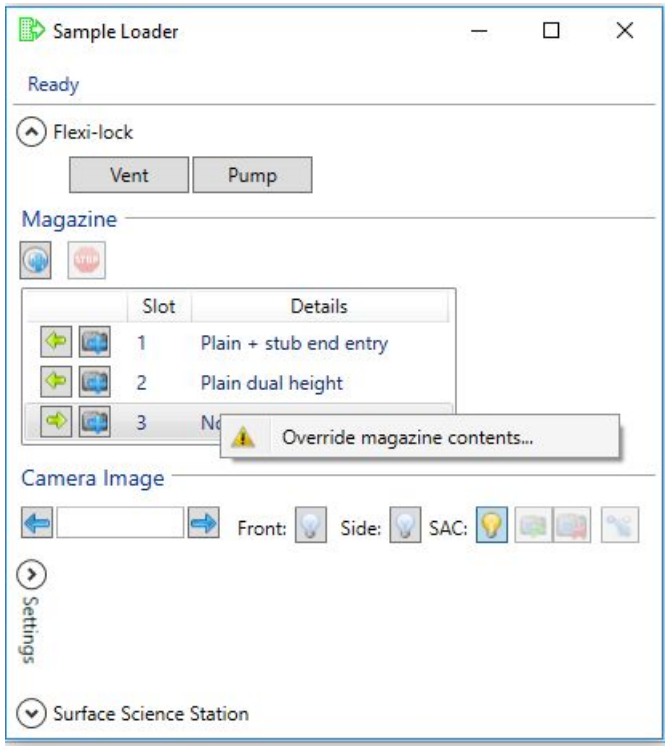
Sample holders are transferred to the analysis location on the sample magazine.

To insert and/or remove sample holders:

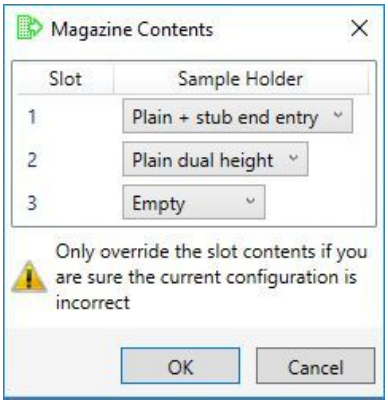
1. Move the Flexi-lock swing bolt to the side and open the door.
2. Turn the latch on the sample magazine clockwise and pull the magazine out until it reaches the stop.



3. Remove any sample holders by lifting them off the locating pin.
4. Purge the images, and their associated locations, of any sample holders which you have removed. Note that once a slot is set to empty it is not possible to reinstate the image.
   a. Open the **Sample Loader** window.
   b. Right click anywhere on the slot list.



c. Click on **Override magazine contents**. The **Magazine Contents** dialogue is displayed.



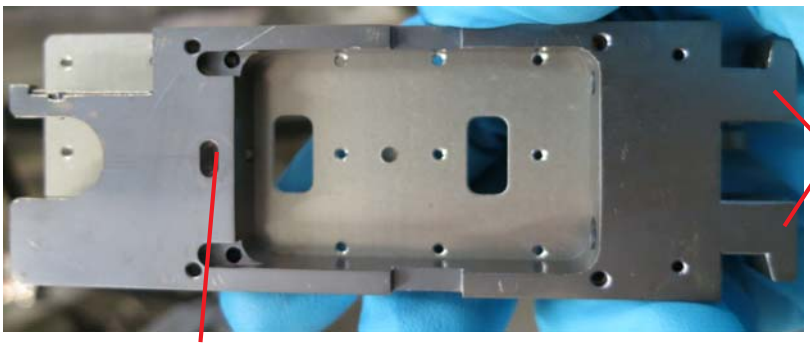
d. Set the contents of the slots from which you have removed the sample holders to **Empty**.

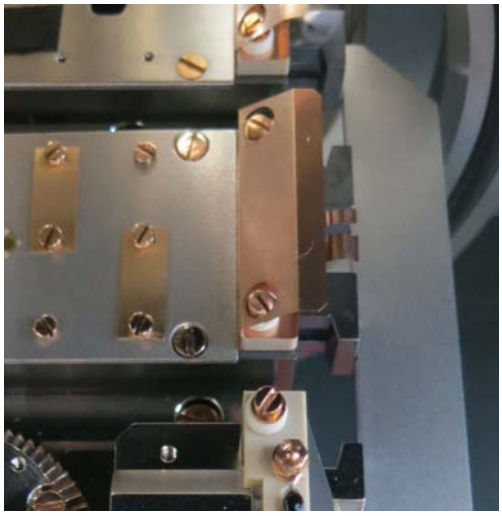
e. Click **OK**.

5. Place the holder in a slot on the magazine so that the locating hole on the bottom of holder fits on the pin and the holder is level. If the holder is correctly located it will not be possible to move the holder more than 0.2 mm from left to right.

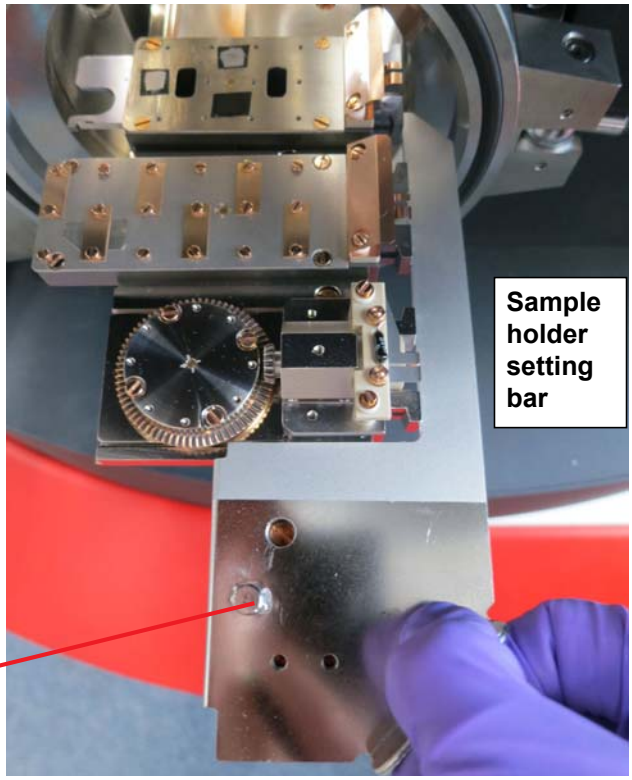
   Check that the holder is correctly located using the sample holder setting bar as shown below. The lugs on the holder should touch the setting bar. Remove the setting bar after use.

   Holders can be fitted in any of the slots (1, 2 or 3).





The dowel on the sample holder setting bar fits in the hole in the top of the magazine

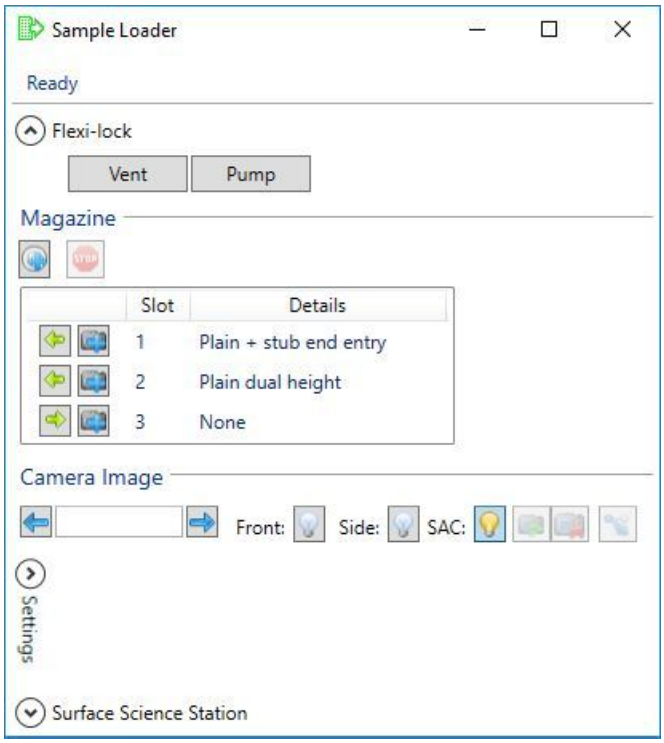


6. Push the magazine fully in.
7. Turn the latch on the magazine anti-clockwise to secure it.
8. Check that the magazine cannot be withdrawn.
9. Close the Flexi-lock door. Move the swing bolt into position and loosely secure it.

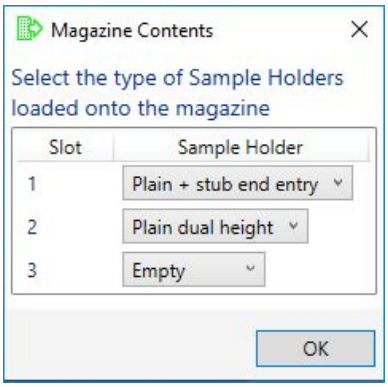
Pumping down the Flexi-lock

With the Flexi-lock swing bolt in position and loosely secured:

1. Open the **Sample Loader** window.
2. If necessary, click on the button to display the **Flexi-lock** controls.



3. Click on **Pump**. The **Magazine Contents** dialogue is displayed.



4. Select the type of sample holder that is loaded onto each of the slots. This can be:
   - **Plain dual height**
   - **Plain + stub end entry**
   - **Plain + stub side entry**
   - **Heat and Cool**
   - **Azimuthal**
   - **Contacting**.

   Note: The heat and cool, azimuthal and contacting sample holders are not covered in this document.

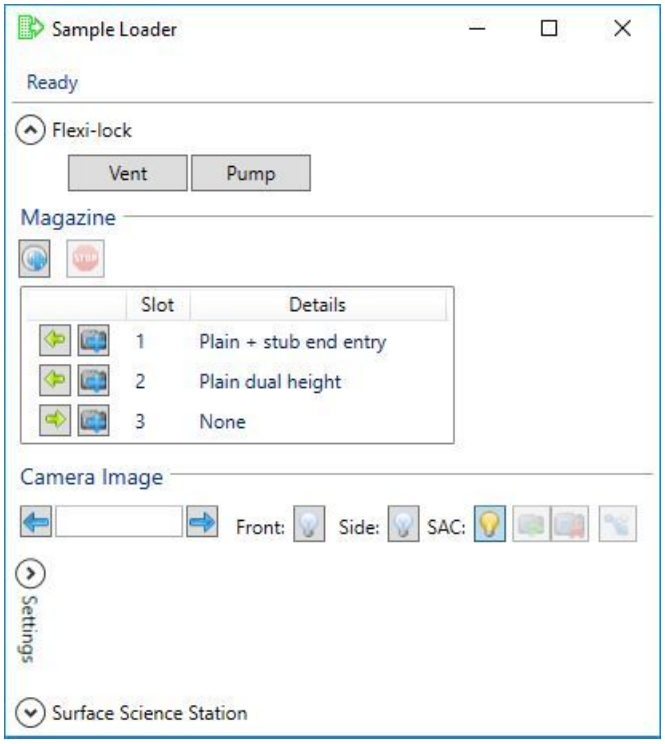
   An automatic sequence pumps the Flexi-lock. During the sequence a message is displayed stating **Pumping Flexi-lock**. This changes to **Pumping Flexi-lock completed** when the sequence is complete.

Obtaining an Image of a Sample Holder

An image is taken using a camera located in the Flexi-lock.This can then be used to define sample locations for analysis.

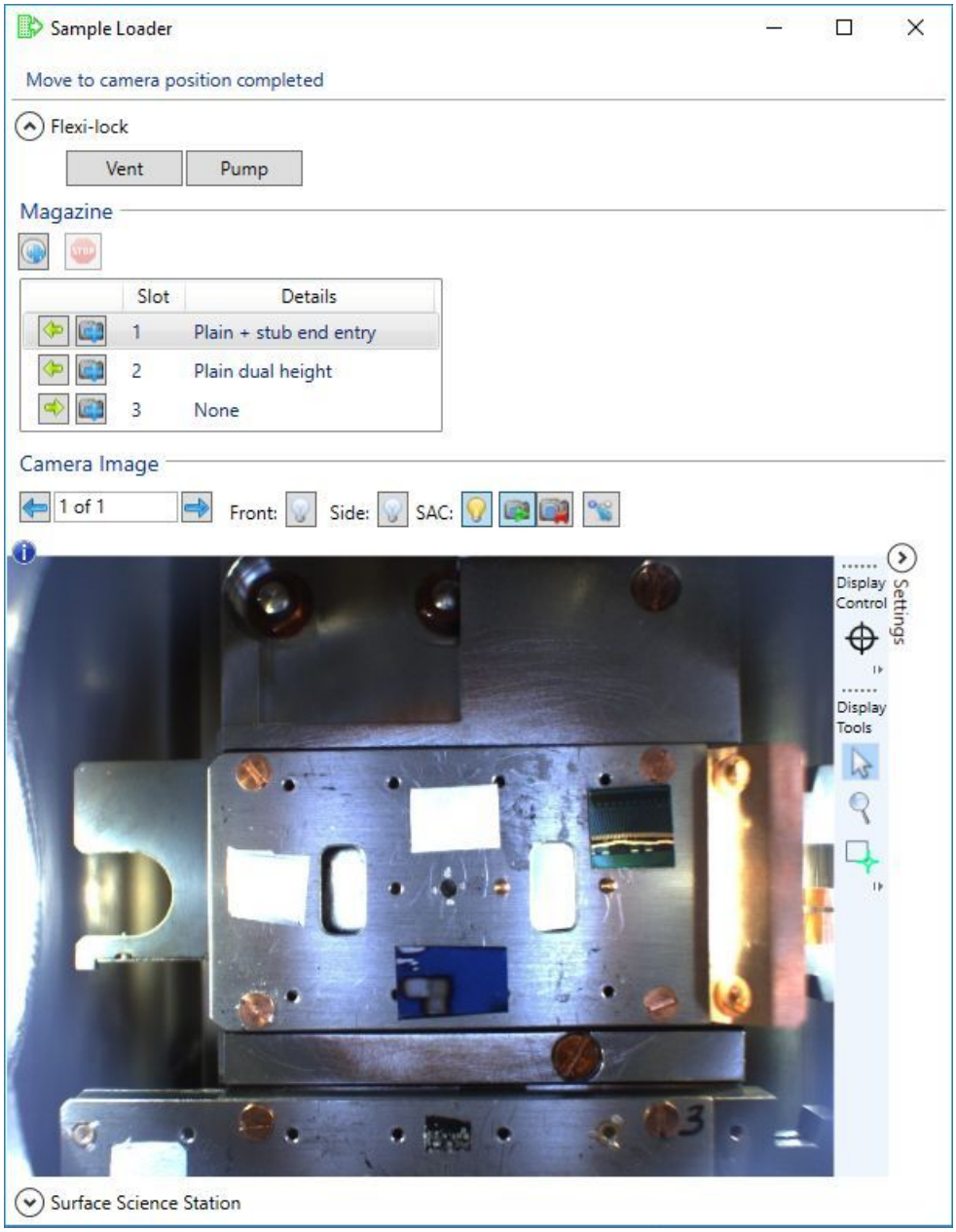
Note: The Flexi-lock/SAC gate valve must open for a sample holder in slot 3 to move to the camera position. This can only happen if the pressure difference between the Flexi-lock and the SAC is low enough.

1. Open the **Sample Loader** window.

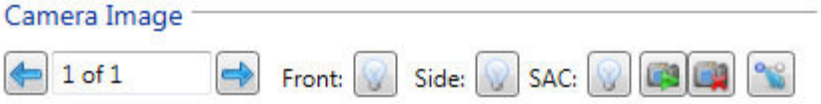


2. In the Magazine section, click the **Move to camera position** button  next to the relevant slot.
3. In the Camera Image section:
   a. Click the **Start loader camera** button  .
      The **Sample Loader** window now includes a live image of the sample holder.
      Note that the **Start loader camera** button changes to the **Stop loader camera** button.
   b. Choose the appropriate lighting by clicking the **Front** and/or **Side** buttons  .

   Note: Some instrument do not have side lights.



c. Click the **Stop loader camera** button  to save the image. The text **Image 1 of 1** is now displayed.



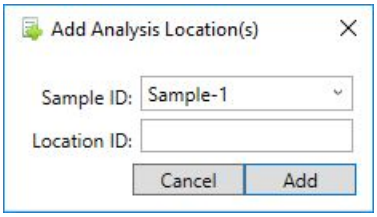
Creating Analysis Locations on a Sample Holder

For your information

This section describes a simple method for creating single locations. Other methods are described in the Advanced Operations User Manual.

1. Open the **Analysis Location** window.
2. Select the **Sample holder** to view the correct image.

3. Click the **Select multiple items** button  found to the right hand side of the image.
4. Click on the sample in the image to determine the location that will be used for analysis.
5. Click the **Add location** button  found below the locations table. The **Add Analysis Location(s)** dialogue is displayed.



6. In the **Sample ID** box, either:
   - Type the name of a new sample.
   - Select an existing ID from the drop-down list.
7. In the **Location ID** box, either:
   - Type the name of a location.
   - Leave the box blank - this gives the location the same name as the sample.
8. Click **Add**.

   A green marker is added to the image in this position and the location is added to the locations table. The Location ID is shown above the marker.
9. Repeat Step 4 to Step 8 to add more samples or locations.

For your information

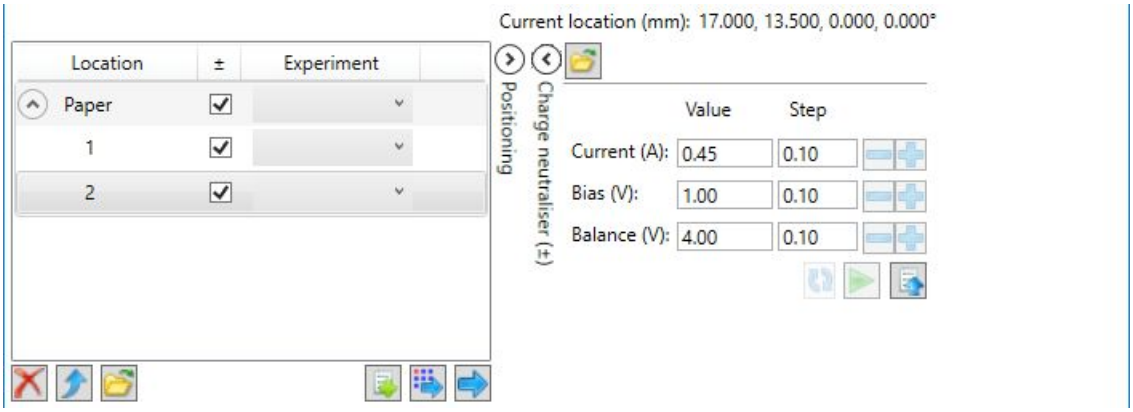
You can move the green marker and/or the Location ID. Select the **Interact with display items** button  , click on the marker or Location ID and click on the **Edit annotation geometry** button  . Now drag the top circle to move the Location ID or the bottom circle to move the green marker and the Location ID.

10. When you have finished adding samples or locations, deselect the **Select multiple items** button  .

Note: These locations do not take into consideration sample thickness. You must now obtain the correct height (Z value) using Auto Z or using the microscope.

Insulating samples require the use of the charge neutraliser. The status of the charge neutraliser can be set for each location using the **±** check box. The default state is with the charge neutraliser on.

If the check box is selected, the charge neutraliser is used when the sample is analysed. The settings that will be used can be seen by clicking on the **Charge neutraliser** reveal arrow .



The settings can be modified for all selected location(s). Type values in the text boxes associated with the three neutraliser parameters; filament **Current (A)**, filament **Bias (V)** and charge **Balance (V)**, then click on the **Update selected sample location** button  .

Settings can be copied from one location to other locations. First select the location with the required settings then use the Ctrl key to select the locations you want to change. Finally click the **Update selected sample location** button  .

For your information

Selecting a check box for the first time sets the charge neutraliser settings to the default values. If a check box is cleared and then re-selected the settings used are the values saved for the location.

Clicking the **Enable real time changes** button  turns the neutraliser on using the parameters displayed. The parameters can be modified by typing them in or using the increment buttons  . This allows the parameters to be optimised in real time and the settings updated to location(s) using the **Update selected sample location** button as described earlier.

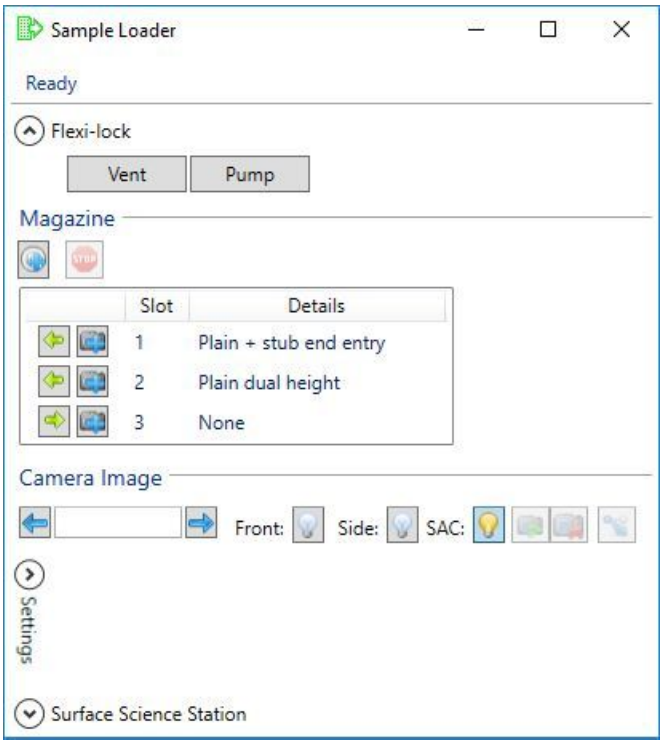
Transferring a Sample Holder

Loading a sample holder

Note: If you load a sample holder without first taking an image of the sample holder then an image is taken automatically as the holder enters the analysis chamber. The lighting and camera settings remain unchanged from the last time an image was taken.

To load a sample holder:

1. Ensure that the Stage is empty. If necessary unload the sample holder first. See "Unloading a sample holder" on page 41.
2. Open the **Sample Loader** window.



3. In the Magazine section, click the **Transfer the sample holder to the stage** button  next to the relevant slot.

   An automatic sequence transfers the sample holder to the Stage.

   Note: The transfer cannot occur if there is already a sample holder on the Stage and a failure message is displayed.

   During the transfer a message is displayed in the Instrument section stating **Transfer sample holder to stage**.

   When the transfer is complete this message changes to **Transfer sample holder to stage completed**.

Unloading a sample holder

1. Open the **Sample Loader** window.
2. Click the **Transfer the sample holder from the stage to the empty magazine slot** button  next to an empty slot.

   An automatic sequence transfers the sample holder to the Sample Magazine.

   During the transfer a message is displayed in the Instrument section stating **Transfer sample holder to magazine slot n**. When the transfer is complete this message changes to **Transfer sample holder to magazine slot n completed**.

Chapter 4
Acquiring Data

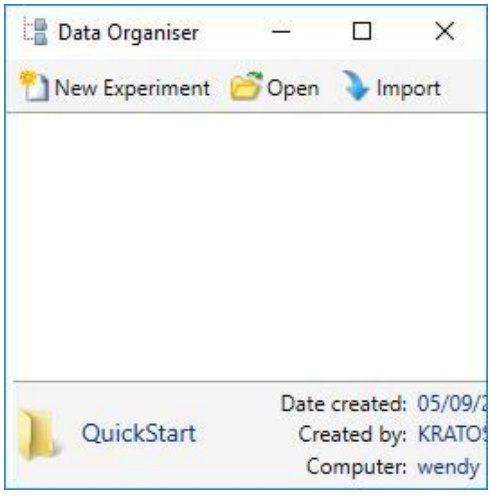
Experiment Files

Acquisition data is written to an experiment file (.experiment). Before you can submit an acquisition to the analysis queue you must create a new experiment file or open an existing experiment file in the **Data Organiser** window.

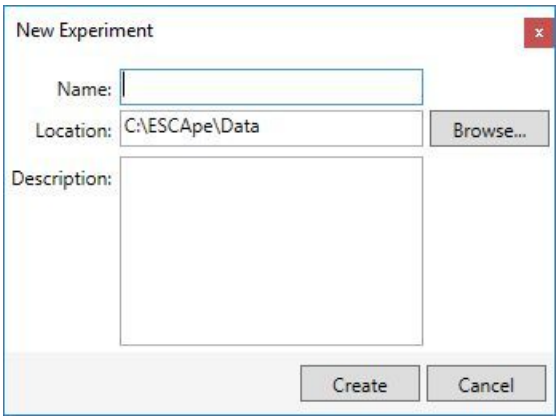
Creating an experiment file

To create an experiment file:

1. Open the **Data Organiser** window. Note that the content of this window depends on how you last used the window

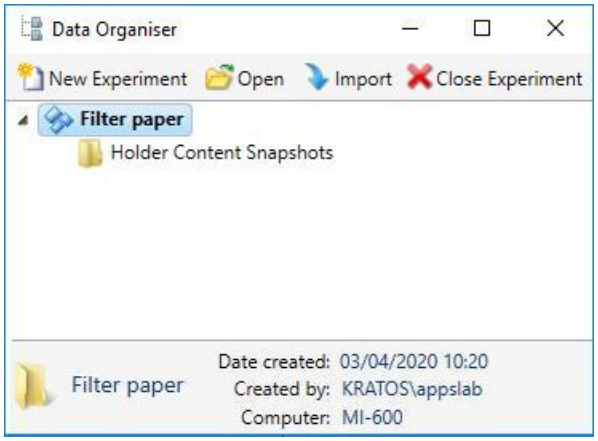


2. Click the **New Experiment** button  to display the **New Experiment** window.



3. Enter a **Name** for the experiment file.
4. If the **Location** needs changing, enter a new location for the file or use the **Browse** button to navigate to the location.
5. If required, enter a **Description**. This is included in the file properties.
6. Click **Create** to create the experiment file and close the **New Experiment** window.

   The experiment is loaded in the **Data Organiser** window.

