INCA Energy

Operator Manual

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Section 1 - INCA Energy System Overview

The main components that make up an INCA Energy system are:

- PC
- x-stream module to control X-ray acquisition and/ or mics module to control imaging
- EDS detector to detect X-rays

The PC is loaded with the INCA Energy software and an IEEE 1394 card.

System Block Diagram

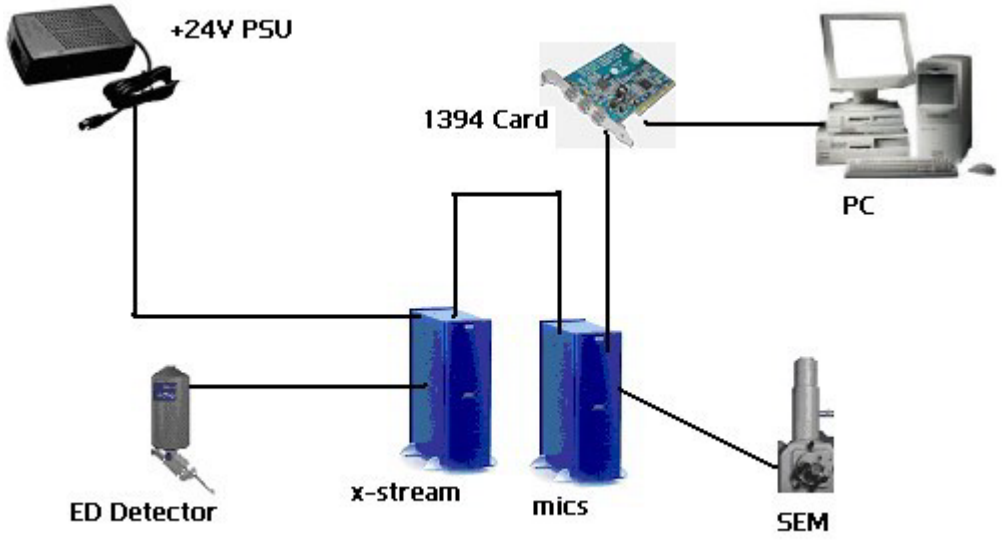


Figure 1: INCA Energy System

Section 2 - Introduction to INCA Energy Software

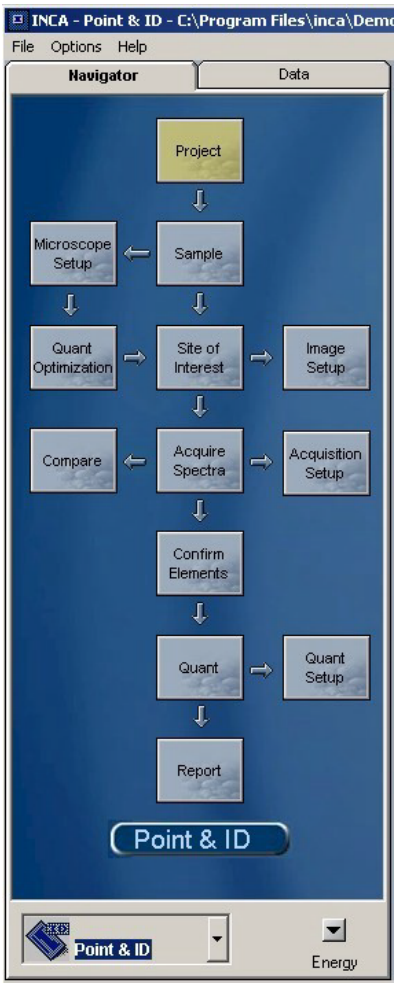
The INCA software platform has four main components:

- Navigators
- Data Management
- Help
- Energy Options

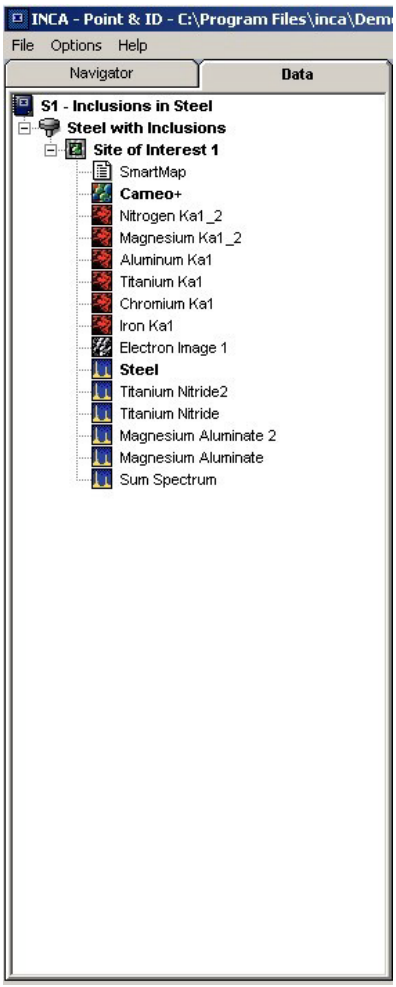
Navigators

The INCA software platform has unique navigators for guiding the user through the stages of the microanalysis process from starting a new project to producing a hardcopy report.

You can easily switch between a Navigator, which directs the work flow, and the data tree which keeps a visual record of work progress.



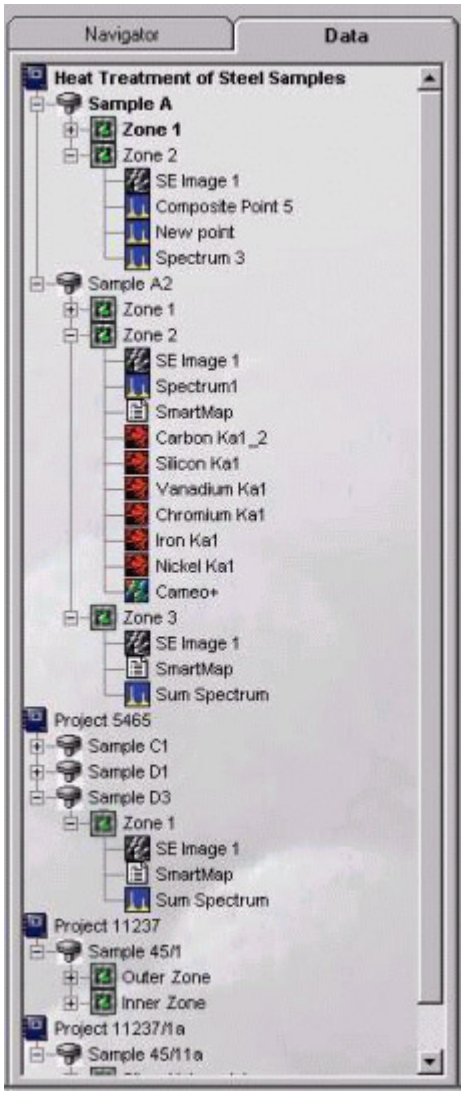
Point & ID Navigator



Data tree

Data Management

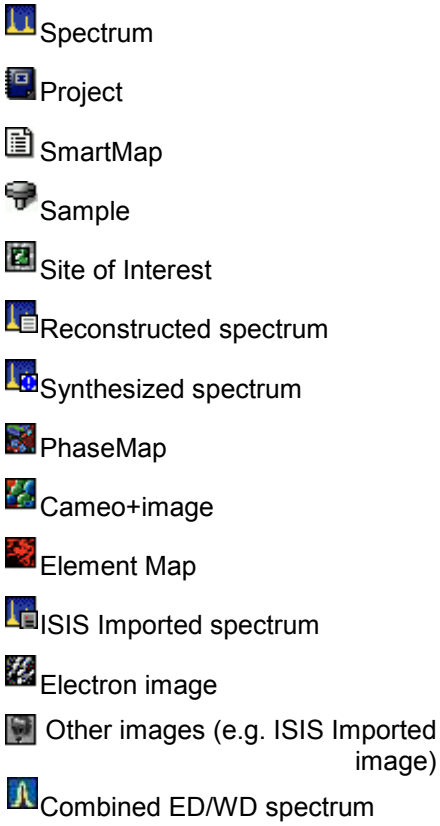
Data within INCA Energy and Energy TEM is archived in a logical and easily accessible manner and can be directly viewed in the Data tree. To access the Data tree, press the Data tab to the right of the Navigator tab.