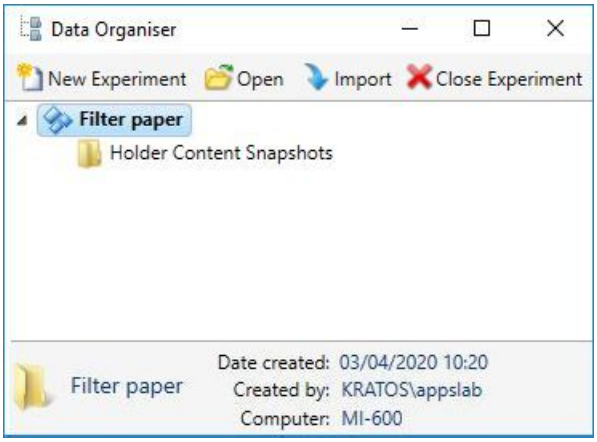


The Holder Contents Snapshots folder is a place holder for optical images. A new image is added to the folder after data has been acquired using a new sample holder image. Sample location markers are overlaid on the image for any location that has data acquired using the optical image and the experiment file.

Opening an experiment file

To open an experiment file:

1. Open the **Data Organiser** window



2. Click the **Open** button .to display the **Open Experiment** window.
3. Navigate to the experiment file.
4. Either:
   - Double-click on the file.
   - Click **Open**.

Acquisition Methods

The **Acquisition Method** window is used to select the settings that are used for analysis. The settings can be selected by opening an acquisition method file (.acquisition) or by selecting each value individually.

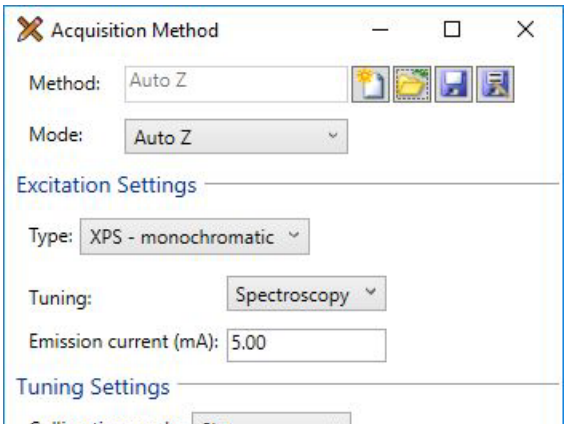
Kratos methods

Standard methods are provided in the C:\ESCApe\Methods\Kratos Methods folder. These give reasonable starting points for commonly used acquisition methods such as large area survey spectra.

The following sections describe how to use these standard methods.

Opening a method

1. Open the **Acquisition Method** window. Note that the content of this window depends on how you last used the window.



2. Click the **Open Method** button  to display the **Open Acquisition Method** window.
3. Navigate to the method.
4. Either:
   - Double-click on the method.
   - Select the method and click **Open**.

Changing a method

For your information

The settings displayed in the **Acquisition Method** window at the time an acquisition is submitted are the settings used for analysis. Changes do not need to be saved unless you want to keep the new values to use again.

If the values provided in a method need changing then

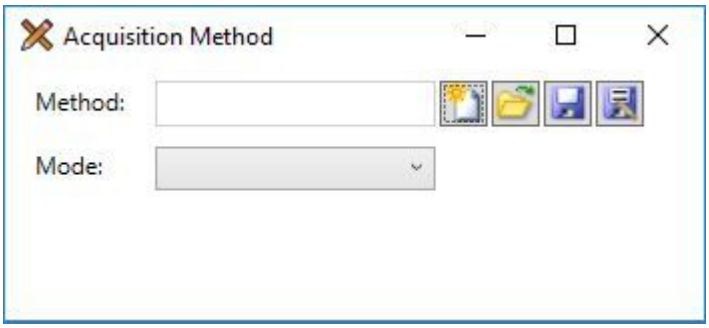
1. In the **Acquisition Method** window, click the **Open Method** button  to display the **Open Acquisition Method** window.
2. Open a method.
3. Amend the parameters as required.
4. If required, either:

   - Click the **Save** button  to overwrite the existing method. Note that the standard methods are read only files.
   - Click the **Save As** button  to display the **Save Acquisition Method** window. Navigate to the folder where you want to save the method. Enter the **File Name** and click **Save**.

Creating a new method

Note: If your new method is similar to an existing method it may be easier to amend the existing method and save it as a new method. See "Changing a method" on page 47.

1. In the **Acquisition Method** window, click the **Create a New Method** button  . This clears the current settings.



2. Select a **Mode**. Appropriate parameters are displayed. The exact parameters depend on the selected mode.
3. Set the parameters as required using the drop-down menus and text boxes.
4. Click the **Save** button  to display the **Save Acquisition Method** window.
5. Navigate to the folder where you want to save the method.
6. Enter the **File Name** and click **Save**.

Analysing Large Areas

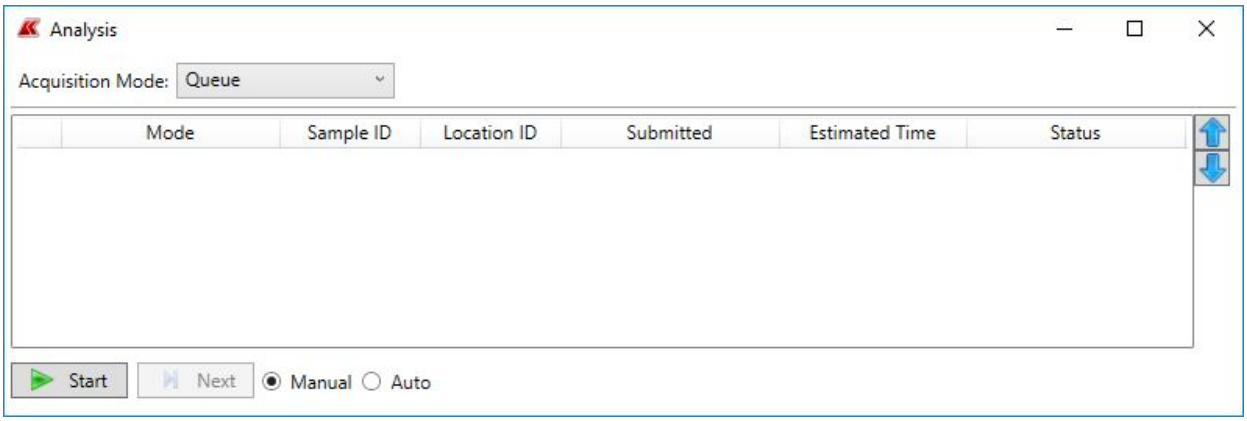
This section assumes that you have:

- loaded a sample to the instrument. This can be located in the Flexi-lock or on the stage.
- taken a photograph of the sample holder. See "Obtaining an Image of a Sample Holder" on page 34.
- defined analysis locations. See "Creating Analysis Locations on a Sample Holder" on page 36.

Auto Z with a survey sequence

The Auto Z+survey sequence method starts by finding the optimum analysis height for a sample location using spectroscopy. A survey spectrum is then acquired using this analysis height.

1. In the **Analysis** window, set the **Acquisition Mode** to Queue.



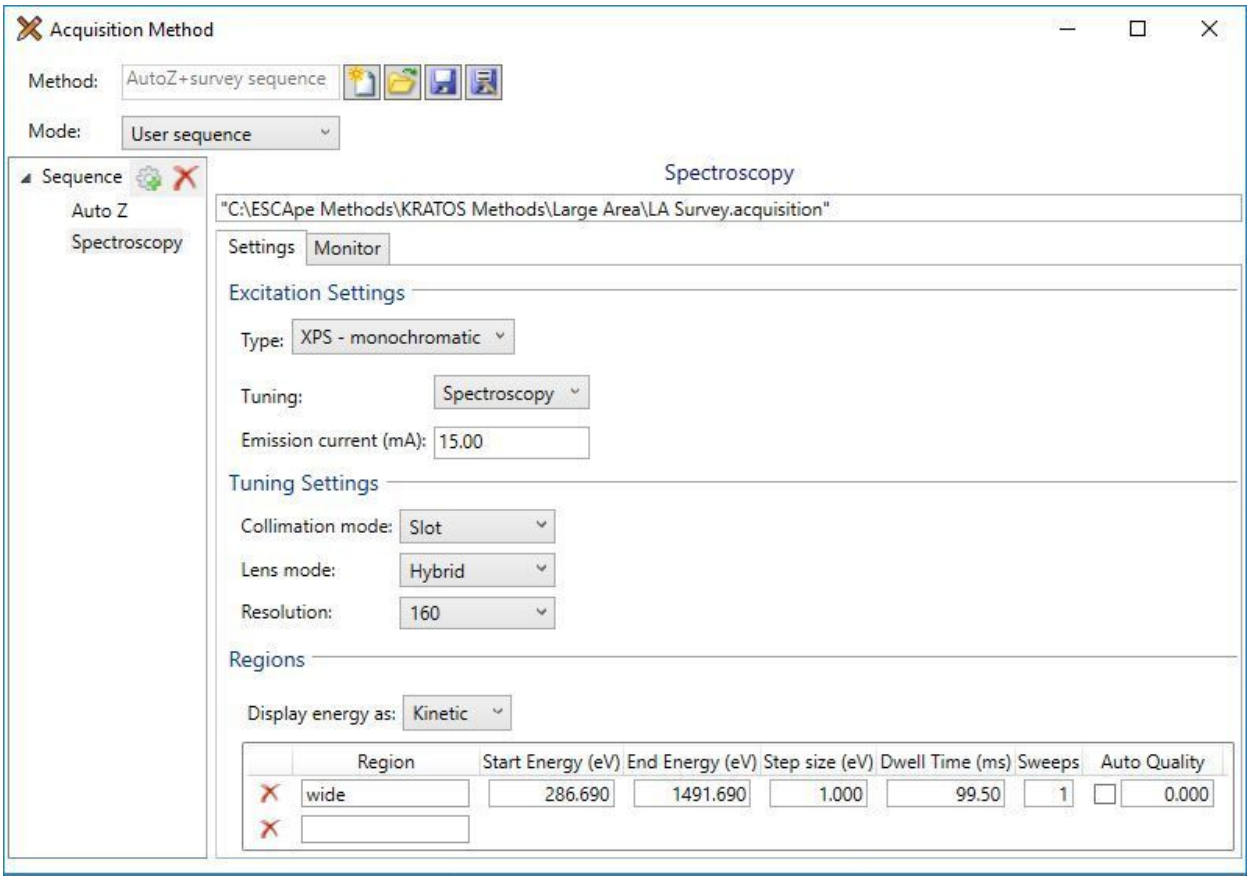
2. In the **Acquisition Method** window, click the **Open Method** button  to display the **Open Acquisition Method** window.

For your information

Standard methods are provided in the C:\ESCApe\Methods\Kratos Methods folder. This includes an XPS Large Area sub folder containing the ***Auto Z+survey sequence*** method.

The large area methods acquire information from a 700 x 300 µm area on the surface of the sample.

3. Open the ***Auto Z+survey sequence*** method.



For your information

These settings have been selected as a reasonable starting point.

The **Emission current** can be increased to give a larger count rate. However the resultant higher X-ray power may cause degradation of delicate samples and saturation of the signal observed by the detector. Also if high count rates are acquired for long periods then the lifetime of the detector is shortened. Take particular care when analysing pure metals as the intensity can be very high.

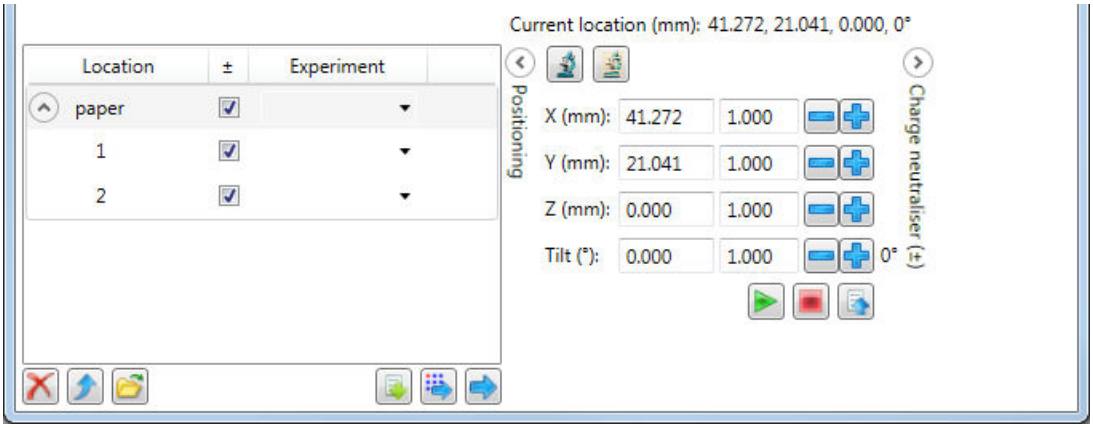
4. In the **Analysis Location** window select the **Sample holder** with the samples you want to analyse. The sample holder may be on the stage or the magazine.

   Then select an **Experiment** file for each location that you want to analyse.

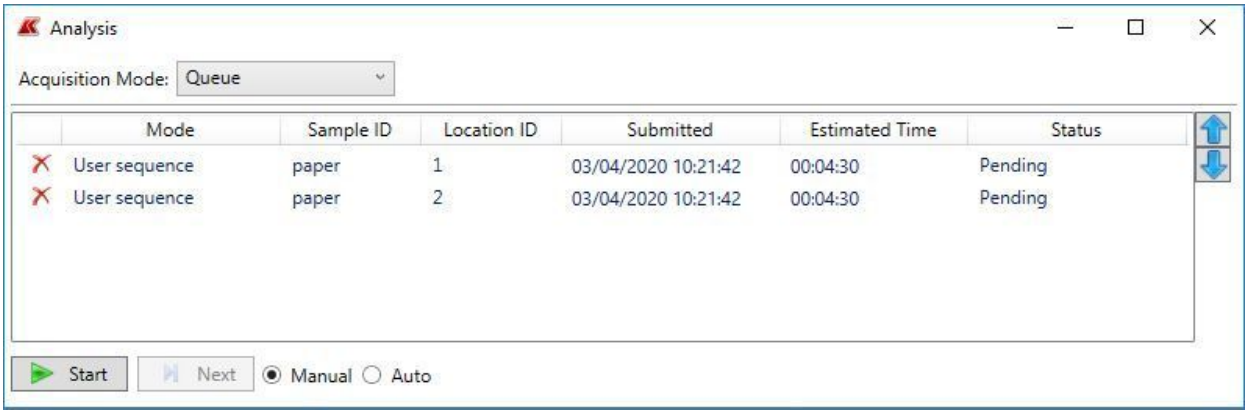
5. In the **Analysis Location** window, either:

   - Click the **Submit location for acquisition** button to submit a location. This button is displayed when you hover your mouse pointer over a location in the locations table.
   - Select a number of locations in the locations table then click the **Submit selected locations for acquisition** button found below the locations table. These locations can be on the same sample or different samples.

     If there are multiple locations on a sample, you must ensure that the list of locations is expanded before selecting them. You can select contiguous locations using the Shift key and non-contiguous locations using the Ctrl key.



The location(s) are submitted to the queue for analysis. If you have submitted multiple locations then each location has a separate entry in the queue and is analysed separately.



For your information

Any pending acquisitions can be reordered using the or arrows.

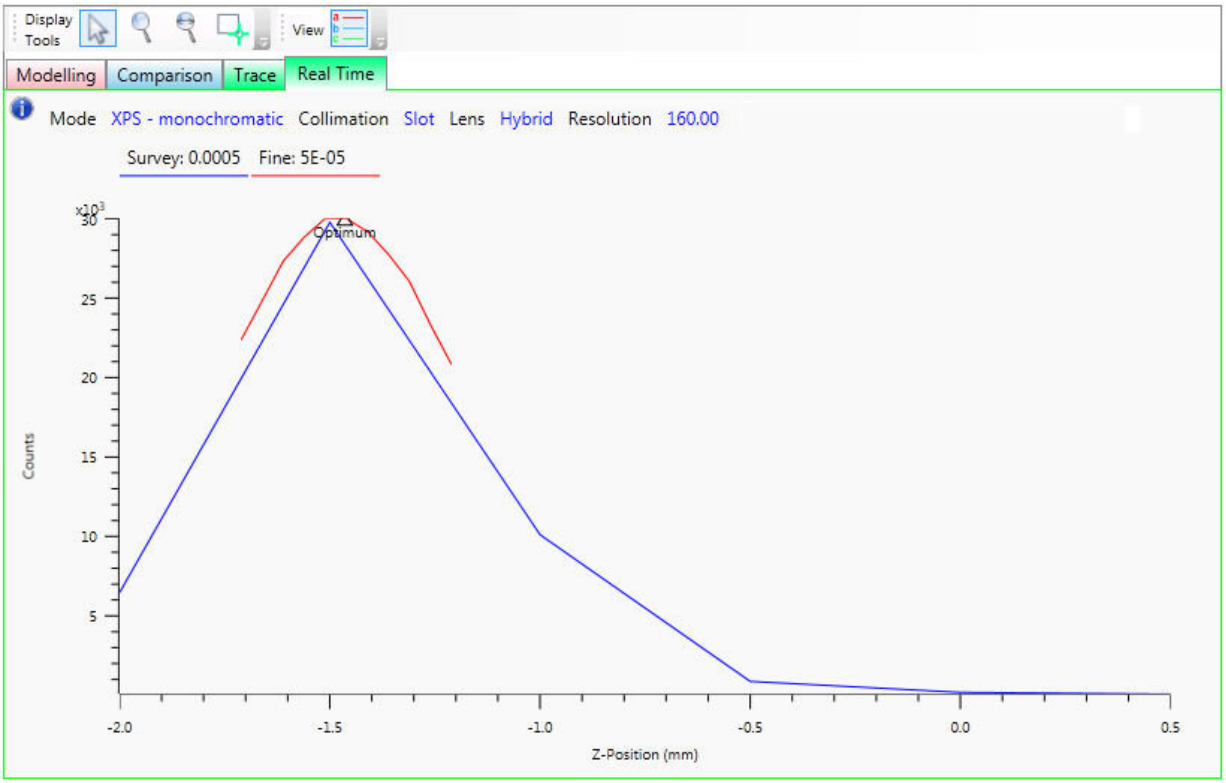
6. If the **Manual** radio button is selected in the **Analysis** window, start the acquisition using one of the following methods:
   - Click the **Start** button. This starts the first acquisition. If you have more than one entry in the queue you have to click the **Start** button for each entry.
   - Select the **Auto** radio button and then click the **Start** button. If this radio button is selected then each entry in the queue starts automatically.

For your information

If the sample holder is located in a slot on the sample magazine then the holder is transferred automatically to the stage for analysis. If there is a sample holder on the stage then this is unloaded first.

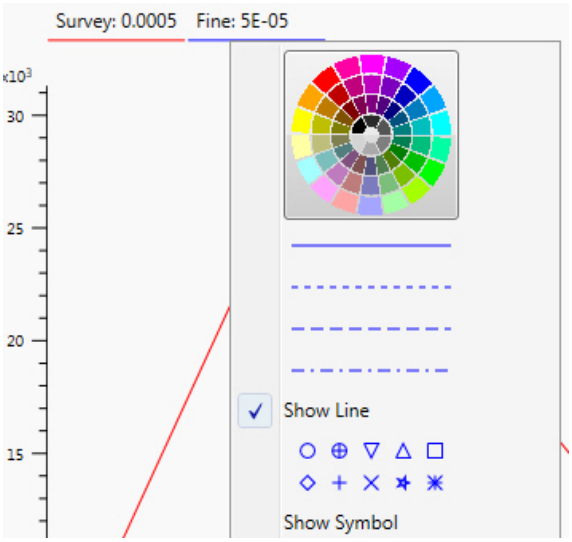
7. Select the **Real Time** tab to see the traces.

   The Auto Z+survey sequence method starts with a coarse trace which takes a snapshot every 0.5 mm. This is followed by a finer trace which takes a snapshot every 0.05 mm round the peak intensity to fine tune the optimum Z value.

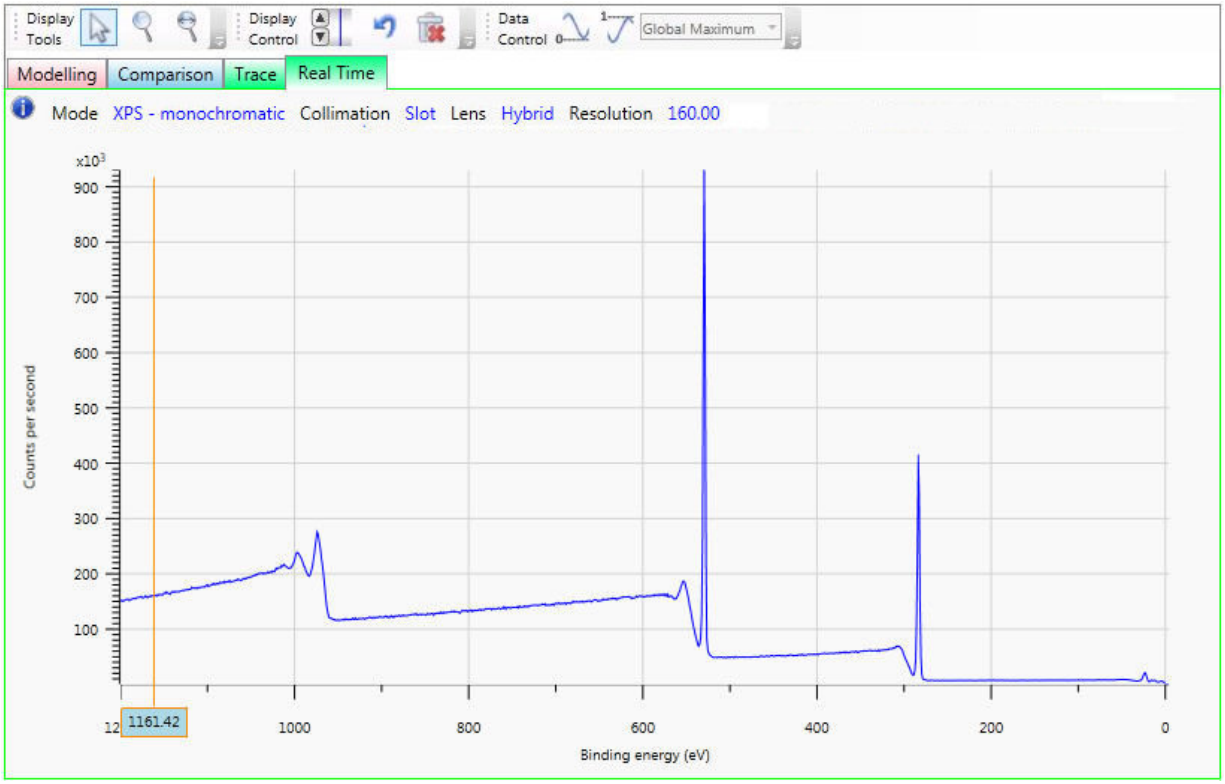


For your information

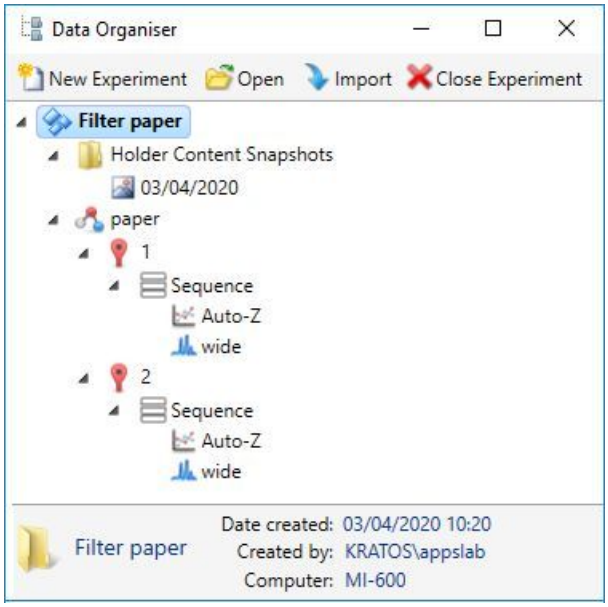
The line can be changed by right clicking on the line at the top left of the display area. This displays a colour wheel. Select a new colour and a line type or select a symbol to display the data points.



The Z position is automatically updated with the optimum value and a survey spectrum is acquired.



As the Auto Z+survey sequence routine is carried out data is added to the experiment file which was selected for the location at the time the location was submitted for analysis. For example, if the Auto Z+survey sequence is carried out on locations 1 and 2 on the paper sample and the same experiment file was selected for both locations the experiment file has data added as follows:



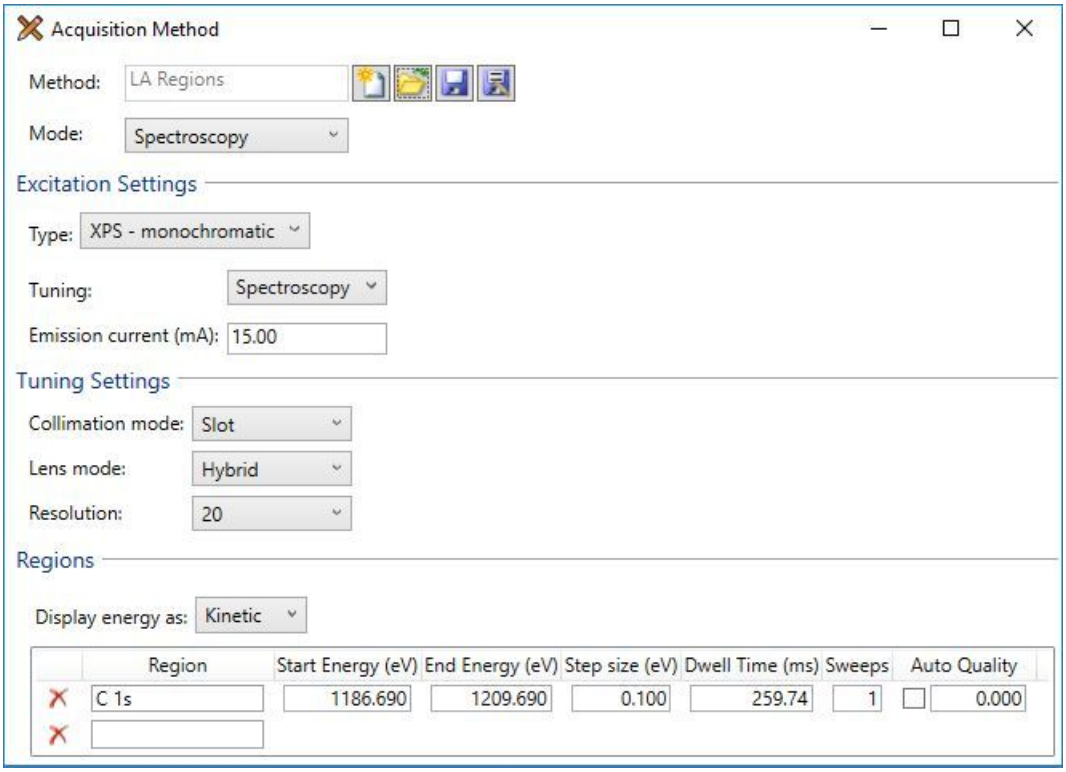
Acquiring large area high resolution region spectra

1. In the **Analysis** window, set the **Acquisition Mode** to Queue.
2. In the **Acquisition Method** window, click the **Open Method** button  to display the **Open Acquisition Method** window.

For your information

Standard methods are provided in the C:\ESCApe\Methods\Kratos Methods folder. This includes an XPS Large Area sub folder containing the LA Regions method for running a large area region scan.

3. Open a Regions method.



For your information

These settings have been selected as a reasonable starting point.

The **Resolution** can be reduced to give a better energy resolution although this gives a lower count rate. This is useful if there are two peaks close together. Conversely the Resolution can be increased to give a lower energy resolution with a higher count rate. This is useful if there is a trace element.

4. Set up the required regions. You can:
   - delete a row by clicking on the X at the beginning of the row.
   - type in an element line in an empty **Region** box and press Enter on your keyboard. This also gives a new line in the Regions table.
   - edit an entry in a **Region** box and press Enter on your keyboard.

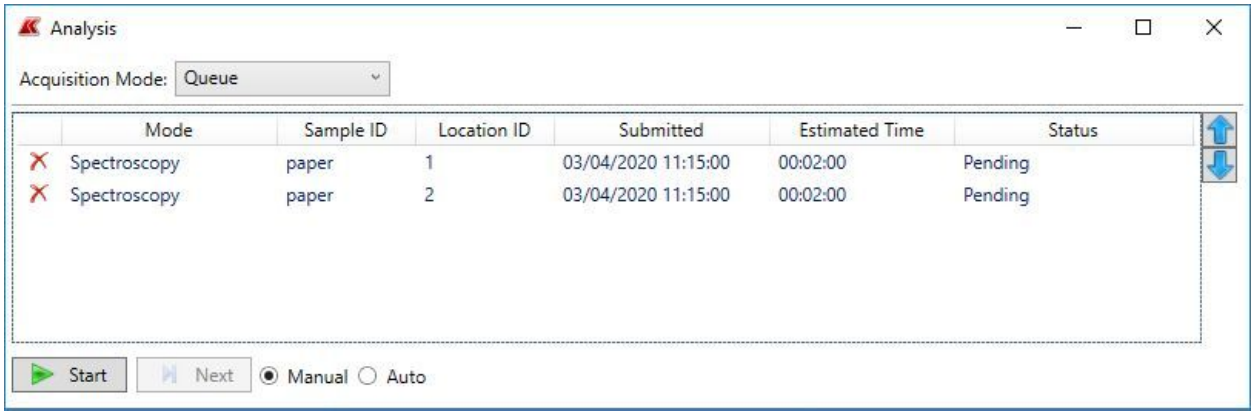
For your information

Right-clicking on the following column headings in the regions table gives a pop-up menu which allows you to change the table:

Column	Menu options
Second or third	Start and End Centre and Width
Fourth	Step Size Number of Steps
Fifth	Sweep Time Dwell Time

If required, the **Auto Quality** function can be used to acquire spectra to a specified signal to noise ratio. Selecting the **Auto Quality** check box, automatically populates the number of **Sweeps** and the **Auto Quality** value (although these can be changed). After the first sweep the quality value is calculated by taking the spectrum maximum and subtracting the spectrum minimum and then dividing this by the peak-to-peak noise. If this value exceeds the **Auto Quality** value no more sweeps are acquired. If not, the acquisition continues with another sweep before recalculating. If the number of **Sweeps** is reached before the **Auto Quality** value then the acquisition terminates. This ensures that the acquisition terminates in the case of low intensity peaks. The calculated Auto quality value is saved with the data and displayed in the header.

5. In the **Analysis Location** window select the **Sample holder** with the samples you want to analyse. The sample holder may be on the stage or on the magazine. Then select an **Experiment** file for each location that you want to analyse.
6. In the **Analysis Location** window, either:
   - Click the **Submit location for acquisition** button  to submit a location. This button is displayed when you hover your mouse pointer over a location in the locations table.
   - Select a number of locations in the locations table then click the **Submit selected locations for acquisition** button  found below the locations table. These locations can be on the same sample or different samples.



The location(s) are submitted to the queue for analysis. If you have submitted multiple locations then each location has a separate entry in the queue and is analysed separately.

7. If the **Manual** radio button is selected in the **Analysis** window, start the acquisition using one of the following methods:
   - Click the **Start** button. This starts the first acquisition. If you have more than one entry in the queue you have to click the **Start** button for each entry.
   - Select the **Auto** radio button and then click the **Start** button. If this radio button is selected then each entry in the queue starts automatically.

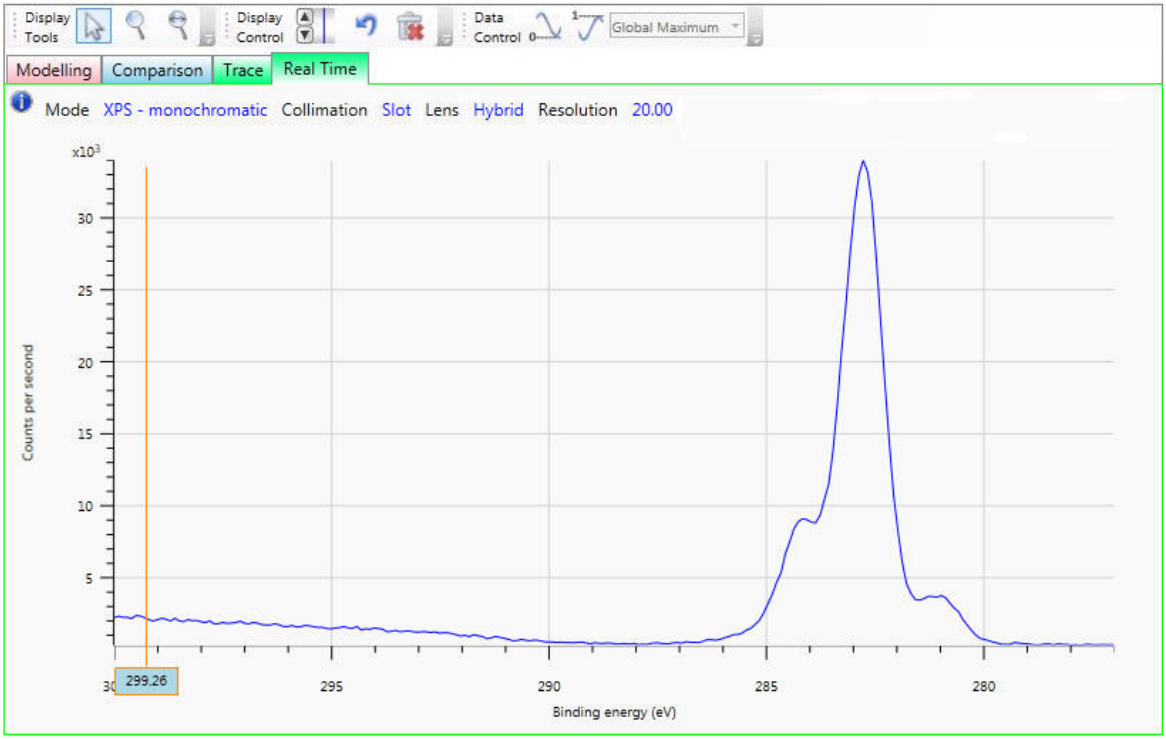
For your information

If you have set up more than one region then a spectrum is acquired for each region. These spectra are acquired in descending BE/ ascending KE order.

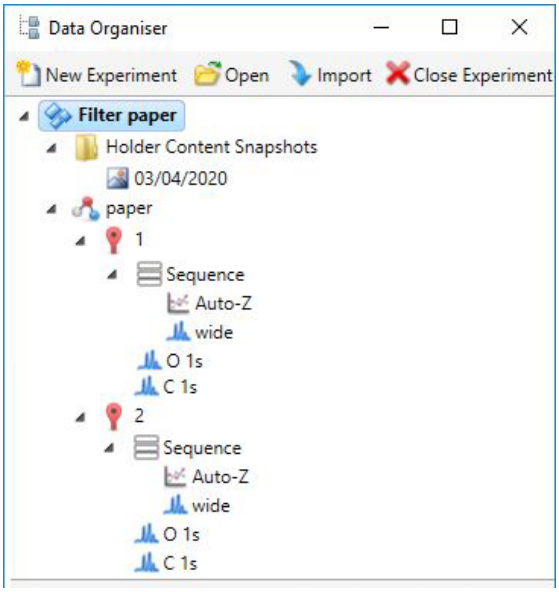
If you have set up more than one sweep then a sweep is carried out of each region in turn. For example, if you set up 4 sweeps of C 1s and 2 sweeps of O 1s and Si 2p then the sweeps would be carried out in the following order:

O 1s, C 1s, Si 2p, O 1s, C 1s, Si 2p, C 1s, C 1s

8. Select the **Real Time** tab to see the spectrum.



9. As region spectra are acquired data is added to the experiment file(s) which were selected at the time the locations were submitted for analysis. The data is added using the names of the regions. For example, if region spectra are acquired for O 1s, and C 1s at locations 1 and 2 on the paper sample, and the same experiment file was selected for both locations, the experiment file has data added as follows:



Analysing Small Features

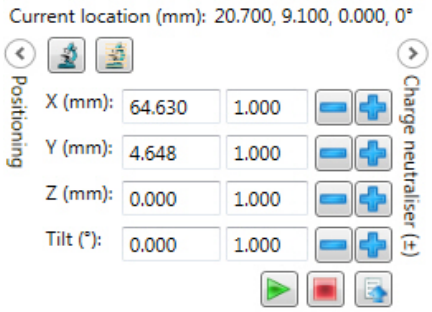
This section assumes that you have:

- loaded a sample to the instrument.
- taken a photograph of the sample holder. See "Obtaining an Image of a Sample Holder" on page 34.
- defined analysis locations. See "Creating Analysis Locations on a Sample Holder" on page 36.
- transferred the sample to the stage. See "Transferring a Sample Holder" on page 40.

Using microscope images to set up small features

This method can be used to obtain the correct height (Z value) of a small feature and to refine the X and Y values.

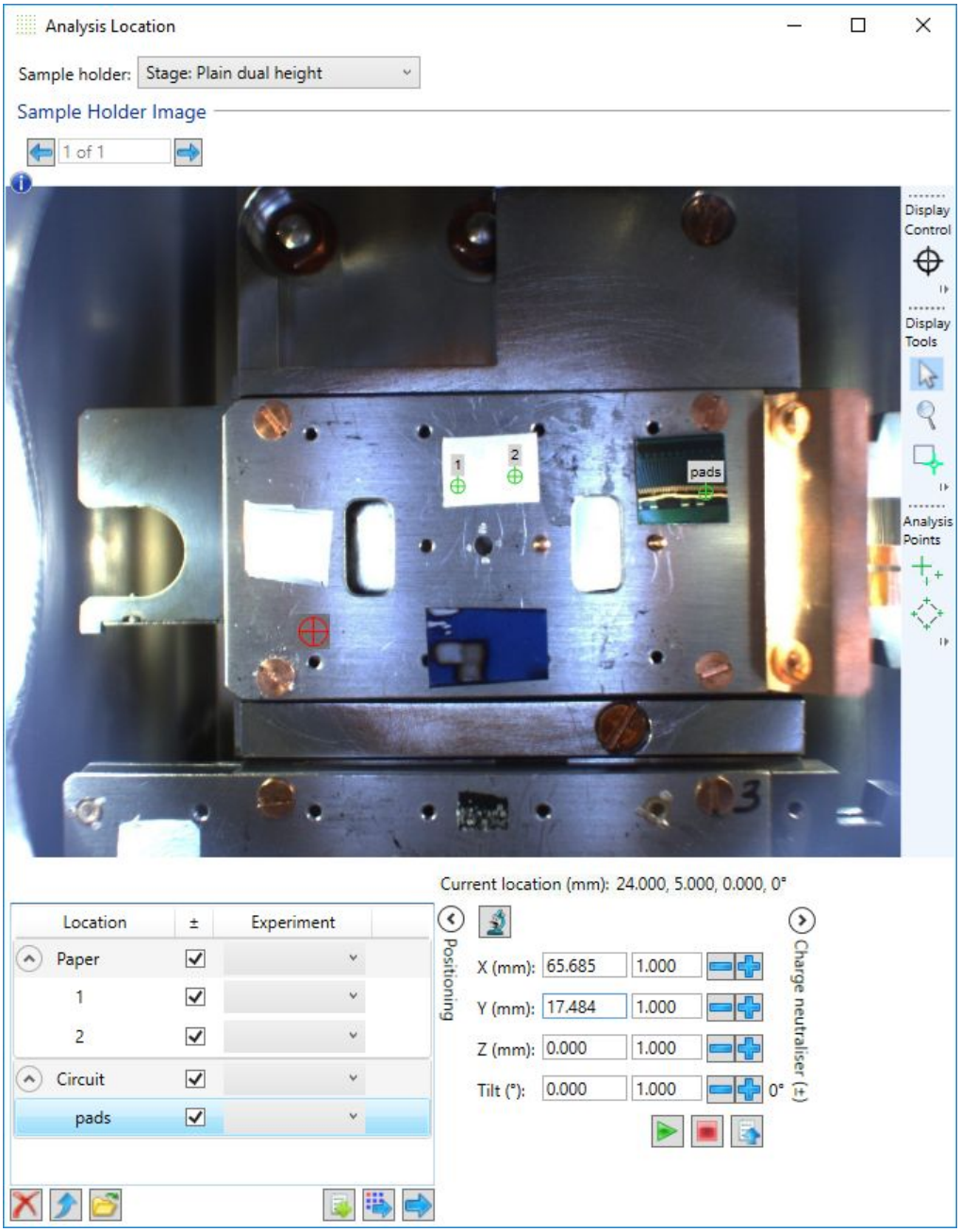
1. Create a sample location close to the feature of interest as described in "Creating Analysis Locations on a Sample Holder" on page 36.
2. If the sample holder is not already on the stage then transfer the sample holder as described in "Transferring a Sample Holder" on page 40.
3. Open the **Analysis Location** window. Ensure that the correct **Sample holder** is displayed.
4. If necessary, click on the button to display the **Positioning** controls.



The first column gives the target sample holder co-ordinates. The second column gives the required increments.

The current analysis location is shown by the on the image. Its co-ordinates are given above the Positioning controls.

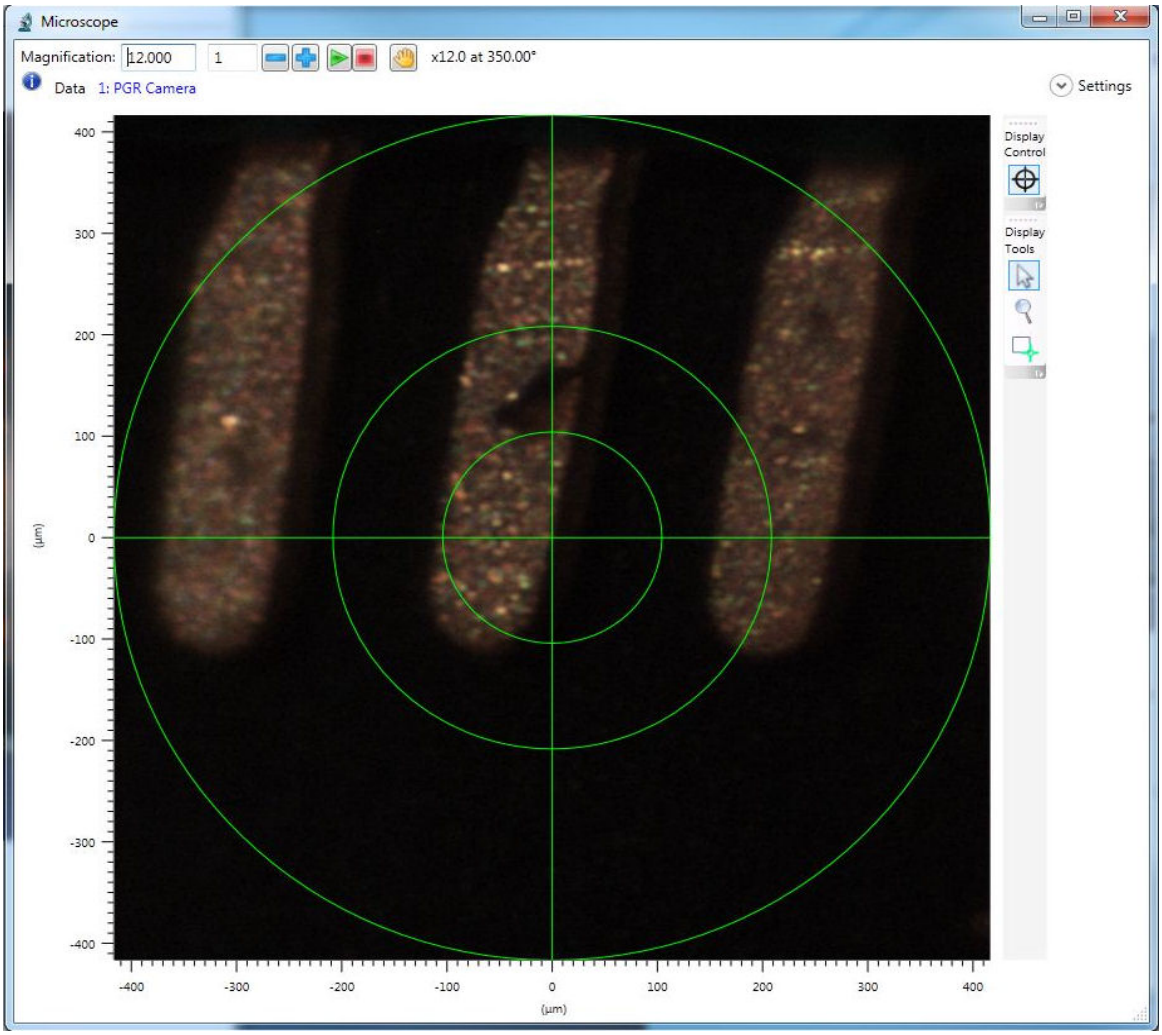
5. Select the required location from the locations table. If necessary, click on the arrow to the left of the Sample Id to list all the locations on the sample. This updates the target sample holder co-ordinates (first column) in the Positioning controls.



6. Click the **Move to sample bar location** button  found below the Positioning controls.

   The stage moves so that the selected location is in the analysis position.

7. Click the **Display microscope** button  to display the **Microscope** window. Click the  button to display the cross hairs.



The magnification can be changed to any value between 1 and 12.

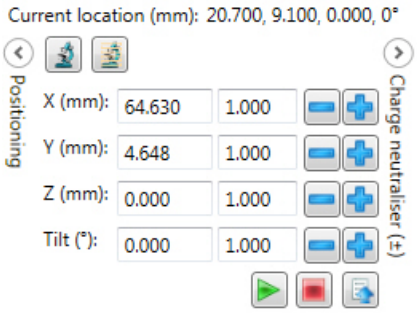
Either:

- Enter the required magnification in the first box and click the button 
- Click the  or  button to change the magnification by the increment in the second box.

For your information

It is easier to start with a low magnification and then increase the magnification when the feature is better focussed.

8. Now use the microscope image in the **Microscope** window and the Positioning controls in the **Analysis Location** window to accurately position the sample.



The target sample holder co-ordinates can be changed as follows:

- Enter a value manually in the first column and then click the button  .
- Hover over a box in the first column and use the middle mouse button to change the co-ordinate by the value of the increment box. Roll the middle mouse button forward to increase the value and towards you to decrease the value.
- Click the  or  button to change the associated target co-ordinate by the value of the increment.

For example, clicking on the  button next to the Z co-ordinate in the screenshot gives the new Z co-ordinate as -1.000. The other co-ordinates are unchanged.

If you try to change a target sample holder co-ordinate to a value outside the analysis zone then a **Stage Move** error message is displayed.

At high magnifications there is a band of focus across the image. Adjust the Z control so that the band of focus is in the centre of the image. This places the sample at the correct analysis height. As the camera is mounted at a slight angle adjusting the Z control moves the feature across the image in the X direction. Now adjust the X and Y controls until the feature and the band of focus are in the centre of the image.The feature is now at the correct analysis position.

For your information

The microscope focus is pre-set at the analysis position. When the sample holder is transferred to the stage this position corresponds with the top surface of the sample holder. The sample holder needs to be moved up or down to account for the height of the sample. For example, if a 3 mm high sample is surface mounted then the sample holder needs to be moved down by 3 mm. This means that the Z control needs to be moved to - 3.

9. Click the **Update the currently selected location with the above position** button  to update the position.
   This position can now be used for analysis.

The microscope can now be used in conjunction with the Positioning controls in the **Analysis Location** window to move to nearby features. If these are new locations then click the **Add location to list** button  and complete the **Add Analysis Location** dialogue.

Acquiring selected area survey spectra from small features

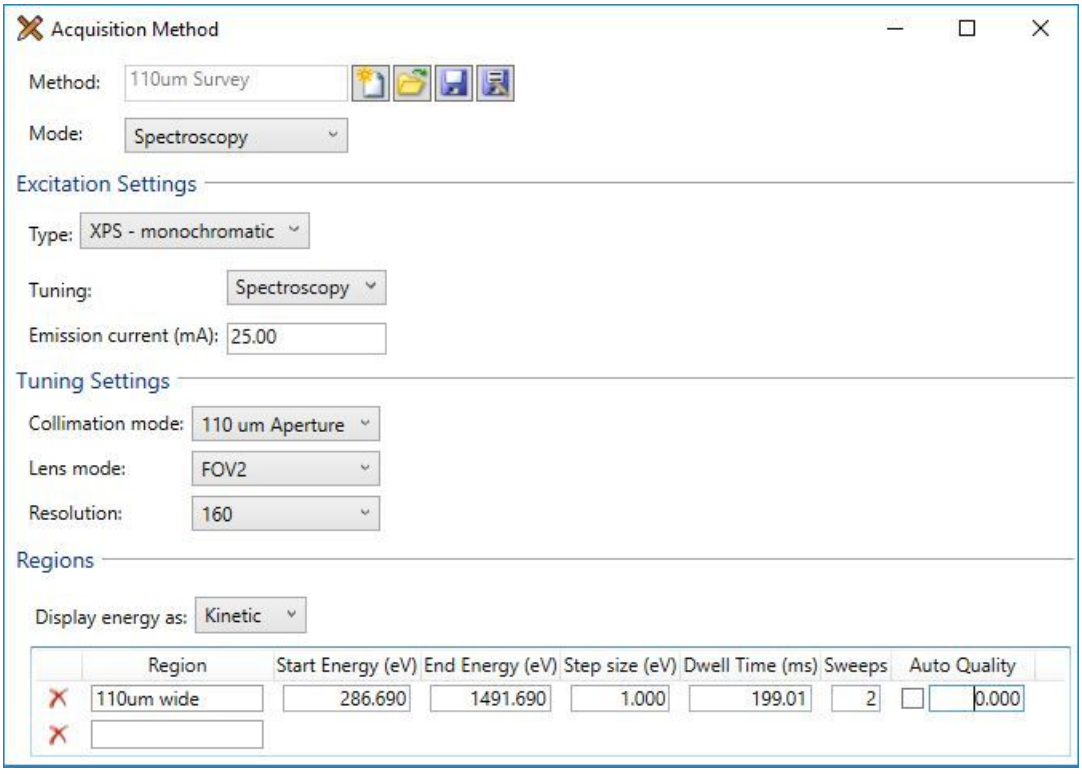
A survey spectrum can be recorded quickly to determine the elements that are present.

1. In the **Analysis** window, set the **Acquisition Mode** to Queue.
2. In the **Acquisition Method** window, click the **Open Method** button  to display the **Open Acquisition Method** window.

For your information

Standard methods are provided in the C:\ESCApe\Methods\Kratos Methods folder. This includes an XPS Selected Area sub folder containing several survey methods for smaller areas.

3. Open a selected area survey method.



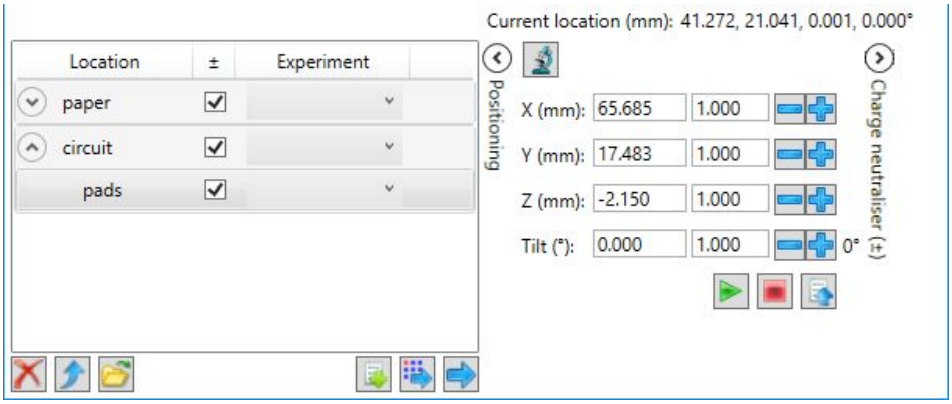
For your information

These settings have been selected as a reasonable starting point.

The **Emission current** can be increased to give a larger count rate. However the resultant higher X-ray power may cause degradation of delicate samples and saturation of the signal observed by the detector. Also if high count rates are acquired for long periods then the lifetime of the detector is shortened.

4. In the **Analysis Location** window select the **Sample holder** with the samples you want to analyse. Then select an **Experiment** file for each location that you want to analyse.
5. In the **Analysis Location** window, either:

   - Click the **Submit location for acquisition** button to submit a location. This button is displayed when you hover your mouse pointer over a location in the locations table.
   - Select a number of locations in the locations table then click the **Submit selected locations for acquisition** button found below the locations table. These locations can be on the same sample or different samples.



   The location(s) are submitted to the queue for analysis. If you have submitted multiple locations then each location has a separate entry in the queue and is analysed separately.
6. If the **Manual** radio button is selected in the **Analysis** window, start the acquisition using one of the following methods:
   - Click the **Start** button. This starts the first acquisition. If you have more than one entry in the queue you have to click the **Start** button for each entry.
   - Select the **Auto** radio button and then click the **Start** button. If this radio button is selected then each entry in the queue starts automatically.
7. Select the **Real Time** tab to see the spectrum.

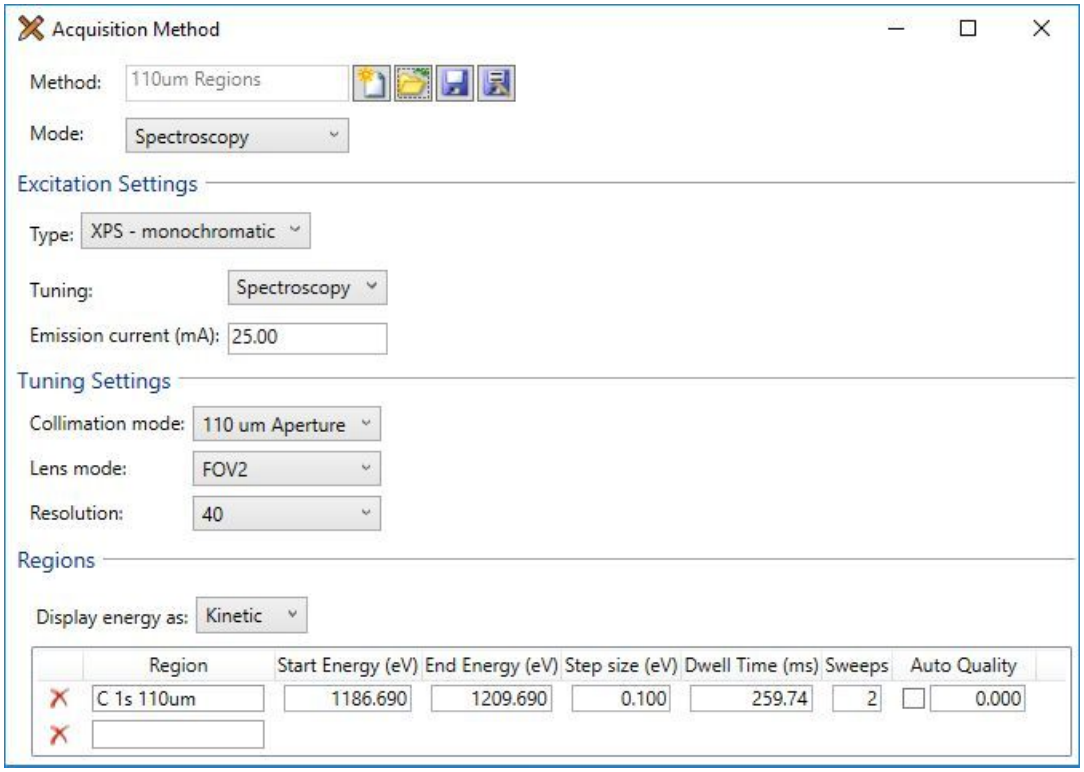
Acquiring selected area high resolution region spectra from small features

1. In the **Analysis** window, set the **Acquisition Mode** to Queue.
2. In the **Acquisition Method** window, click the **Open Method** button  to display the **Open Acquisition Method** window.

For your information

Standard methods are provided in the C:\ESCApe\Methods\Kratos Methods folder. This includes an XPS Selected Area sub folder containing several regions methods for smaller areas.

3. Open a selected area regions method.



For your information

These settings have been selected as a reasonable starting point.

The **Resolution** can be reduced to give a better energy resolution although this gives a lower count rate. This is useful if there are two peaks close together. Conversely the Resolution can be increased to give a poorer energy resolution with a higher count rate. This is useful if there is a trace element.

4. Set up the required regions. You can:
   - delete a row by clicking on the X at the beginning of the row.
   - type in an element line in an empty **Region** box and press Enter on your keyboard. This also gives a new line in the Regions table.
   - edit an entry in a **Region** box and press Enter on your keyboard.

For your information

Right-clicking on either the second or third column heading in the regions table gives a pop-up menu which allows you to change the table between Start Energy/End Energy and Centre Energy/Width.

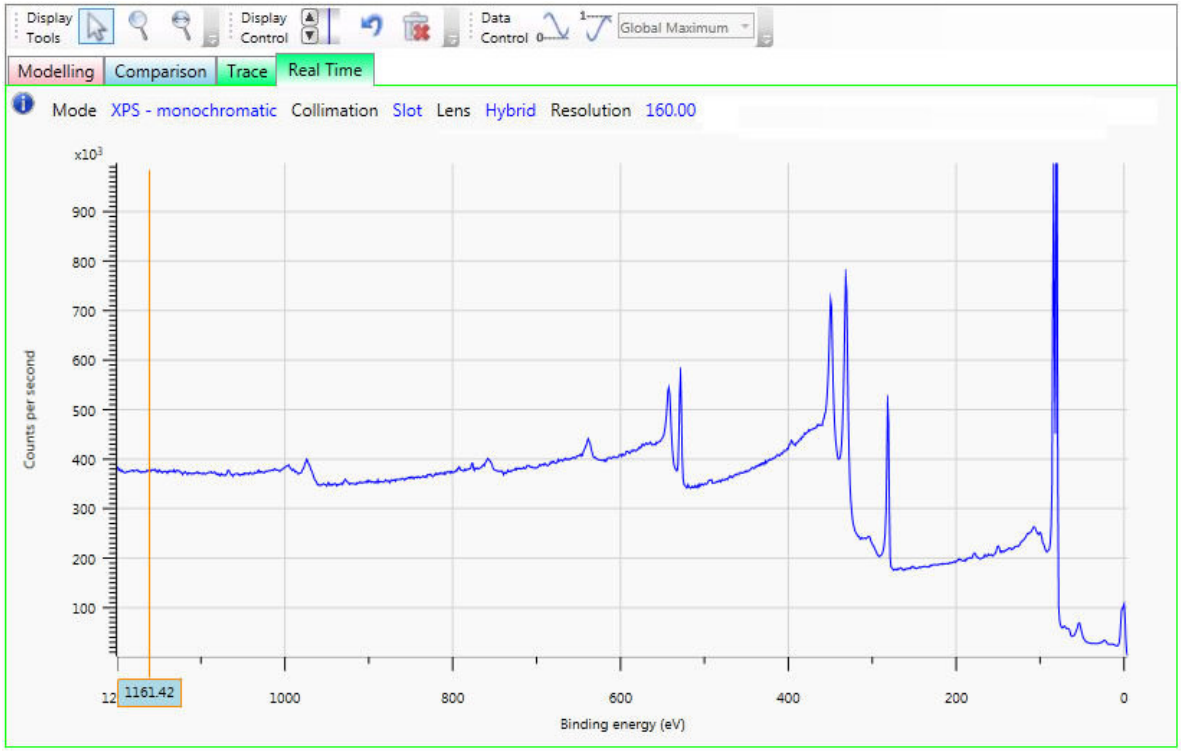
5. In the **Analysis Location** window select the **Sample holder** with the samples you want to analyse. Then select an **Experiment** file for each location that you want to analyse.
6. In the **Analysis Location** window, either:
   - Click the **Submit location for acquisition** button to submit a location. This button is displayed when you hover your mouse pointer over a location in the locations table.
   - Select a number of locations in the locations table then click the **Submit selected locations for acquisition** button found below the locations table. These locations can be on the same sample or different samples.

   The location(s) are submitted to the queue for analysis. If you have submitted multiple locations then each location has a separate entry in the queue and is analysed separately.
7. If the **Manual** radio button is selected in the **Analysis** window, start the acquisition using one of the following methods:
   - Click the **Start** button. This starts the first acquisition. If you have more than one entry in the queue you have to click the **Start** button for each entry.
   - Select the **Auto** radio button and then click the **Start** button. If this radio button is selected then each entry in the queue starts automatically.
8. Select the **Real Time** tab to see the spectrum.

Acquiring parallel XPS images

First determine the energy of the photoelectrons to image. This can be achieved as follows:

1. Either:
   - Obtain a spectrum of the region of interest.
   - Open an existing survey spectrum of the region of interest from the **Data Organiser** window. Drag the spectrum into the **Real Time** tab or double click on the spectrum to open a new tab,



2. If required, select the **Zoom and scroll the display in both axes** button  or the **Zoom and scroll the display horizontally** button  and zoom into the area of interest. These buttons are found at the top of the display area.

For your information

With the  or button  selected, you can hold down the middle mouse button and draw a selection rectangle round the peak of interest. This area is then enlarged.

Click the middle mouse button anywhere in the display area to return to the original magnification.

3. Note the actual energy of the peak. Either:
   - In the display area, select the **Interact with display items** button  . Then hover your mouse pointer over the top of the peak.This displays an information box giving the energy of the peak.
   - Drag the orange energy marker to the top of the peak and read off the energy. See "Energy Marker" on page 98 for more details.

Now acquire the image:

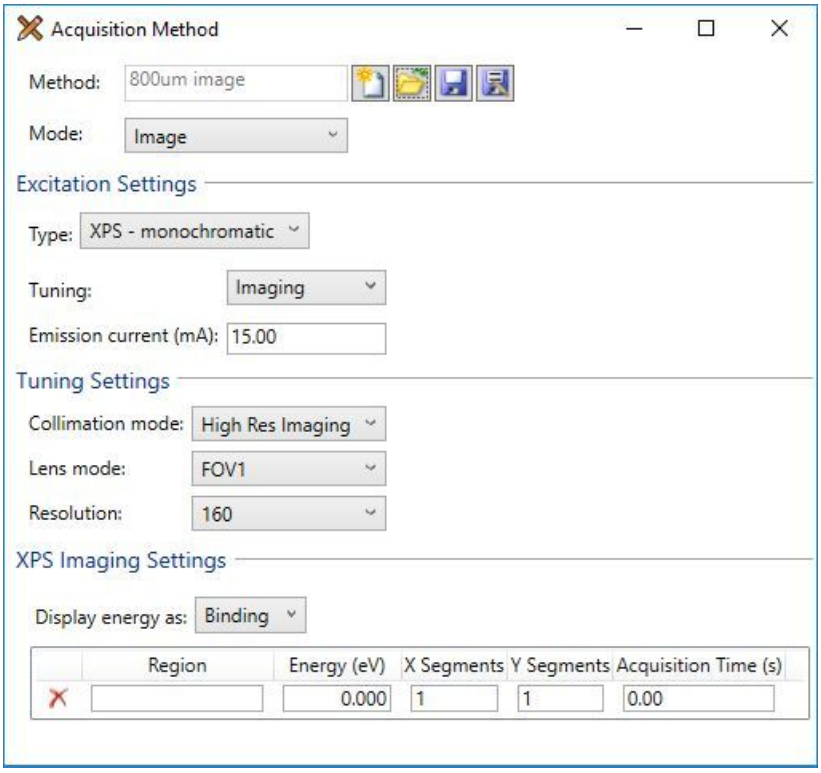
1. In the **Analysis** window, set the **Acquisition Mode** to Queue.
2. In the **Acquisition Method** window, click the **Open Method** button  to display the **Open Acquisition Method** window.

For your information

Standard methods are provided in the C:\ESCApe\Methods\Kratos Methods folder. This includes an XPS Imaging sub folder containing imaging methods.

For example, the 800um Image method acquires an image with a Field of View of 800 μm x 800 μm (FOV1). This is the same as a fully zoomed in microscope image (x12 magnification).

3. Choose an image method.

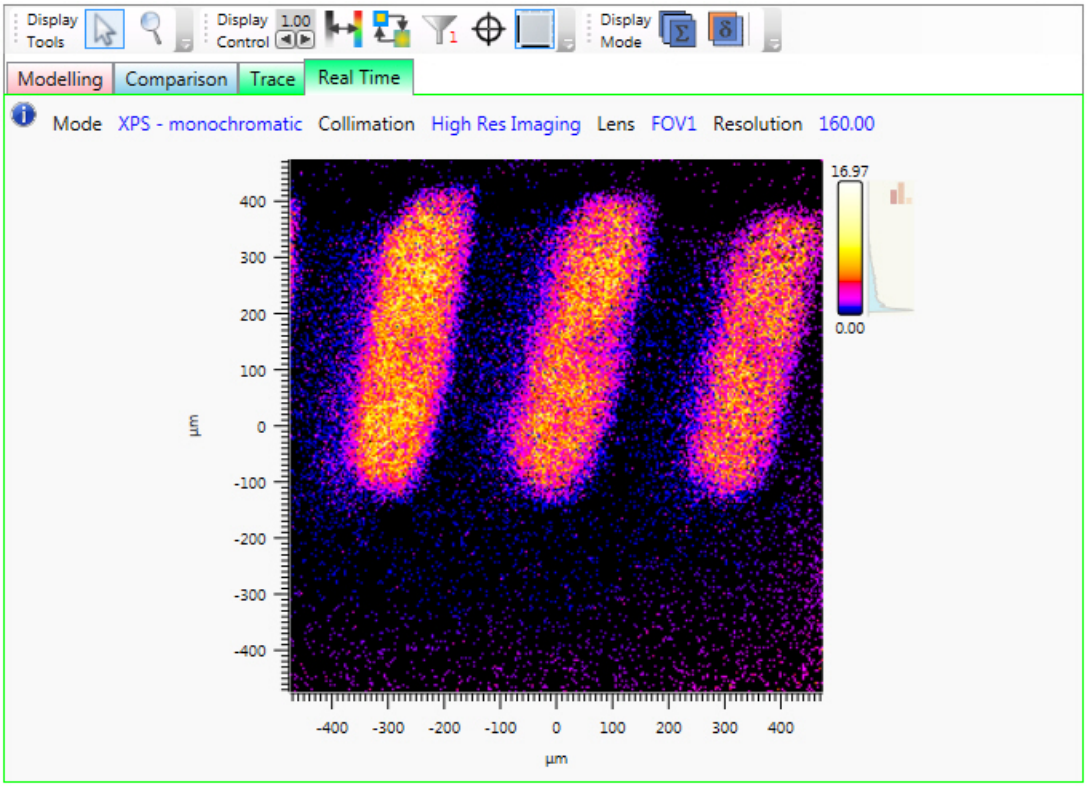


4. Type in the appropriate element line.
5. If necessary, modify the value in the **Energy (eV)** box to correspond to the energy obtained from the spectrum.
6. Change the **Acquisition Time** depending on the intensity.
7. In the **Analysis Location** window select the **Sample holder** with the samples you want to analyse. Then select an **Experiment** file for each location that you want to analyse.
8. In the **Analysis Location** window, either:

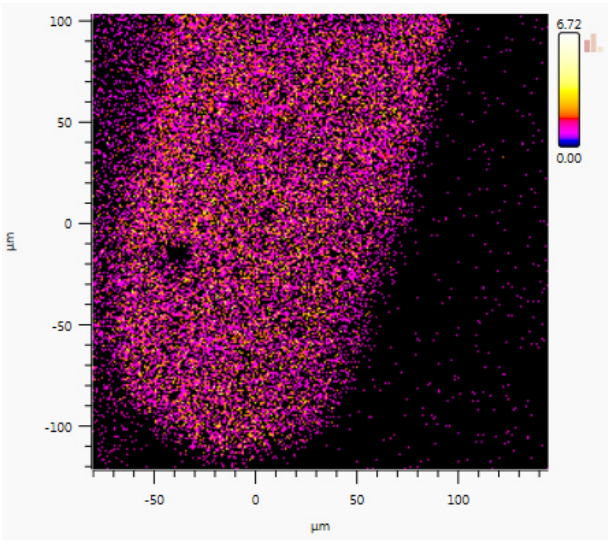
   - Click the **Submit location for acquisition** button to submit a location. This button is displayed when you hover your mouse pointer over a location in the locations table.
   - Select a number of locations in the locations table then click the **Submit selected locations for acquisition** button found below the locations table. These locations can be on the same sample or different samples.

   The location(s) are submitted to the queue for analysis. If you have submitted multiple locations then each location has a separate entry in the queue and is analysed separately.
9. If the **Manual** radio button is selected in the **Analysis** window, start the acquisition using one of the following methods:
   - Click the **Start** button. This starts the first acquisition.
   - Select the **Auto** radio button and then click the **Start** button.
10. Select the **Real Time** tab to see the image.

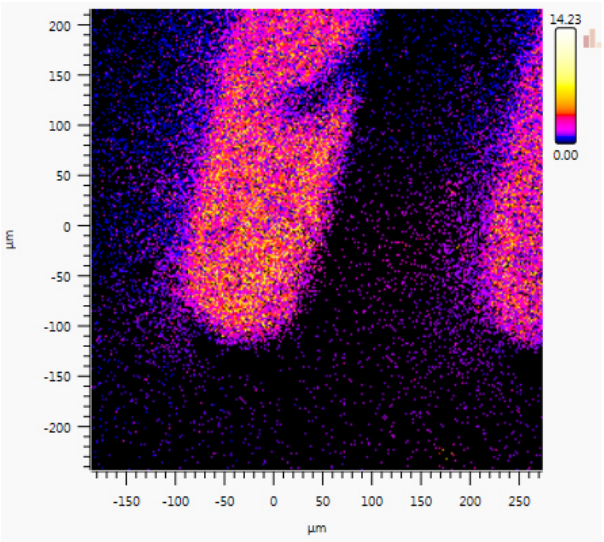
    Note: The image can be switched between a grey scale and a heat map using the button.



For comparison the following images were taken of the same feature using the 200um image and 400um image methods:

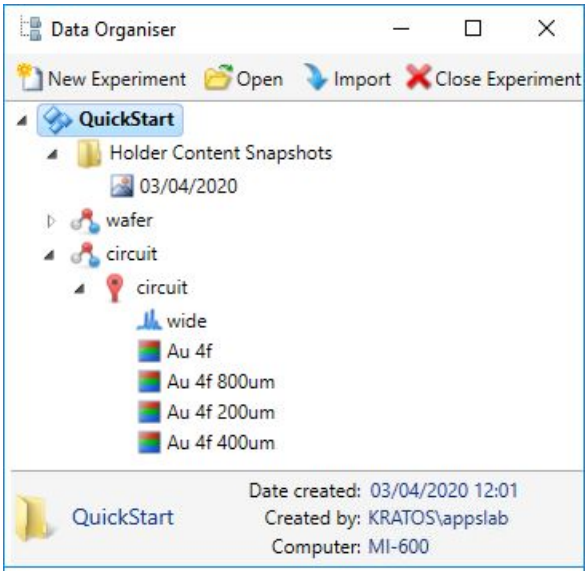


200um image



400um image

As an image is acquired data is added to the selected experiment file using the name of the region. In the example below 4 images have been acquired for the Circuit location on the Circuit sample.



The Au 4f and the Au 4f 800um images were obtained using the same imaging method. In the case of Au 4f 800um the name of the region was changed before the acquisition was submitted.

Acquiring selected area spectra from a parallel image

The parallel image must have been acquired using the 400um image method from the Kratos methods folder.

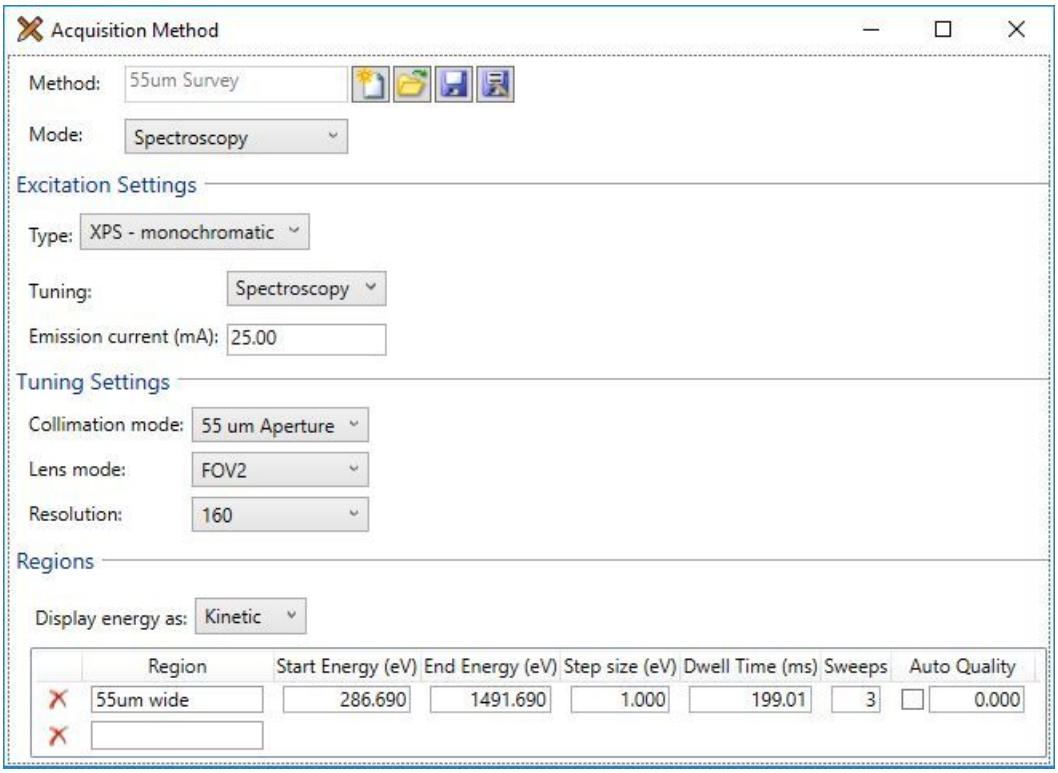
1. Open the parallel image by double clicking on the image in the **Data Organiser** window.

   This is opened in the display area.
2. In the **Acquisition Method** window, click the **Open Method** button  to display the **Open Acquisition Method** window.

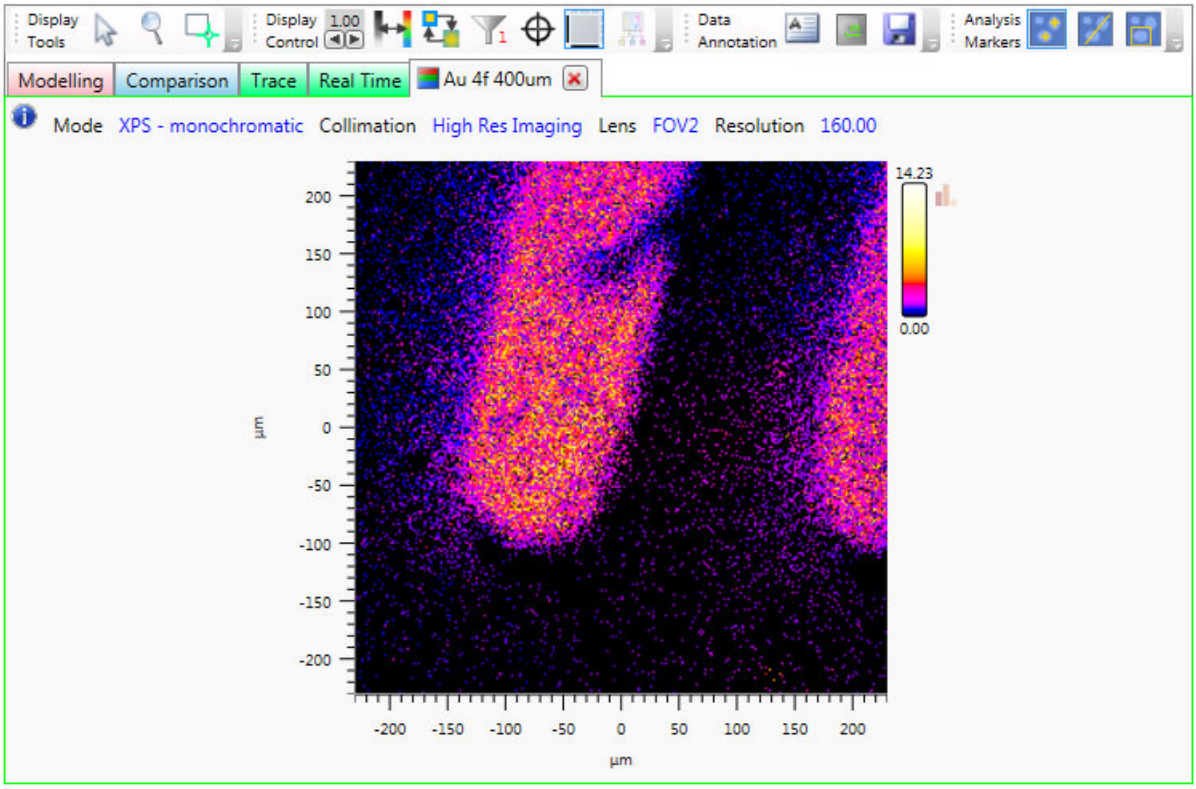
For your information

Standard methods are provided in the C:\ESCApe\Methods\Kratos Methods folder. This includes an XPS Selected Area sub folder for running smaller area scans.

3. Choose a Selected Area method. For example, 55um Survey.

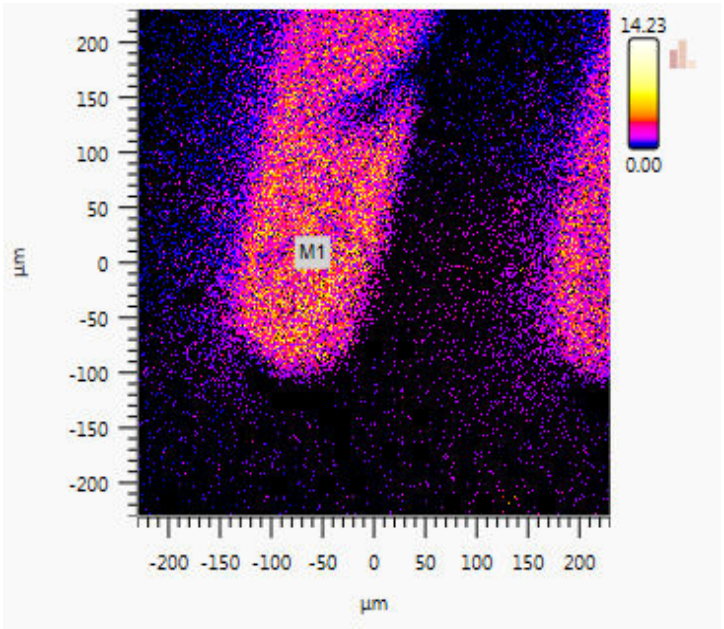

Acquisition Method
Method:
55um Survey
Mode:
Spectroscopy
Excitation Settings
Type:
XPS - monochromatic
Tuning:
Spectroscopy
Emission current (mA):
25.00
Tuning Settings
Collimation mode:
55 um Aperture
Lens mode:
FOV2
Resolution:
160
Regions
Display energy as:
Kinetic
Region
Start Energy (eV)
End Energy (eV)
Step size (eV)
Dwell Time (ms)
Sweeps
Auto Quality
55um wide
286.690
1491.690
1.000
199.01
3
0.000

4. In the display area, select the **Point of interest** Analysis Marker  .

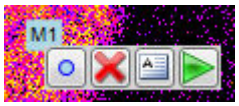


5. Click on the image where you want the selected area spectroscopy acquisition to be centred.

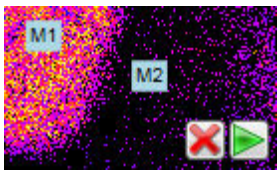
   A rectangle is displayed with a Marker ID.



6. Repeat Step 5 if you want to acquire a spectrum from more than one location.
7. If you have only selected one location:
   a. Select the **Interact with display items** Mouse Mode .
   b. Click on the Marker ID to display some extra buttons.



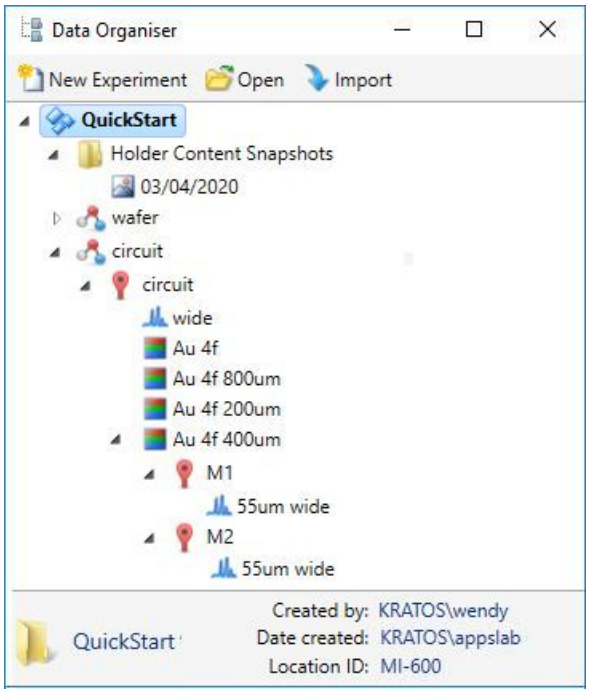
   c. If you want to change the name of the marker to a more meaningful name, click the **Edit marker name** button and edit the name. This name is used in the experiment file.
   d. Click the **Submit analysis for acquisition** button .
   This submits the location to the queue for analysis.
8. If you have selected multiple locations:
   a. If you want to change the names of the markers, follow Step 7 a to c.
   b. Select the **Select multiple items** Mouse Mode .
   c. Select the markers by creating a selection rectangle round the markers. This displays two extra buttons.



   d. Click the **Submit analyses to the queue** button .
   This submits the locations to the queue for analysis. Each location has a separate entry in the queue and is analysed separately.
9. If the **Manual** radio button is selected in the **Analysis** window, start the acquisition using one of the following methods:
   - Click the **Start** button. This starts the first acquisition. If you have more than one entry in the queue you have to click the **Start** button for each entry.
   - Select the **Auto** radio button and then click the **Start** button. If this radio button is selected then each entry in the queue starts automatically.

10. Select the **Real Time** tab to see the data.

    As spectra are acquired data is added to the experiment file as a child of the parallel image. The data is added using the names of the regions. For example, if selected area spectra are acquired for M1 and M2 on the Au 4f 400um image the experiment file has data added as follows:



Setting Acquisition Preferences

Automatically

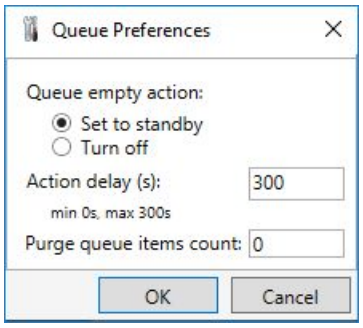
Depending on the way you work you may want to turn off the instrument automatically when the queue is empty. This action is controlled by the **Queue Preferences** dialogue.

Caution

Selecting **Turn off** does not turn off all voltages. To electrically isolate the instrument the 50 A circuit breaker on the instrument's mains distribution unit or the 'wall circuit breaker' must be turned OFF.

Individual units can be made electrically safe by switching the mains switch off and removing the IEC mains cable.

1. Right-click on the Status icon  in the bottom left hand corner of the main window and select **Instrument > Acquisition preferences**. The **Queue Preferences** dialogue is displayed.



2. Select the action required when the queue is empty. This can be:
   - **Set to standby** - the ion guns and X-ray guns are placed in standby, the detector and charge neutraliser are turned off and the analyser energy is set to 100 V.
   - **Turn off** - the analyser energy is set to 0 V and the following items are turned off:
     - X-ray guns
     - ion guns
     - ion gun gases
     - detector
     - charge neutraliser.

   Note: Not all accessories are turned off.
   Some accessories that are turned off may take a considerable

period to stabilize when they are switched back on. For example, the Field Emission electron Gun (FEG) can take up to 12 hours to fully stabilize.

3. Select the **Action delay** in seconds (maximum 300 s). This is the time waited after completion of the last acquisition in the queue before the instrument is set to standby or turned off.
4. If required, amend the **Purge count**.

For your information

The **Purge count** parameter controls the number of completed or skipped acquisitions that are left visible in the queue in the **Analysis** window. Failed acquisitions must be removed manually.

5. Click **OK** to set up the required action.

Manually

You can place the instrument in a non operating state to extend the life of the instrument or as the first step in switching the instrument off for maintenance.

Caution

Selecting **Turn off** does not turn off all voltages. To electrically isolate the instrument the 50 A circuit breaker on the instrument's mains distribution unit or the 'wall circuit breaker' must be turned OFF.

Individual units can be made electrically safe by switching the mains switch off and removing the IEC mains cable.

1. Right click on the Status icon  in the bottom left hand corner of the main ESCApe window.
2. Select **Instrument > Turn off**.

The analyser energy is set to 0 V and the following items are turned off:

- X-ray guns
- ion guns
- ion gun gases
- detector
- charge neutraliser.

Note: Not all accessories are turned off.
Some accessories that are turned off may take up to 12 hours to stabilize when they are switched back on.

Chapter 5
Processing

Introduction

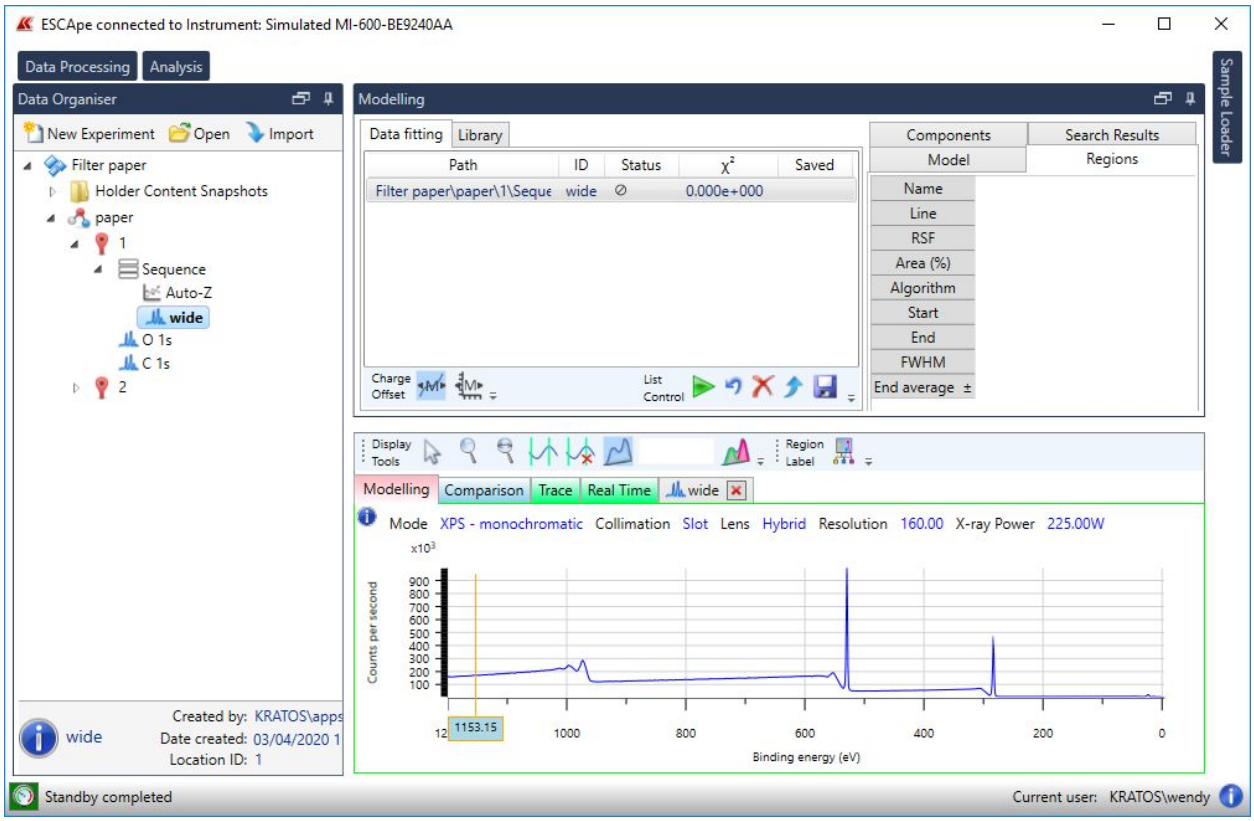
The steps required to quantify survey and high resolution (narrow region) spectra are illustrated using the data acquired in the filter paper example used in Chapter 4.

Quantifying survey spectra

1. Pin or float the **Modelling** window.
2. Open the **filter paper.experiment** file in the **Data Organiser** window.
3. Expand the experiment file and drag a spectrum called **wide** into the **Data fitting** section of the **Modelling** window.
4. Select the item in the **Data fitting** list (note the full data path is shown to clearly identify the spectrum). This displays the spectrum in the **Modelling** tab in the display area.

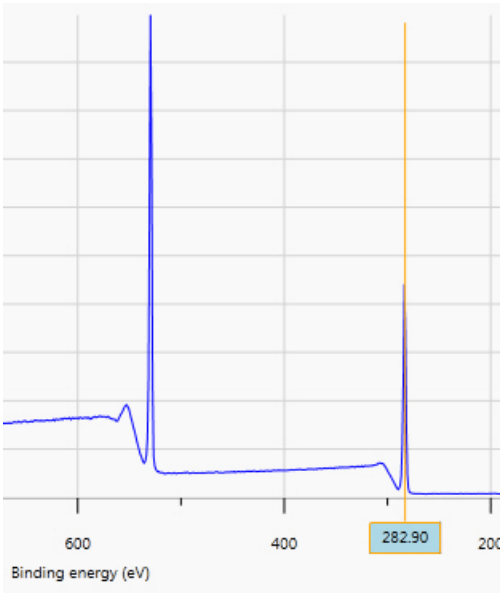
   The survey scan contains photoemission peaks for oxygen and carbon and possibly some other minor elements. Note the **Add Element Lines** tool can be used to help with peak identification – see "Element Lines tools" on page 102.

   In this instance we will define regions for O 1s and C 1s.

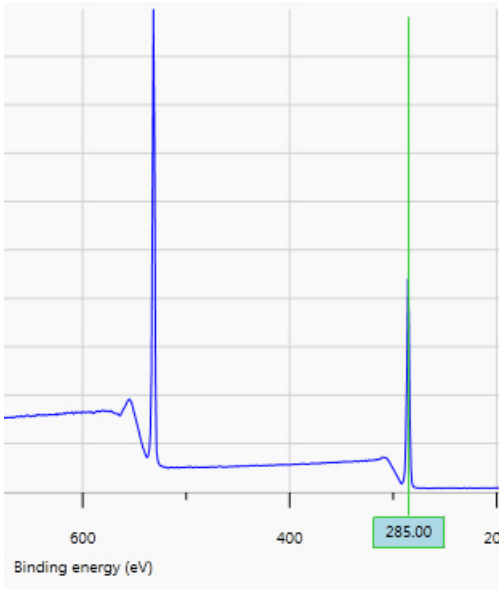


5. If your spectrum is charge shifted, as in the case of the filter paper, apply a charge correction.
   a. If required, zoom into the C 1s peak (~288 eV BE) to make it easier to see the start and end regions.
   b. Drag the value box to move the charge correction marker line to the C 1s peak.
   c. When in position double click in the box to highlight the energy.
   d. Type in the correct energy, in this case 285, then press the return key.

      The spectrum shifts to the corrected energy and the marker line turns green to show that the correction has been applied. The **Status** of the spectrum in the **Modelling** window shows a  icon.



Before charge correction



After charge correction

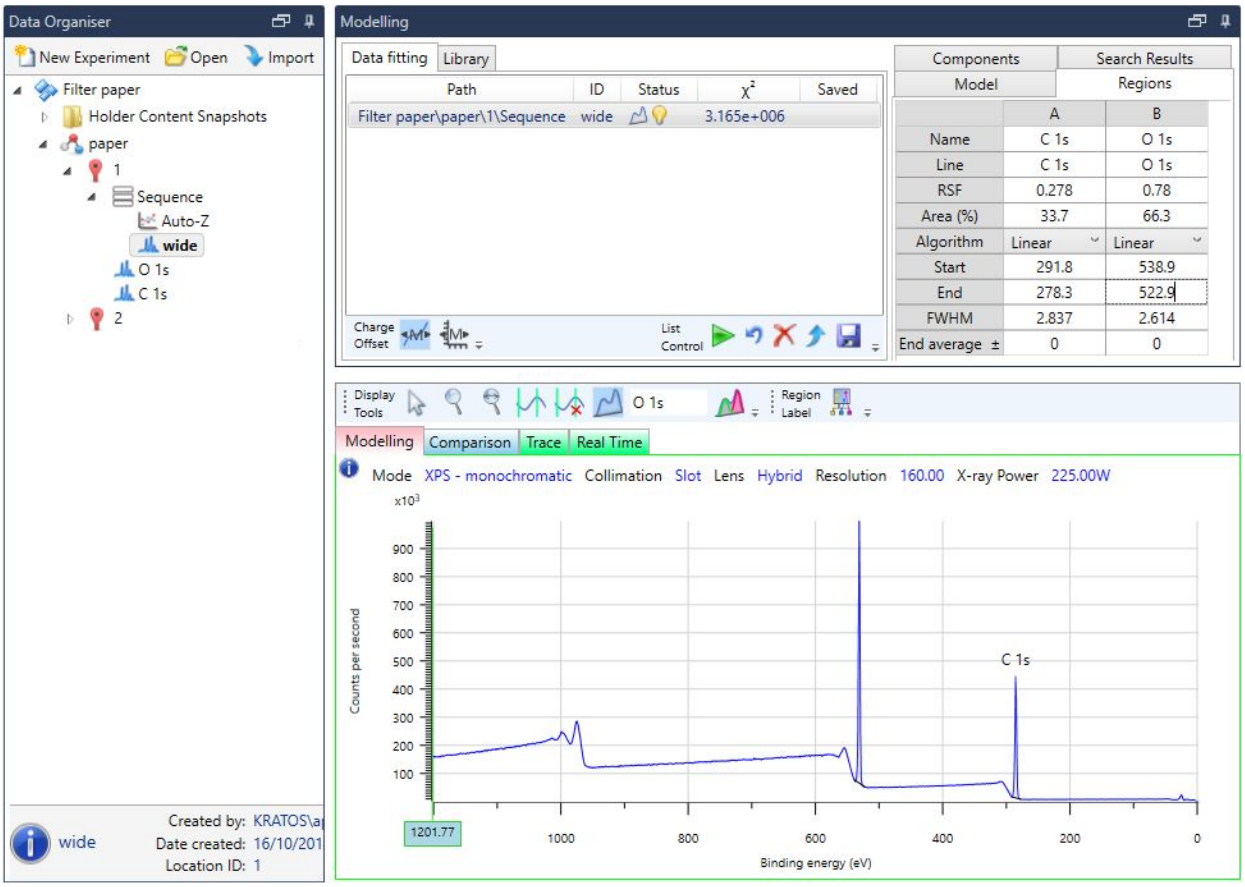
6. Define a region for C 1s as follows:
   a. Ensure the **Edit background regions**  button is selected. This displays a text entry box.
   b. Add a linear background to the spectrum and create a column in the regions table in the **Modelling** window as follows. Either:
      - Enter the element line in the text box.
      - Use the mouse to drag a line across the C 1s peak. This line defines the start and end values for the region. Then enter C 1s in the **Line** box or see "Select from library tool" on page 99 to use the **Select library line** window.

For your information

If appropriate, the algorithm used to calculate the background can be changed using the **Algorithm** drop-down menu in the regions table.

As soon as one region is defined the **Status** of the spectrum in the **Modelling** window shows a  icon.

7. Define regions for O 1s (~530 eV BE) as described in Step 6. This time enter O 1s in the **Line** box.



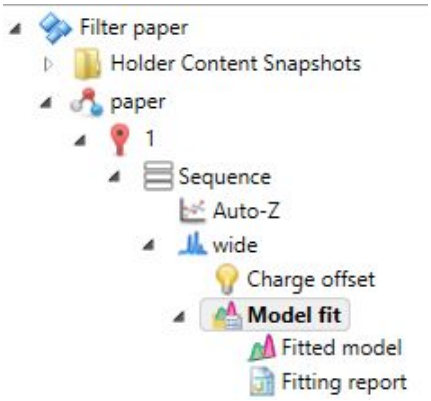
For your information

You can amend the background by entering new values in the **Start** and/or **End** boxes in the regions table. Alternatively select the **Interact with display items** button and click on the appropriate region to display a number of options . Select the blue circle to display a pair of blue circles - one at each end of the background. Drag a blue circle to adjust its position.

To delete a region either click on the region using the **Interact with display items** tool and click on the red cross or click on the column header in the regions table and select the red cross which appears there.

8. Click the **Save selected models as experiment data** button in the **Modelling** window to save the charge offset and data model and to generate a quantification report.

   A charge offset and a folder containing the fitted model used to fit the data (in this case two regions) and a fitting report containing the quantification results are saved as children of the spectrum.

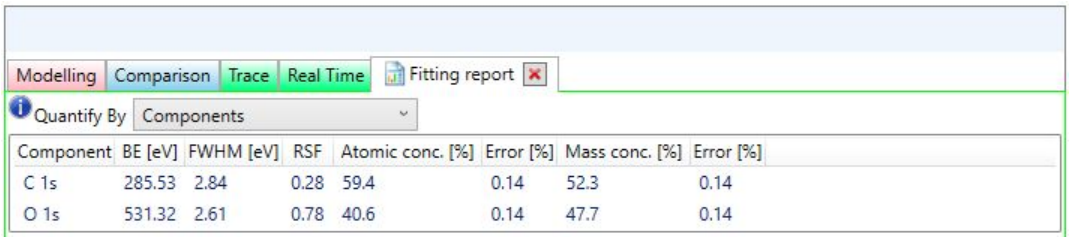


   An icon is added to the **Status** of the spectrum in the **Modelling** window. (Note that this icon is removed if any further changes are made to the model.)

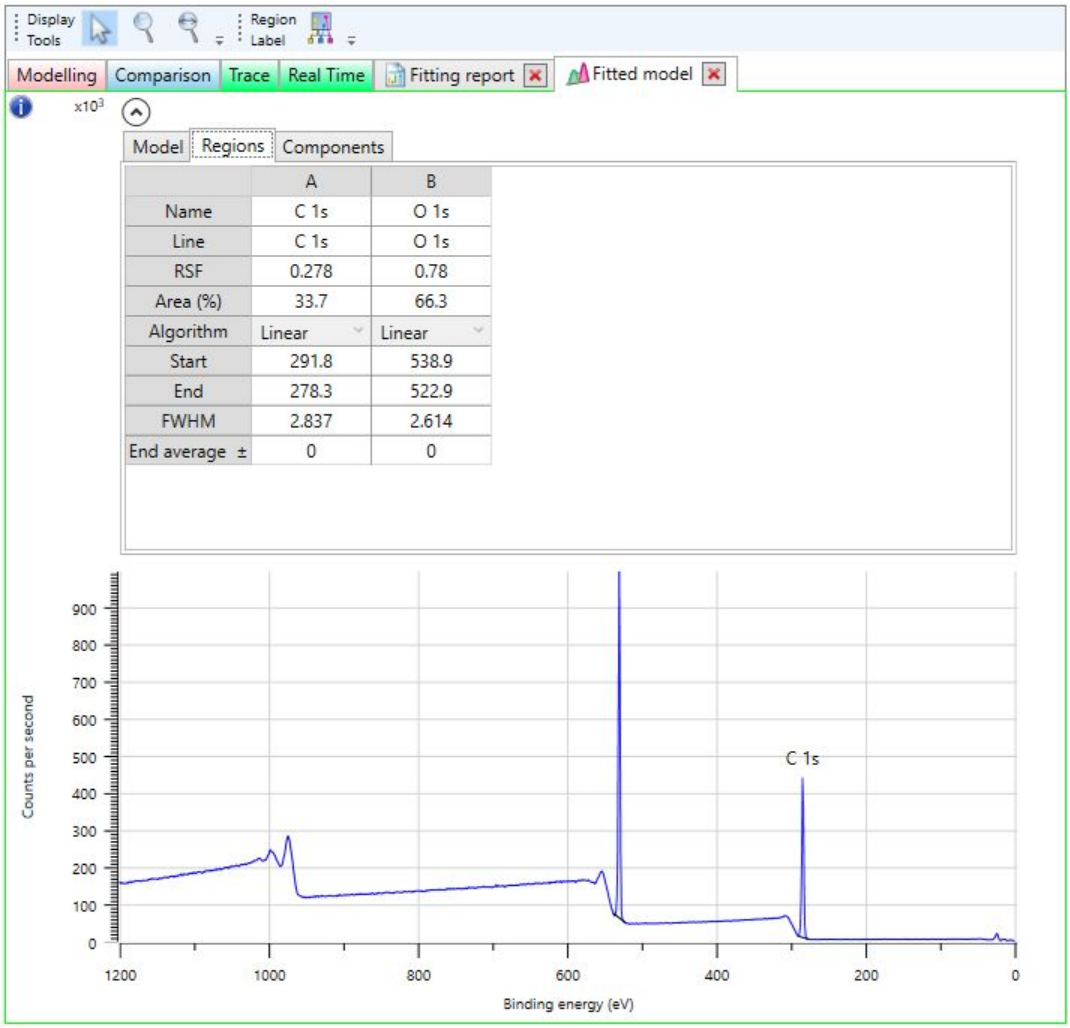
9. Double click on the Fitted model or Fitting report to view them.

   The Fitting report can be viewed as either a function of regions or components (names) if present.

   Note: The Fitted model and Fitting report cannot be edited.



Component	BE [eV]	FWHM [eV]	RSF	Atomic conc. [%]	Error [%]	Mass conc. [%]	Error [%]
C 1s	285.53	2.84	0.28	59.4	0.14	52.3	0.14
O 1s	531.32	2.61	0.78	40.6	0.14	47.7	0.14



	A	B
Name	C 1s	O 1s
Line	C 1s	O 1s
RSF	0.278	0.78
Area (%)	33.7	66.3
Algorithm	Linear	Linear
Start	291.8	538.9
End	278.3	522.9
FWHM	2.837	2.614
End average ±	0	0

Curve Fitting for Quantification

Curve fitting is the term generally used for the process of creating a model of component peaks which describes chemical states present within the data.

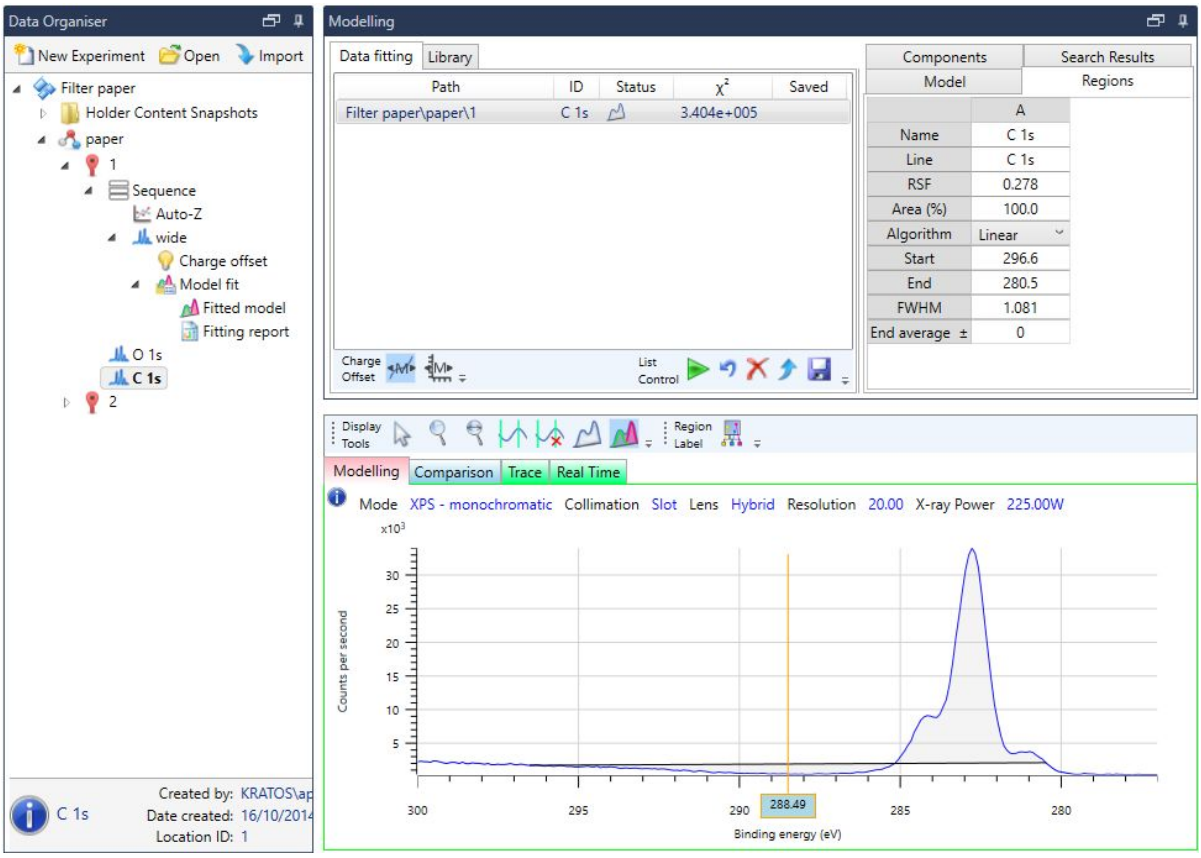
A C 1s spectrum is used to explain the process of curve fitting and the tools available to facilitate this process.

1. Pin or float the **Modelling** window.
2. Open the **Filter paper.experiment** file in the Data Organiser window.
3. Expand the experiment file and drag the **C 1s** spectrum into the **Data fitting** section of the **Modelling** window. The full data path is shown to clearly identify the spectrum.

   The spectrum is displayed in the **Modelling** tab in the display area.

For your information

If the spectrum has been recorded using a region name that starts with an element name and transition, as in the case of C 1s, then a region is automatically shown when the spectrum is dropped into the **Modelling** window. This region uses data from the element library and may require modification. See the **For your information** box on page 83.



Creating a region

Components can only be defined once a background region has been defined. If a region has not been created automatically when the spectrum is dropped into the **Modelling** window, create a region as follows:

1. Ensure the **Edit background regions**  button is selected in the display area. This displays a text entry box.
2. Add a linear background to the spectrum and create a column in the regions table in the **Modelling** window. Either:
   - Enter the element line in the text box.
   - Use the mouse to drag a line across the peak. This line defines the start and end values for the region. Then enter the element name and transition in to the **Line** box in this column.

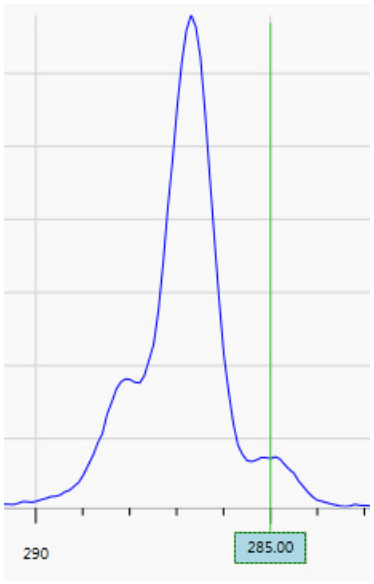
   A  icon is added to the **Status** of the spectrum in the **Modelling** window.

Applying a charge correction

If your spectrum is charge shifted, as in the case of the filter paper, apply a charge correction.

1. Ensure that the **Model energies are adjusted with the charge offset of the spectrum** button  is selected in the **Modelling** window. This ensures that the region stays with the data when the charge correction is applied.
2. Drag the value box to move the charge correction marker line to the C-C peak.
3. When in position double click in the box to highlight the energy.
4. Type in the correct energy, in this case 285, then press the return key.
5. The spectrum shifts to the corrected energy and the marker line turns green to show that the correction has been applied. The **Status** of the spectrum in the **Modelling** window shows a  icon.

For more information on charge correction, see "Charge correction tool" on page 100.


290
285.00

Creating components

Components can be created using the **Edit components** button  and either:

- Clicking within the area defined by the background region. A component is automatically added at the highest intensity part of the spectrum with a peak which best fits the data using the default peak shape. Clicking again adds another peak at the highest intensity not included in the previous component(s).
- Drawing a line at the height and position of the required peak. The line length determines the full width half maximum (FWHM) of the peak.

When you have defined more than one component in a region a black dashed line is added indicating the synthetic spectrum formed by the sum of the components. In a well fitted model this synthetic spectrum should be close to the actual spectrum.

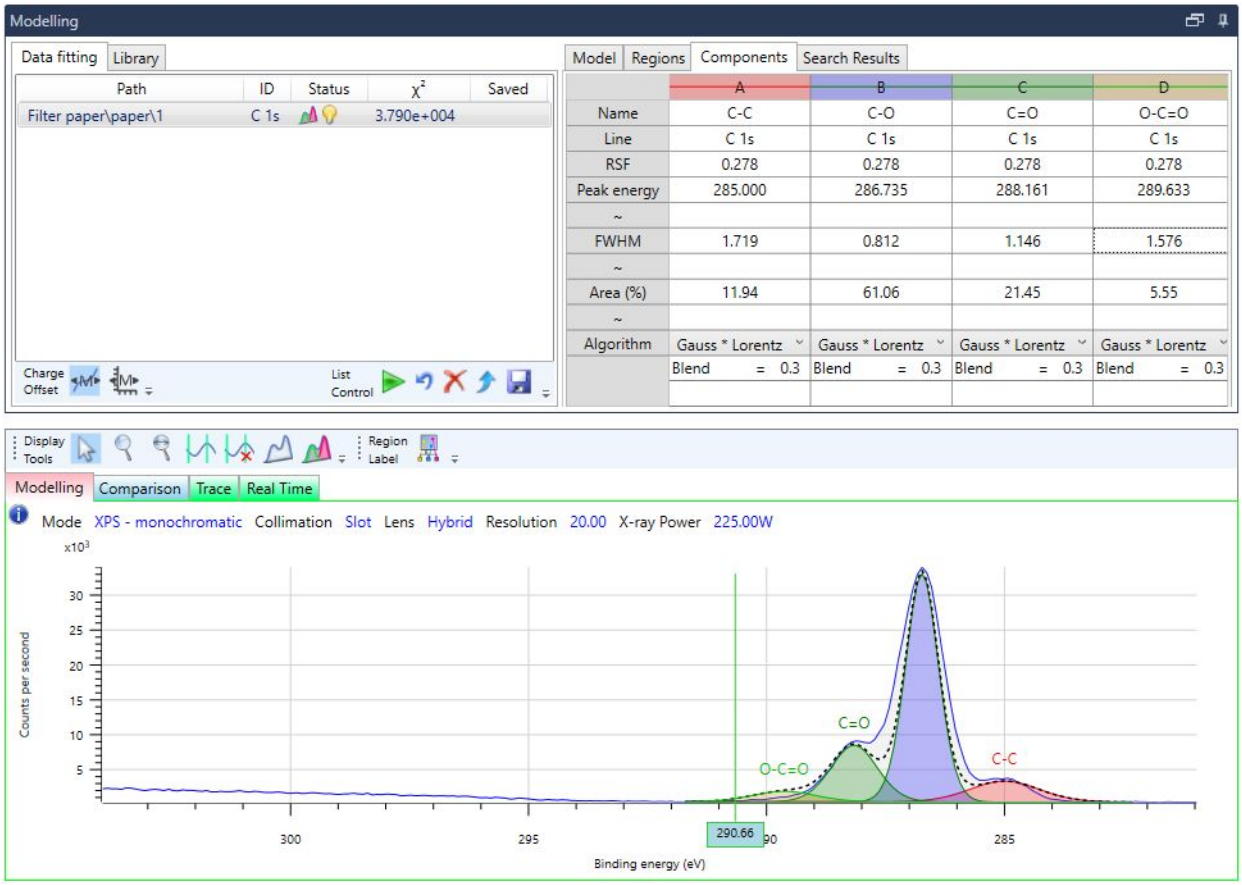
With the **C 1s** spectrum:

1. Select the **Edit components** button  in the display area. The **Components** tab is automatically selected in the **Modelling** window.
2. Create four components by drawing lines at the height and position of the four required peaks. Start with the lowest binding energy peak.

   This creates 4 columns in the components table.
3. In the **Name** boxes at the top of columns enter the following:

Column	Name
A	C-C
B	C-O
C	C=O
D	O-C=O

   This allows the relative concentrations of the different chemical states to be determined.
4. If required, right click on the component identifier at the top of the column to display a colour wheel then select a new colour and the line type and/or click fill to give each component a different colour.



For your information

You can modify components by editing the appropriate boxes in the components table. Alternatively select the **Interact with display items** button and click on the appropriate component to display a number of options . Select the blue circle to display 3 blue circles - one at the top which adjusts the peak height and position and two at either side which adjust the peak width. Drag a blue circle to adjust its position.

To delete a component either click on the component using the **Interact with display items** tool and click on the red cross or click on the top box of the column in the regions table and select the red cross which appears there.

Adding fitting constraints

It is possible to link some of the component attributes together to take into account the various physical phenomena such as spin orbit splitting and chemical shifts. For example, in Aluminium the 2p peaks are a doublet, the separation and relative areas of which are known. When the Auto fit routine is run or the components are manually adjusted these constraints restrict the movement of the components.

Below each component parameter (Peak energy, FWHM and Area) is an extra box labelled ~. These are used to enter component constraints.

Applying an absolute value constraint

Click in the constraint box and press the space bar. The current value is shown twice in the box separated by three dots. For example, 75.3…75.3.

These 2 numbers represent the maximum and minimum values of the range within which the component can be adjusted. To fix a value the two numbers must be the same.

For example to allow the position of a peak to vary between 75 and 77 eV enter 75.0…77.0.

Constraining a component relative to another

Relative constraints can be used for:

- the separation of the peaks, for example in a doublet
- the relationship of the peak widths
- the relative area ratio, for example in a doublet

To constrain the components:

1. In the column of the component you want to constrain, click in the relevant constraint box.

   This component can be thought of as the slave and the component that you are constraining it relative to as the master.

   In the slave component constraint box, type in the column letter of the master component and press the space bar. The current relationship between the two components is displayed and the master parameter is shaded blue in the **Components** table.

For your information

Once a constraint box is shaded blue indicating that it is a master parameter it cannot be a slave of another component.

In the C 1s example, typing B in the **FWHM** constraint box in column A then pressing the space bar displays 2.12*B.

For Peak energy the relationship is expressed as an offset (for example, B+0.398). For FWHM and Area the relationship is expressed as a multiple (for example 1.0*B).

2. Modify the constraint to the required value and press Enter.

   Note that the type of constraint cannot be changed. If the constraint is shown as an offset it must remain an offset or if it is shown as a multiple it must remain a multiple.

   In the C 1s example, modify the FWHM constraint from 2.12*B to 1.0*B.

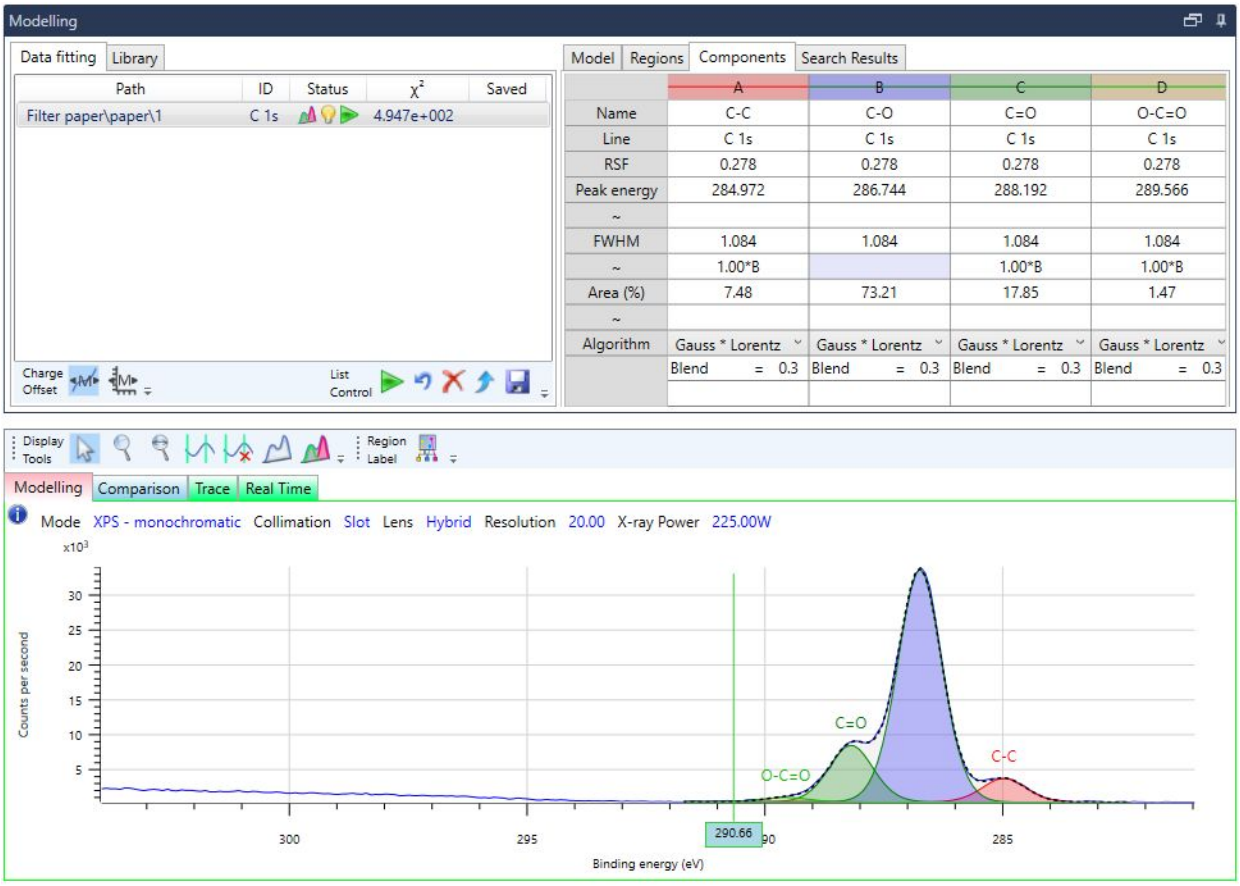
3. Repeat Step 1 to Step 2 to apply additional constraints.

   In the C 1s example, also constrain the FWHM of component C and component D to be 1.0*B.

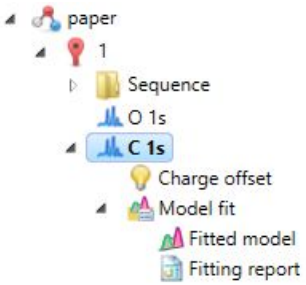
4. After adding peak constraints, apply a peak fitting algorithm to the data by clicking the **Auto fit components** button  in the display area.

   Master parameters are optimised using downhill simplex method [1] to minimise χ^2 . During each iteration the values of slave parameters are calculated from the master parameters using any relative constraints with the value of each parameter being kept within any absolute value constraints.

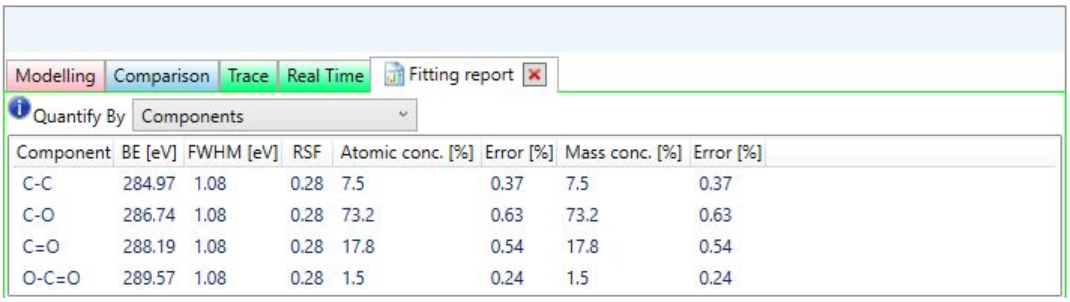
1. Numerical Recipes in C++, William H. Press et al., Second Edition, ISBN 05-521-75033-4, Section 10.4, pp 413-417.



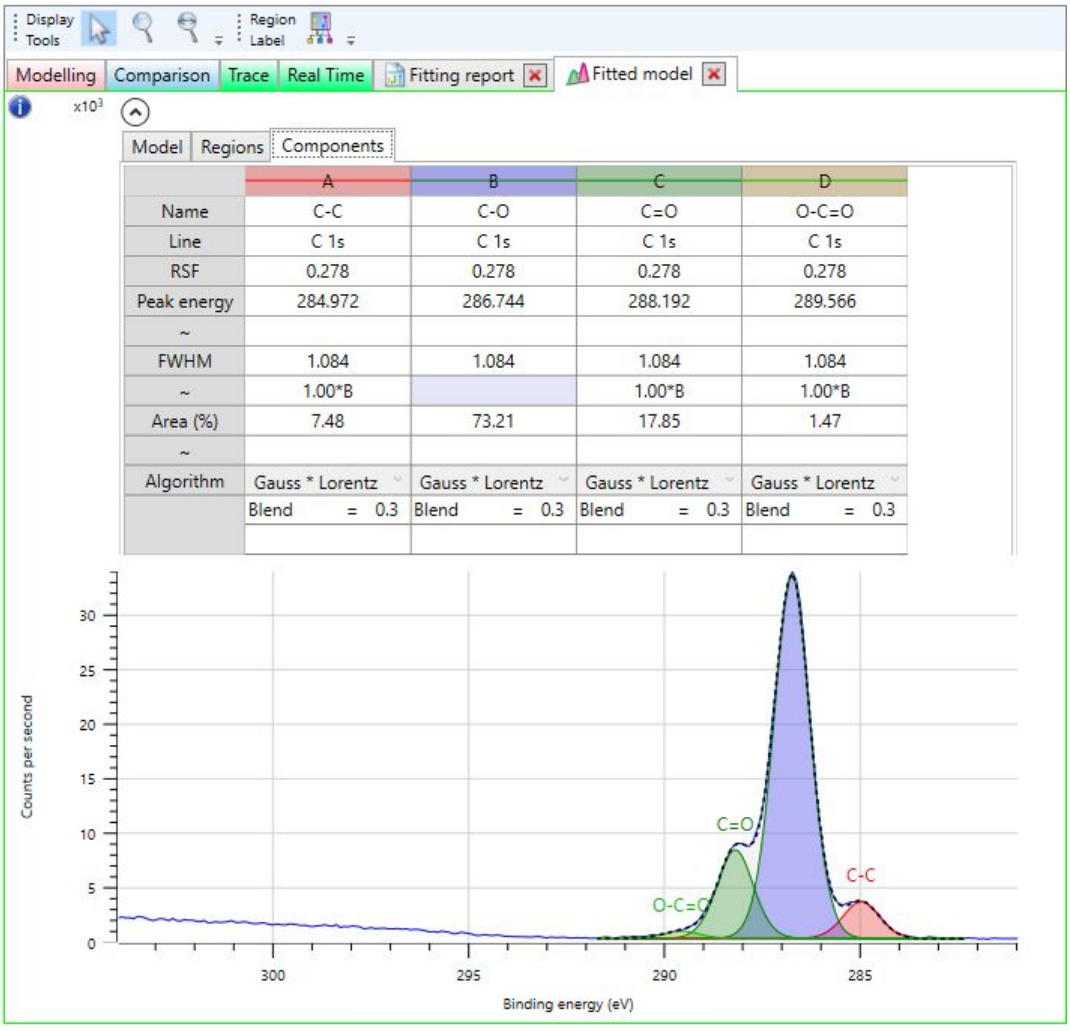
5. When you are happy with the data, save it to the experiment by clicking the **Save selected models as experiment data** button  . A folder containing the fitted model used to fit the data and a fitting report containing the quantification results is saved as a child of the spectrum.



6. Double click on the Fitted model or Fitting report to view them.
   The quantification report can be viewed as either a function of regions or components (names) if present.



Component	BE [eV]	FWHM [eV]	RSF	Atomic conc. [%]	Error [%]	Mass conc. [%]	Error [%]
C-C	284.97	1.08	0.28	7.5	0.37	7.5	0.37
C-O	286.74	1.08	0.28	73.2	0.63	73.2	0.63
C=O	288.19	1.08	0.28	17.8	0.54	17.8	0.54
O-C=O	289.57	1.08	0.28	1.5	0.24	1.5	0.24



	A	B	C	D
Name	C-C	C-O	C=O	O-C=O
Line	C 1s	C 1s	C 1s	C 1s
RSF	0.278	0.278	0.278	0.278
Peak energy	284.972	286.744	288.192	289.566
~				
FWHM	1.084	1.084	1.084	1.084
~	1.00*B		1.00*B	1.00*B
Area (%)	7.48	73.21	17.85	1.47
~				
Algorithm	Gauss * Lorentz	Gauss * Lorentz	Gauss * Lorentz	Gauss * Lorentz
	Blend = 0.3	Blend = 0.3	Blend = 0.3	Blend = 0.3

Applying a Charge Correction to Other Spectra

The charge offset data can be applied to other spectra. For example, other elemental regions acquired at the same time.

In the filter paper example, the charge correction applied to the C 1s spectrum at location 1 can be applied to the O 1s spectrum acquired at the same location.

1. Pin or float the **Modelling** window.
2. Open the relevant experiment file in the **Data Organiser** window.
3. Drag and drop the spectra to which you want to apply the charge correction into the **Data fitting** section of the **Modelling** window.

For your information

You can drag and drop an experiment, sample or location into the **Data fitting** section. This drags all child spectra into the table.

4. Select the chosen spectra from the table in the **Data fitting** section. You can use the Ctrl and Shift keys to select multiple files or a range of files. Ctrl + A selects all items in the table.

For your information

There are two **Charge Offset** buttons  and  . Use these as follows:

- If you have created models (that is, regions and/or components) for the chosen spectra select the **Model energies are adjusted with the charge offset of the spectrum** button  . This ensures that a model stays with the data when the charge correction is applied.
- If you have applied models to the chosen spectra from other data (see "Applying a Model to Other Spectra" on page 95) select the **Model energies are independent of the charge offset of the spectrum** button  . This ensures that a model does not move when the charge correction is applied to a spectrum.
- If you have not yet created or applied models then either button can be selected.

5. Drag and drop the Charge offset data from the **Data Organiser** window into the **Data fitting** section of the **Modelling** window. This applies the charge offset to all selected spectra.

Applying a Model to Other Spectra

Models that have been applied to one spectrum can be propagated to other spectra in the same or different experiment files.

To apply a fitted model to other spectra:

1. Pin or float the **Modelling** window.
2. Open the relevant experiment files in the **Data Organiser** window.

   Drag and drop the spectra to which you want to apply the model into the **Data fitting** section of the **Modelling** window.

For your information

You can drag and drop an experiment, sample or location into the **Data fitting** section. This drags all child spectra into the table.

3. Select the chosen spectra from the table in the **Data fitting** section. You can use the Ctrl and Shift keys to select multiple files or a range of files. Ctrl + A selects all items in the table.
4. Drag and drop the Fitted model from the **Data Organiser** window into the **Data fitting** section of the **Modelling** window. This superimposes the model on all selected spectra adjusting for intensity.
5. Click on an item in the table to view the model in the **Modelling** tab in the display area. You can then edit the model or auto fit as required.
6. Save the models to the experiment file:
   a. Select the spectra in the table.
   b. Click the **Save selected models as experiment data** button  in the **Modelling** window.

Quantification from Multiple Spectra

To quantify from several spectra, for example narrow regions from the same location, each spectrum must first have a model applied and saved. In its most simple form this model could consist of just a region but can also consist of components.

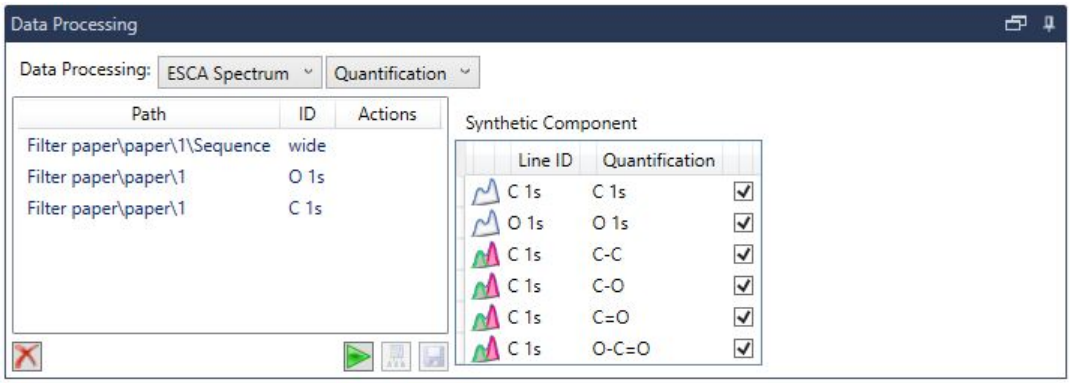
1. Pin or float the **Data Processing** window.
2. Select the ESCA Spectrum option from the left hand **Data Processing** drop-down menu.
3. Select the Quantification option from the right hand **Data Processing** drop-down menu.
4. Open the relevant experiment files in the **Data Organiser** window.
5. Drag and drop all the spectra which you want to quantify into the table in the **Data Processing** window.

For your information

You can drag and drop an experiment, sample or location into the **Data Processing** window. This drags all child spectra into the table and may include spectra which are not required.

6. Select the chosen spectra from the table. These must all come from the same location. You can use the Ctrl and Shift keys to select multiple files or a range of files. Ctrl + A selects all items in the table.
7. Click the **Process selected data** button  .

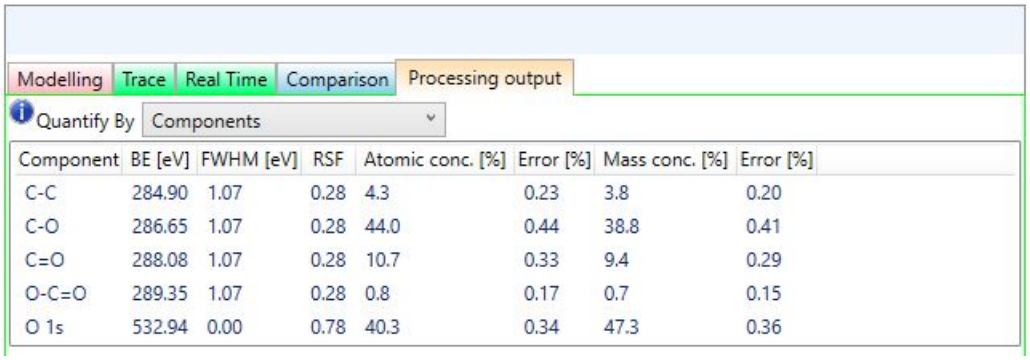
   The data is quantified based on the last model applied to each spectrum. When this is complete the **Save selected results** button is enabled.



8. Click the **Save selected results** button  to save the data to the experiment file.

   A **Processed** folder containing a Quantification report is saved as a child of the location.

9. Double click on the **Quantification** report to open it in a new tab. The quantification report can be viewed as either **Regions** or **Components**. For elements which do not possess any components in **Components** view the region is used instead, that is the equivalent of one component.



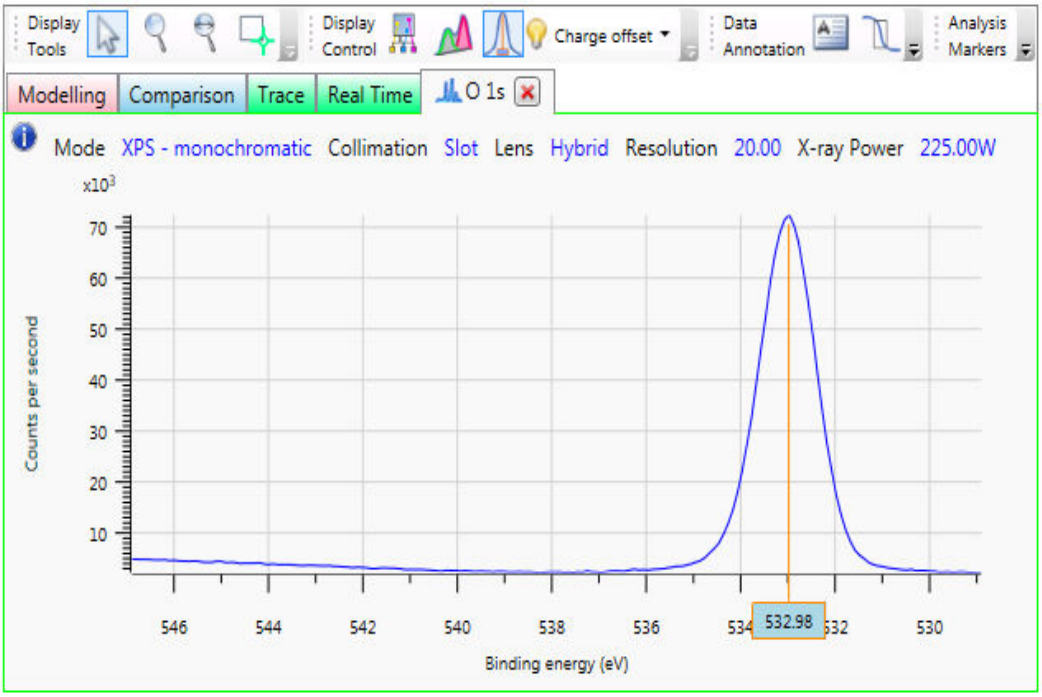
Component	BE [eV]	FWHM [eV]	RSF	Atomic conc. [%]	Error [%]	Mass conc. [%]	Error [%]
C-C	284.90	1.07	0.28	4.3	0.23	3.8	0.20
C-O	286.65	1.07	0.28	44.0	0.44	38.8	0.41
C=O	288.08	1.07	0.28	10.7	0.33	9.4	0.29
O-C=O	289.35	1.07	0.28	0.8	0.17	0.7	0.15
O 1s	532.94	0.00	0.78	40.3	0.34	47.3	0.36

Interacting with Data

Energy Marker

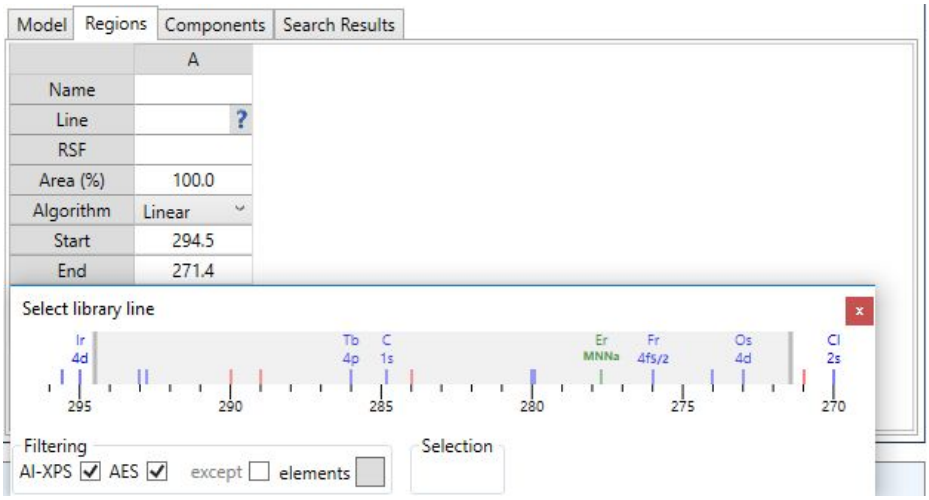
The energy marker tool is available for a spectrum displayed in the **Real Time** tab or opened in a new tab. If the spectrum is opened in a new tab then select the **Energy Measurement** button  to display the tool.

The orange vertical line is designed to help measure off energy values. Drag the box at the bottom of the line across the spectrum. The energy of the line is shown in the box.



Select from library tool

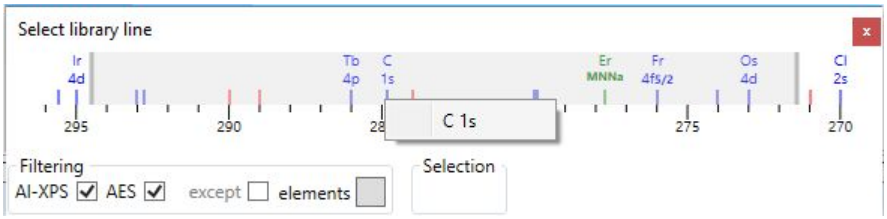
This tool is available in the **Modelling** window in both the **Regions** and **Components** tabs. When you define a region or component a column is created in the table. Click in the **Line** box to display the ? button then click on this button to display the **Select library line** window. This shows the selected energy region of the spectrum and the element lines in this range.



The red lines on the scale indicate that more than one line is present; either two halves of a doublet or the overlap of two different photoelectron lines. The green lines on the scale indicate AES lines.

Hovering the mouse over the energy scale axis changes the mouse pointer to ◀•▶ which allows the middle mouse button to be used to expand or collapse the energy scale range about the pointer. The scale can also be moved left and right by dragging the axis.

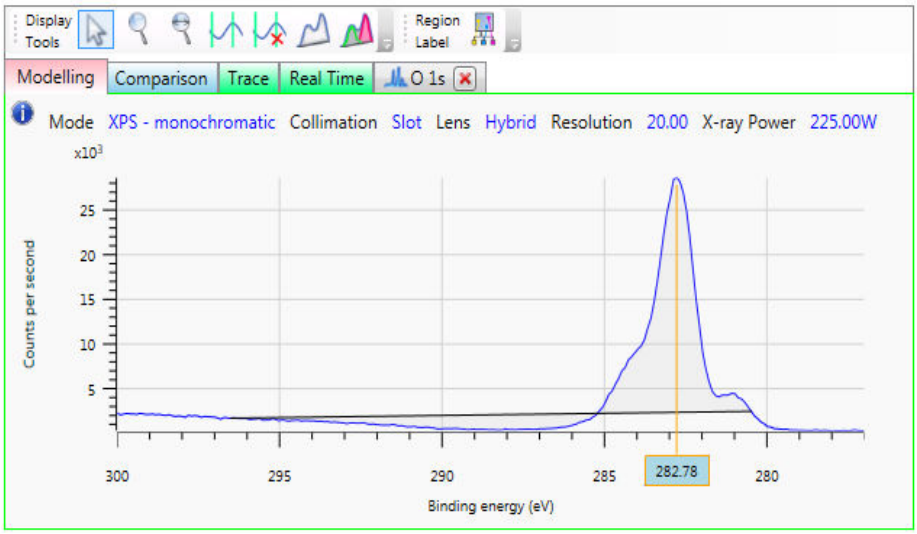
To populate the **Line** box, click on one of the photoelectron line names shown to get a list of the possible photoelectron lines and then select the appropriate line.



Charge correction tool

The charge correction tool is located in the **Modelling** tab. It is used with a spectrum acquired from an insulating sample to correct for the shift to lower binding energy as a result of the charge neutralisation process. The energy of the orange energy marker line is shown in the box below it.

Note that the charge correction can be applied either before or after a model has been applied but it must be applied before saving a model if the saved model is to include the charge correction.



For your information

There are two **Charge Offset** buttons and in the **Modelling** window. Use these with the charge correction tool as follows:

- If you have created models (that is, regions and/or components) for the chosen spectra select the **Model energies are adjusted with the charge offset of the spectrum** button . This ensures that a model stays with the data when the charge correction is applied.
- If you have applied models to the chosen spectra from other data (see "Applying a Model to Other Spectra" on page 95) select the **Model energies are independent of the charge offset of the spectrum** button . This ensures that a model does not move when the charge correction is applied to a spectrum.
- If you have not yet created or applied models then either button can be selected.

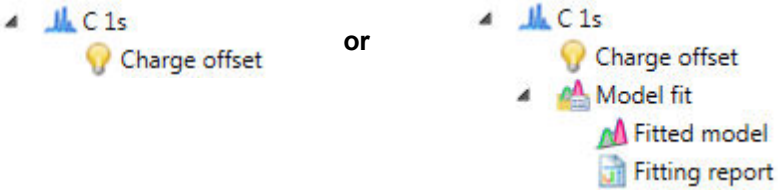
To correct the spectrum:

1. Drag the value box to move the line to a known peak, usually C 1s in a wide scan or the C-C peak of a narrow region C 1s spectrum.
2. When in position double click in the box to highlight the energy.
3. Type in the correct energy then press the return key.

   The spectrum shifts to the corrected energy and the marker line turns green to show that the correction has been applied.

   If a red rectangle is shown after the spectrum has been shifted then the incorrect **Charge Offset** button was selected and the region now extends beyond the acquired spectral range. The region can be modified to bring it back inside the spectral range.
4. To view the value of the offset hover over the energy value box.
5. Save the charge correction by clicking the **Save selected models as experiment data** button  below the **Data Fitting** table.

   Charge offset data is added to the experiment file as a child of the corrected spectrum. If a model has been applied then clicking the save button saves both the model and charge correction.

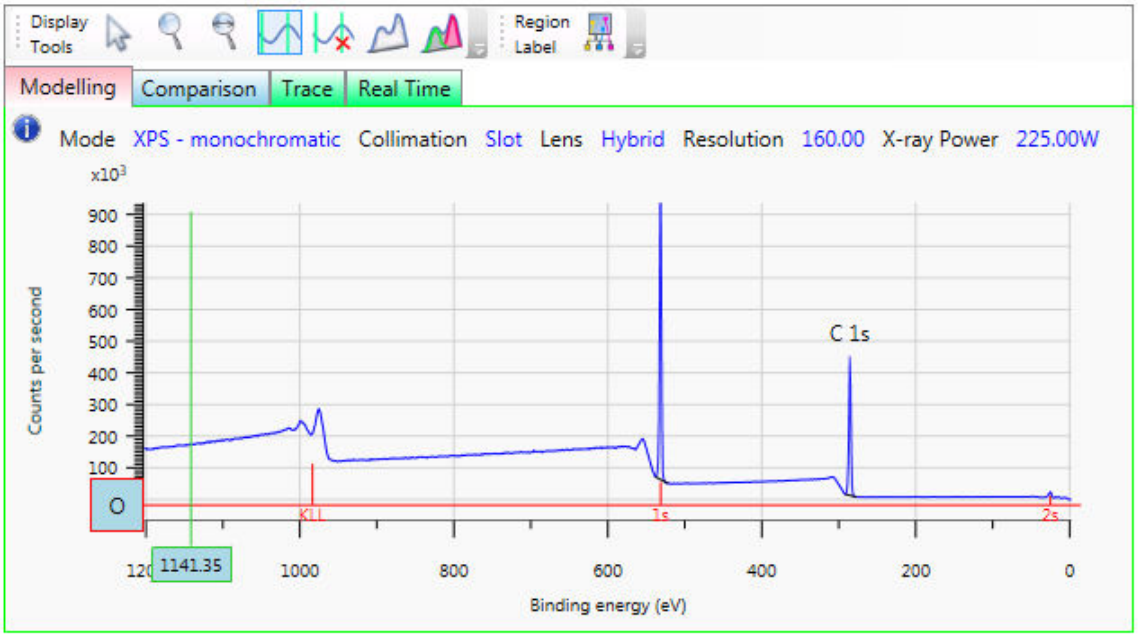


6. Double click on the Fitted model to view the spectrum with the model and charge correction applied.

Element Lines tools

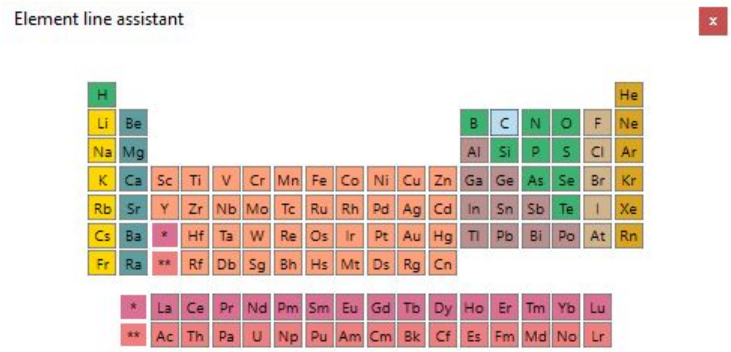
This tool is designed to help with the identification of element lines usually from survey spectra. It is available in both an opened spectrum tab and in the **Modelling** tab. However it is best used in the **Modelling** tab because the spectrum can be charge corrected first.

Click on the **Add Element Lines** button  to display the Element marker bar. This consists of an element text box showing the selected element and a horizontal red bar with a series of red marker lines which show the photoelectron lines for the selected element.



To change the element displayed, either:

- Click in the element text box to obtain a flashing cursor. Type in the new element symbol, for example C or Sb. Note this is case sensitive.
- Right click in the element text box and select **Periodic table** to display the **Element line assistant** window. Select the element from the periodic table.

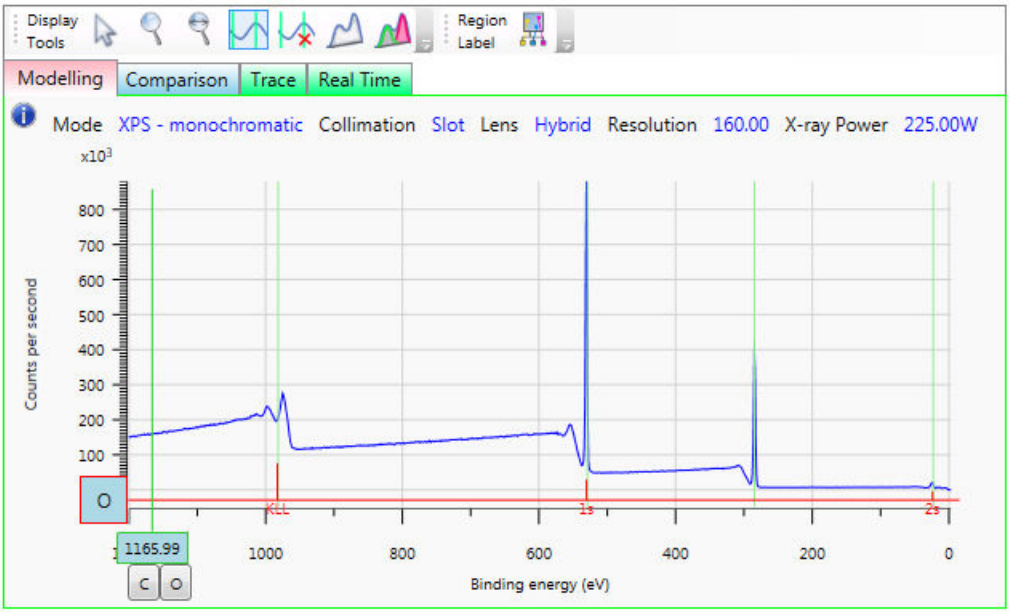


The marker bar can be moved up and down to make it easier to match the data by dragging the element text box. The height of the photoelectron lines can also be changed by hovering over the element text box and rolling the middle mouse button forward to increase the height and towards you to decrease the height.

For your information

The relative line heights represent the relative peak areas. Since Auger peaks tend to be quite broad they may appear to be relatively tall compared to the core lines.

Double clicking in the element text box (or on an element in the **Element line assistant** window) adds the selected element lines to the spectrum display. These are shown as green vertical lines. A small button labelled with the element selected is added below the X axis. This can be repeated for several elements allowing the lines of multiple elements to be viewed at the same time. Clicking on one of the small buttons displays the lines for that element in the marker bar.



The **Add Element Lines** tool  can also be used to determine which elements have lines within a selected energy range. Draw a line to define the energy range. All the elements which possess lines within this range are displayed as buttons at the bottom of the spectrum and all their lines are displayed in green. Clicking on an element button displays its photoelectron lines on the red marker bar. These can be compared with the peaks present in the spectrum. The element lines and button for an incorrect match can be removed by double clicking on the button. The range selection can also be used in conjunction with the **Zoom and scroll the display in both axes** tool  or the **Zoom and scroll the display horizontally** tool  to refine the selection range. A better match is also obtained if a spectrum from an insulating sample is charge corrected first.

The **Remove element lines** tool  can be used in a similar manner to remove elements from the button list. With this tool selected draw a line over the region from which you wish to remove the element lines. The elements with lines in this range are removed from the button list and their lines are removed from the spectrum. By drawing a large energy range all elements can be effectively removed from the list.

Overlaying Spectra

Overlaying of spectra is performed in the **Comparison** tab.

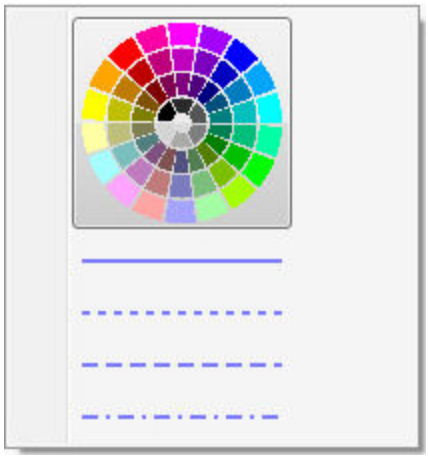
To overlay spectra, ensure that the **Comparison** tab is selected and then drag and drop each spectrum from the **Data Organiser** window to the display area.

For your information

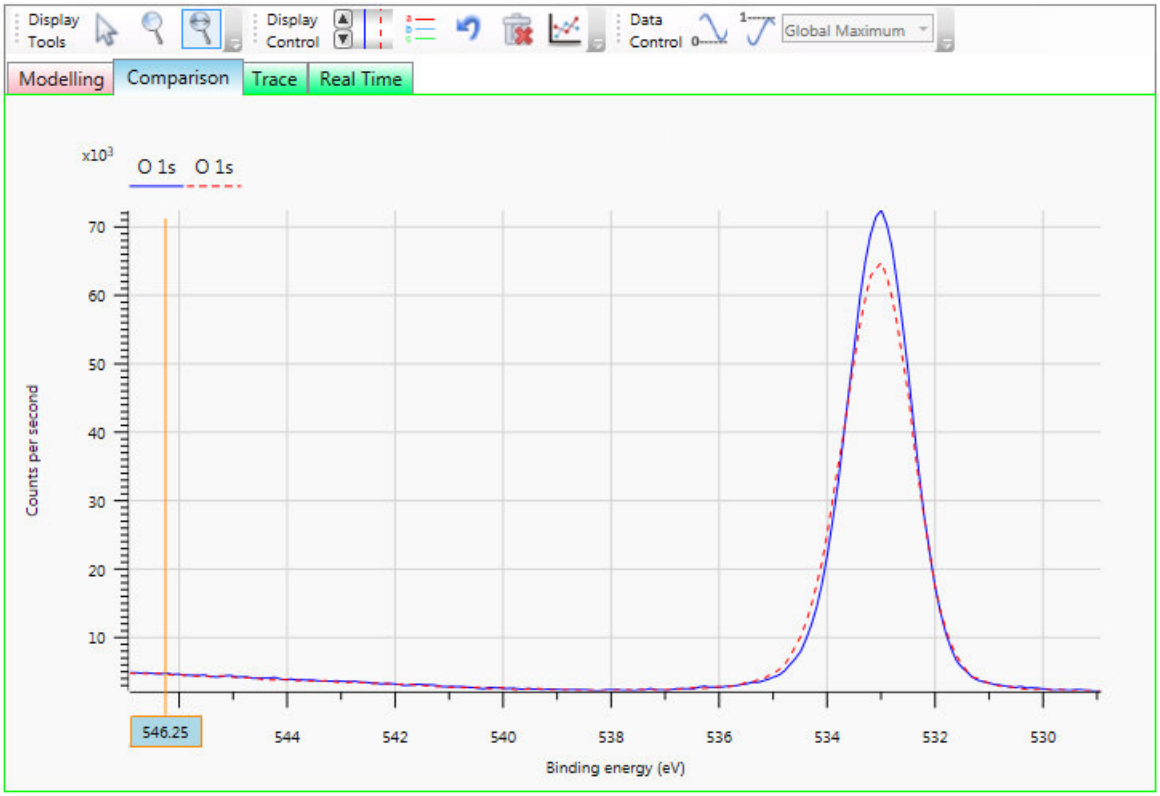
The Comparison display can also be populated by exporting spectra from the **Modelling** window. This is particularly useful for experiment types where large numbers of spectra need to be compared. See the Advanced Operations User Manual for more details.

Once you have dragged the first spectrum into the display area a control bar is displayed. The colour and appearance of the lines used for the spectra are determined in the **Display Control** section of the control bar.

A series of lines is displayed to the right of the arrow buttons. The number of lines is controlled using the up and down arrows. The colour and appearance of the lines determines the colour and appearance of the lines used for the spectra. These can be changed by right clicking on a line to display a colour wheel and then selecting the new colour and appearance of the line. If more spectra are displayed than the number of lines then the colours cycle round.



The spectra can be offset on the Y axis by hovering over the number scale and rolling the middle mouse button. The spectra can be offset on the X axis also by hovering over the number scale and rolling the middle mouse button.

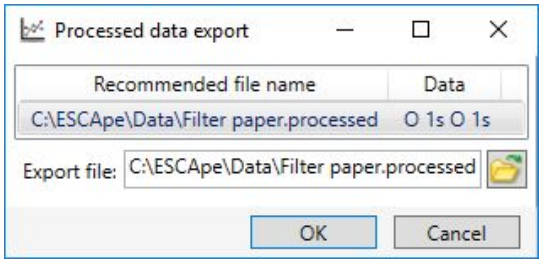


When you are happy with the look of the display you can create a report (.processed file) from the display data. This allows the data to be plotted and viewed in Excel. To do this:

1. Click the **Make report from display** button  .

   The **Processed data export** dialogue is displayed. This shows a list of one or more recommended file names.

   - If all spectra originate from the same experiment file, this is a file with the same name and location as the experiment file.
   - If spectra originate from more than one experiment file, a recommended file name and location is listed for each experiment file.



2. Either:
   - Ensure that a recommended file name is selected.
   - Enter a new name and/or location or use the **Select the folder for comparison data export** button  to browse to the new folder.
3. Click **OK** to make the report. If a file with the selected name and location already exists the report is added to the file. If the file does not exist a new file is created.

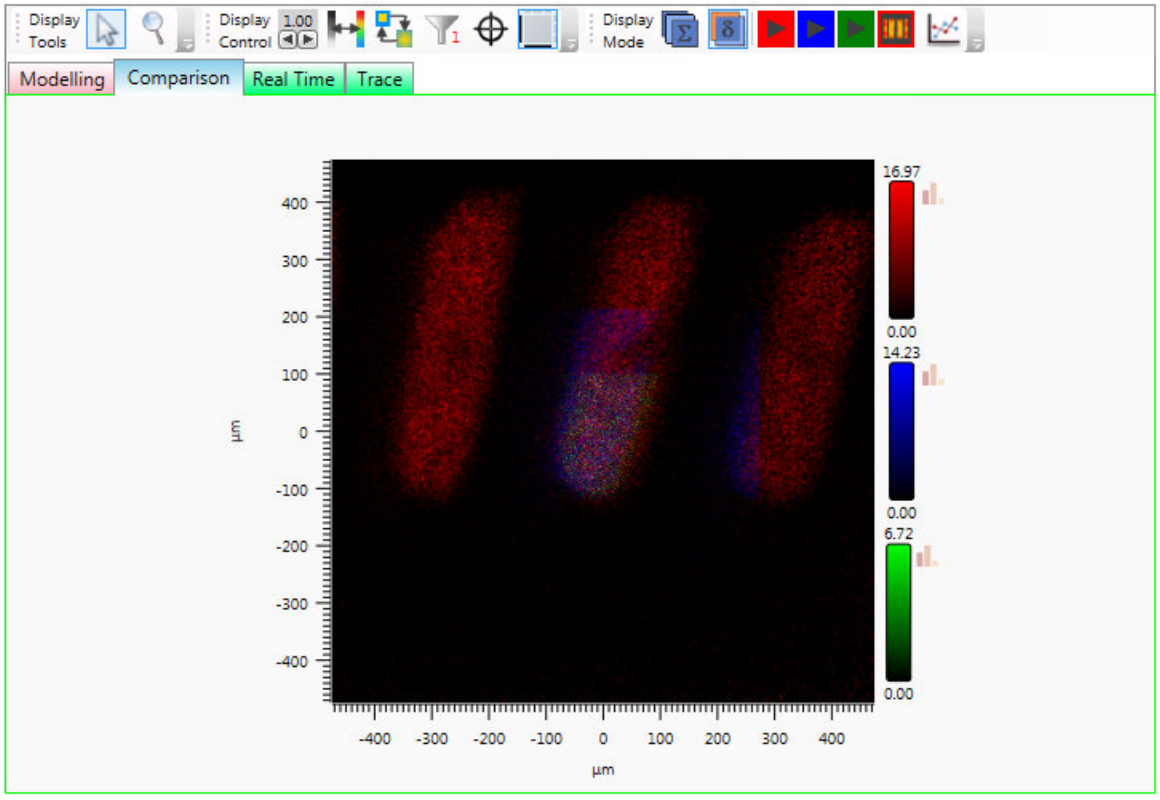
The report can be viewed using the Kratos Add-In to Excel as detailed in "Importing Data from .Processed Files into Excel" on page 120.

Overlaying Images

Overlaying of up to three XPS images is performed in the **Comparison** tab.

To overlay images:

1. Ensure that the **Comparison** tab is selected.
2. Drag and drop the first image on to the display area.
3. Click on the **Display image differences** button  to display four more buttons  . This turns the image red.
4. Drag and drop the next image on to the blue or green arrow.
5. If required, drag and drop a third image on to the third arrow.



6. If required use the **Adjust image brightness** buttons  to alter the brightness of the images.

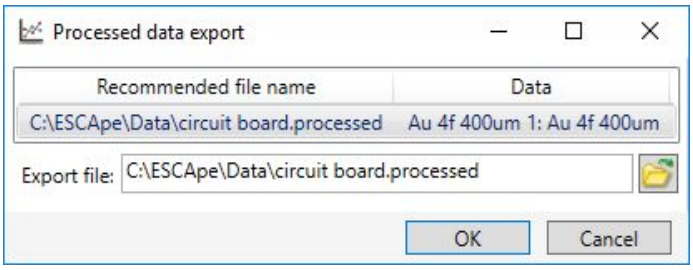
An image can be replaced by dragging a new image on to the associated arrow. An image can be enabled or disabled by clicking on the button with the associated colour. If an image is disabled the arrow changes to ⊘.

To return the display to a single image, click on the **Display image differences** button  again.

When you have overlayed the images you can create a report (.processed file) from the display data. This allows the data to be viewed in Excel.

To do this:

1. Click the **Make report from display** button  .
   The **Processed data export** dialogue is displayed. This shows a list of one or more recommended file names.
   - If all images originate from the same experiment file, this is a file with the same name and location as the experiment file.
   - If the images originate from more than one experiment file, a recommended file name and location is listed for each experiment file.



2. Either:
   - Ensure that a recommended file name is selected.
   - Enter a new name and/or location or use the **Select the folder for comparison data export** button  to browse to the new folder.
3. Click **OK** to make the report. If a file with the selected name and location already exists the report is added to the file. If the file does not exist a new file is created.

The report can be viewed using the Kratos Add-In to Excel as detailed in "Importing Data from .Processed Files into Excel" on page 120.

Appendix A
Data

Data Organiser

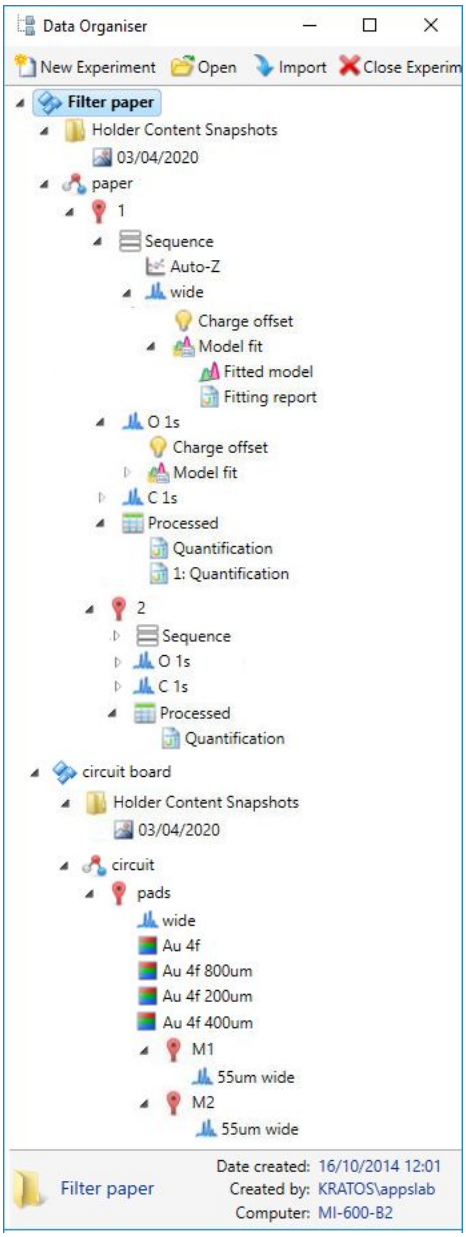
Structure

The Data Organiser window displays the hierarchical structure of the experiment files that are open in ESCApe. Within each file, data is grouped according to sample. Within each sample, data is grouped according to location.

In this example:

- Filter paper and circuit board are both experiments.
- paper is a sample that has been used for acquisitions with the Filter paper experiment selected.
- 1 and 2 are locations on the paper sample and pads is a location on the circuit sample.
- Data is listed under a location in the order in which it is acquired. The exception to this is when data is acquired from other data. For example, when the spectrum was acquired from the Au 4f 400um image then data for this spectrum was added as child data of the image.
- Where the method includes regions the data is named after the region.
- Extra information is available as follows:
    - Click on the data item to display extra information such as the date and time the data was created. For data such as a spectrum or image this is time the acquisition started.

- The Holder Contents Snapshots folder in the Filter paper experiment file contains an optical image which shows green markers indicating the positions of sample locations 1 and 2.
- Click on the data item and then hover over the icon to display the acquisition settings used to obtain the data.



Opening data from an experiment file

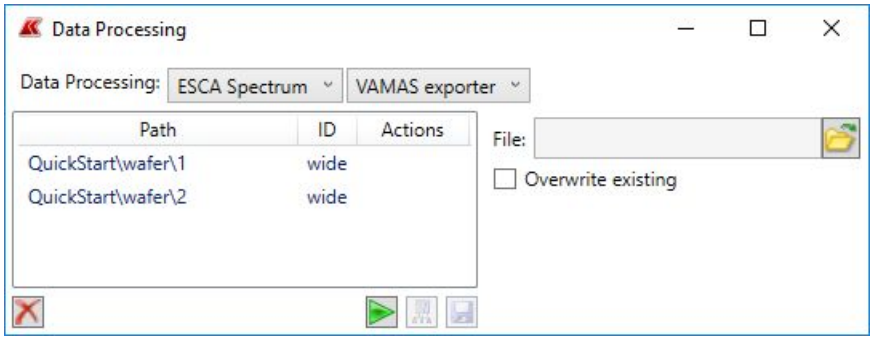
1. Expand the experiment file as required to display the relevant entry in the file.
2. Double click on the relevant entry to display the data in the display area.

Exporting Data Using the VAMAS Format

Raw data from ESCApe can be exported to a file using the VAMAS standard data transfer file format.

Note that data from more than one spectrum can be exported in a single file provided that all spectra are from the same experiment file. Similarly data from more than one image can be exported in a single file provided that all images are from the same experiment file. However data from spectra ***and*** images cannot be exported in a single file.

1. Pin or float the **Data Processing** window.
2. Select the type of data, for example ESCA spectrum or ESCA image, from the left hand **Data Processing** drop-down menu.
3. Drag and drop all the data which you want to export into the table in the **Data Processing** window.
4. Select the VAMAS exporter option from the right hand **Data Processing** drop-down menu.
5. Select the chosen data from the table. Remember that it must all be from the same sample.
6. If a file name is already shown in the **File** box and you want to overwrite the data in this file, select the **Overwrite existing** check box then continue with Step 8.
7. If you want to create a new file or overwrite a different file, use the  button to display the **Select VAMAS Export File** window. Navigate to the appropriate folder. Either:
   - Select an existing VAMAS (.vms) file to overwrite, click **Save** and then **Yes** to confirm that you want to replace the file.
   - Enter a new **File name** and click **Save**.



8. Click the **Process Selected Data** button  .
   Data is written to the file specified in Step 6 or Step 7.

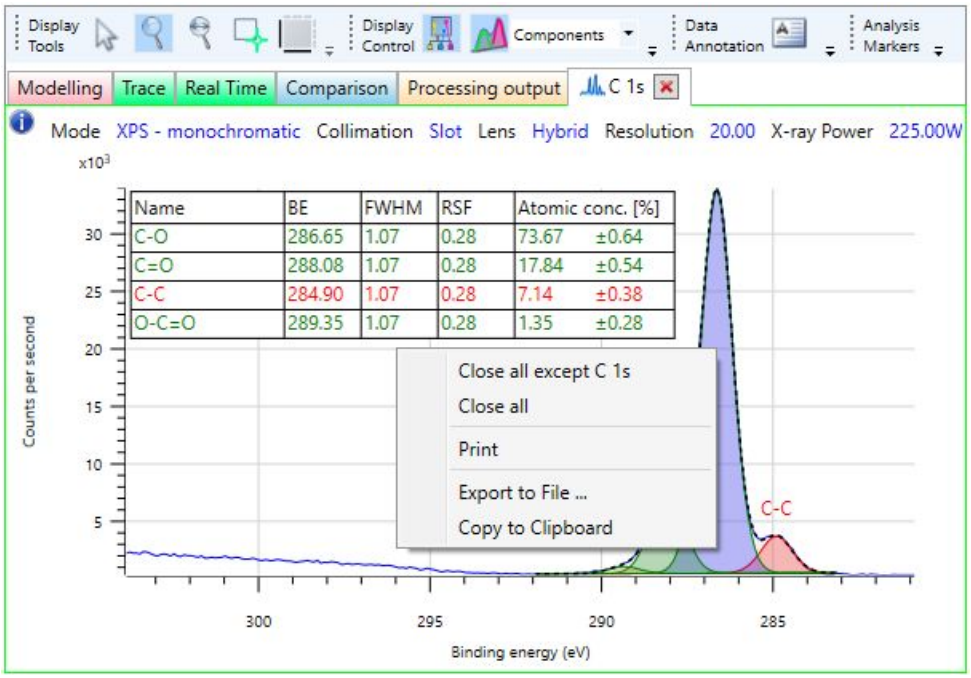
Copying a Displayed Data Item to the Clipboard

Displayed data items can be copied as images to the clipboard. This includes spectra, images, depth profiles and models.

Some examples are given below.

Spectra

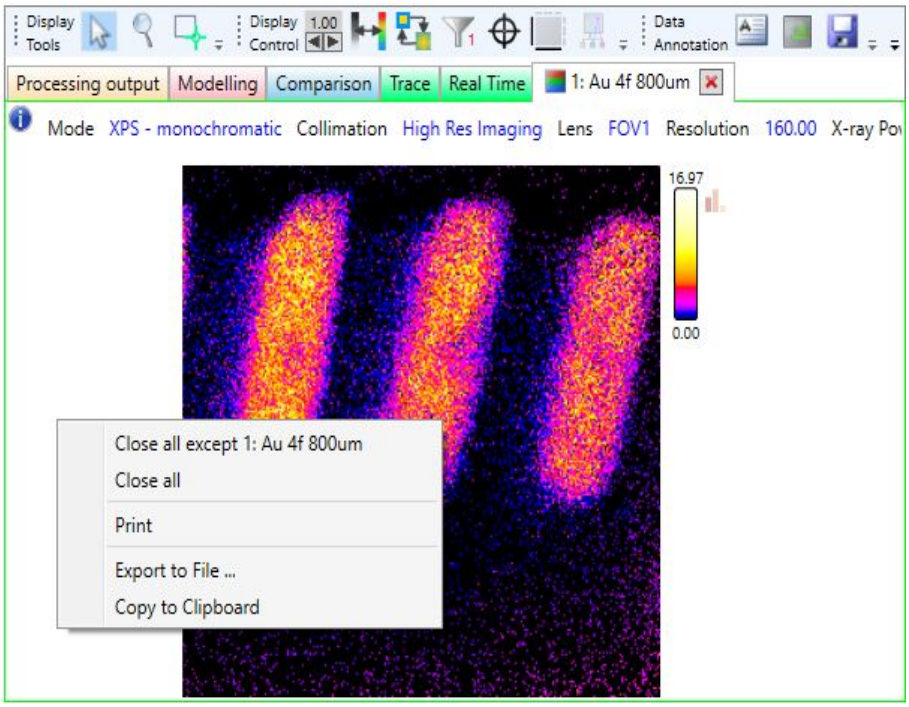
1. Open a spectra in the display area.
2. If required, display the components or regions.
3. Right-click within the graph to display a context menu.



4. Select **Copy to Clipboard**.
   An image of the spectra is copied to the clipboard.

Image

1. Open an image in the display area.
2. Right-click in the white area surrounding the image to display a context menu.



3. Select **Copy to Clipboard**.
   The image is copied to the clipboard.

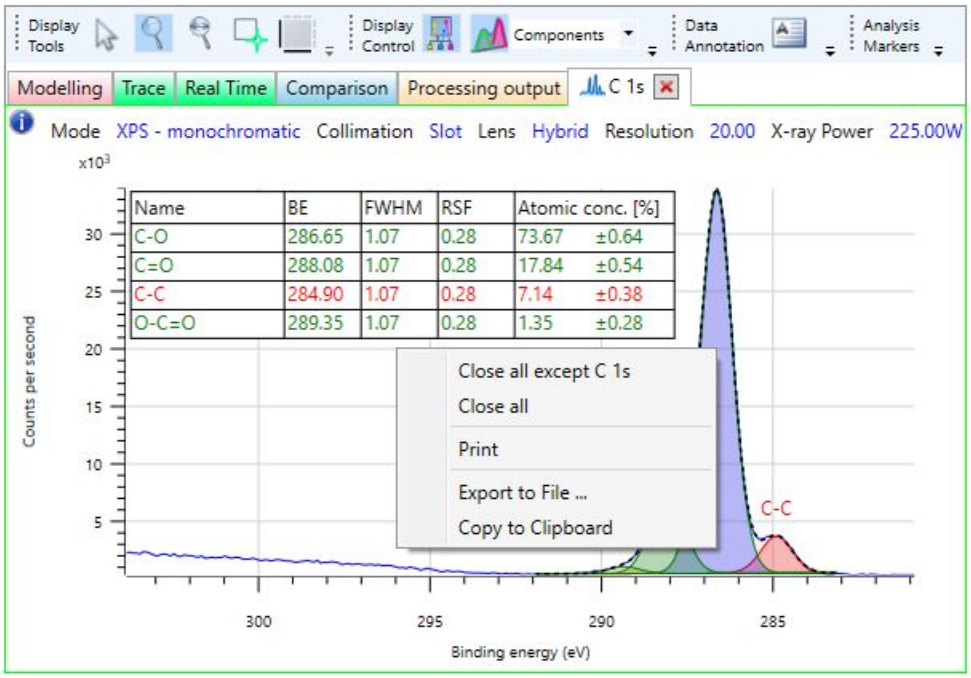
Exporting a Displayed Data Item

Displayed data items can be exported as images. This includes spectra, images, depth profiles and models.

Some examples are given below.

Spectra

1. Open a spectra in the display area.
2. If required, display the components or regions.
3. Right-click within the graph to display a context menu.

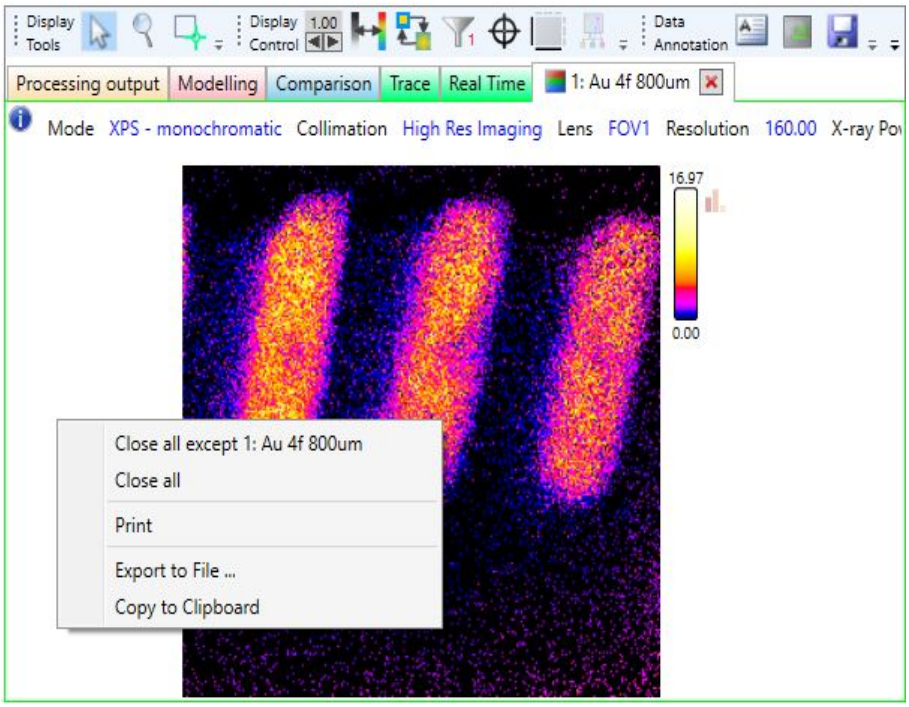


4. Select **Export to File....**
5. An **Export image** dialogue is displayed.
6. Select the location, name and image type for the file.

   An image of the spectra is saved.

Image

1. Open an image in the display area.
2. Right-click in the white area surrounding the image to display a context menu.



3. Select **Export to File....**
4. An **Export image** dialogue is displayed.
5. Select the location, name and image type for the file.

   An image of the spectra is saved.

Importing Data from .Experiment Files into Excel

Data from experiment files can be imported directly into Microsoft Excel using the Kratos Excel Add-In.

To import data:

1. Open Excel.
2. Select the **Data** tab.
3. Click on the **Experiment** button 

to display an **Open experiment file** window.
4. Navigate to the experiment file (.experiment).
5. Either:
   - Double-click on the file.
   - Click **Open**.

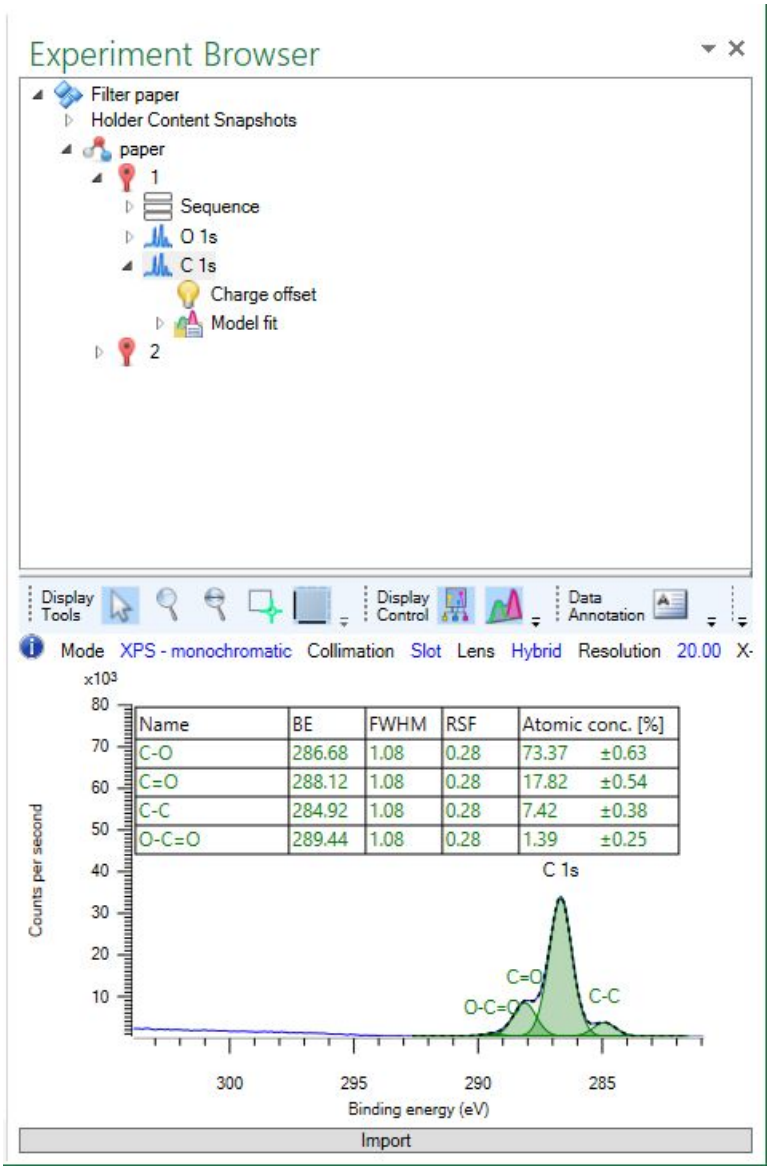
   An **Experiment Browser** pane is added to the right hand side of the Excel window.

   This displays the data in the same format as the ESCApe experiment tree in the **Data Organiser**.
6. Click on an item to show the data in the display area. Selecting a data item shows the raw data and selecting a model shows the model and the data. Similarly selecting a report shows the tabulated data.

For your information

If you click on the  button in the display area to open the display information then the content of the display information is also imported as the first two columns of the Excel spreadsheet.

7. Make any changes to the data in the display area. For example:
   - Change between Binding energy or Kinetic energy by right clicking on the axis label.
   - Change between Counts per second and Counts by right clicking on the axis label.
   - Hide or display grid lines.
   - Add the Components or Region information.
   - Change the magnification.
   - Add text annotations.



8. Copy the data into Excel by clicking on the **Import** button below the data. This creates a new sheet containing all the data points and reproduces the graphical data.

   The data can then be manipulated using the tools available in Excel.

Importing Data from .Processed Files into Excel

Data from .processed files can be imported directly into Microsoft Excel using the Kratos Excel Add-In.

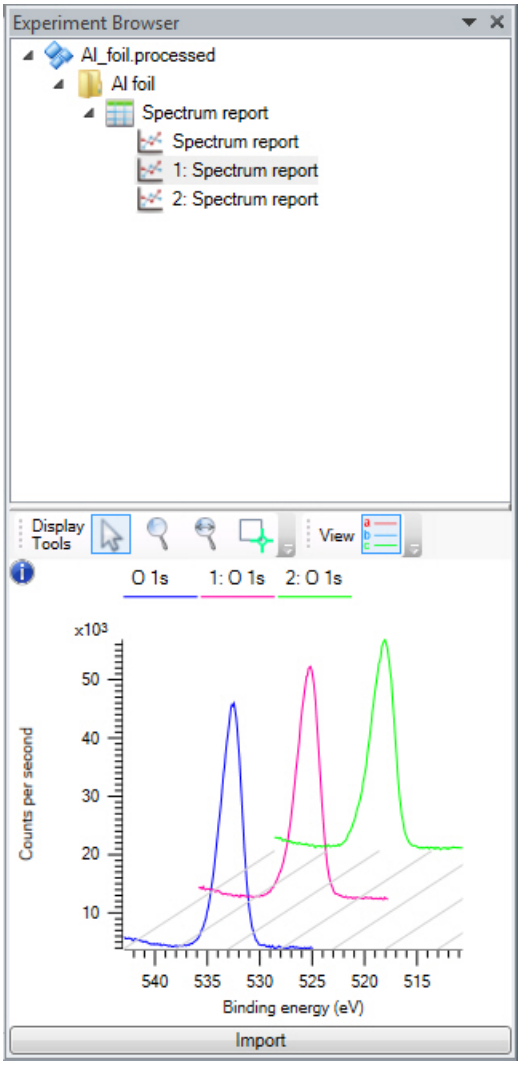
To import data:

1. Open Excel.
2. Select the **Data** tab.
3. Click on the **Experiment** button  to display an **Open experiment file** window.
4. Select **Processed file (*.processed)** from the drop-down menu just above the **Open** and **Cancel** buttons.
5. Navigate to the processed file (.processed).
6. Either:
   - Double-click on the file.
   - Click **Open**.

   An **Experiment Browser** pane is added to the right hand side of the Excel window. This displays the structure of the processed file.
7. Click on an item to show the data in the display area.

For your information

If you click on the  button in the display area to open the display information then the content of the display information is also imported as the first two columns of the Excel spreadsheet.



8. Copy the data into Excel by clicking on the **Import** button below the data. This creates a new sheet containing all the data points and reproduces the graphical data.

   The data can then be manipulated using the tools available in Excel.