Projects

- Within INCA Energy, data is managed within Projects as illustrated by the Data tree shown above. As you acquire data, you will see your data build up in the Data tree. The entries in the Data tree can be renamed within the Data tree view.

- Within a Project you may examine more than one Sample, and on each Sample, more than one Site of Interest. Data acquired from an individual Site of Interest is stored together for example electron or Cameo+ image, spectra and maps as easily recognizable icons as displayed below:



- Note that more than one Project can be viewed at any one time in the Data tree.
- A Project is a single file with the extension .ipj which can be saved in any directory on any drive in the conventional Windows™ way. Note that the name of the current Project is displayed as part of the Menu Title.
- At any time you can save your Project by going to the main menu, under File and selecting 'Save Project as' or using Ctrl and S on the keyboard.

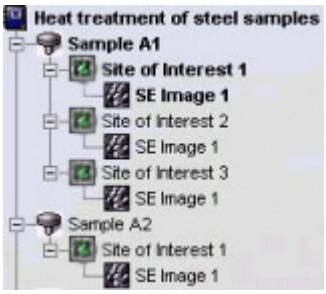
Autosaving your Project

- You can set the Autosave function to save your current Project at regular intervals. This can be set by going to the main menu, under Options, and checking the box in Preferences under 'Saving'.
- If you open a Project and you have the Autosave function on, Autosave will save the Project on top of the original.
- If you create a Project and save it and you have the Autosave function on, further autosaves will save the Project to the original saved location.
- If you create a new Project and you have the Autosave function on, Autosave will save the Project to a recovery file.
- The recovery file is located under Program files\INCA\Data. If you wish to open this file, make a copy of this file with a different name in a different directory.

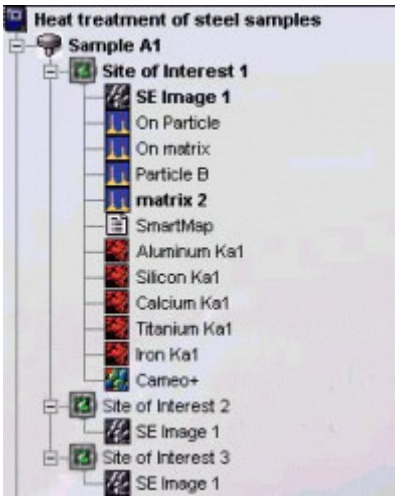
- Note that Autosave does not take effect during data acquisition.
- Autosave does not function on read-only Projects.
- If you have multiple Projects open, ensure that you save your Projects.

Samples and Site of Interest

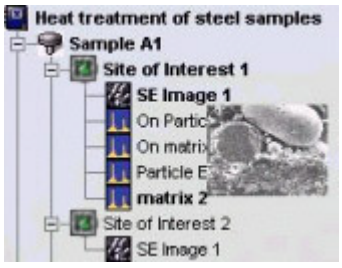
- Within a Project you may analyze more than one Sample, and on each Sample, more than one Site of Interest.



- Data acquired from an individual Site of Interest is stored together for example electron or Cameo+ image, spectra and maps as easily recognizable icons. The current analytical conditions such as kV, Mag and Quant optimization data are also stored with the data.



- Thumbnail images of the individual images and spectra can be easily viewed by hovering over the data labels with the mouse.



Exporting spectra, images, maps, linescans and Cameo+ data

Electron images, maps, linescans, Cameo+ images and spectra can be exported from INCA in a variety of formats thus allowing them to be read by appropriate software packages. You can export either from the data tree or from the image/spectrum viewer:

Data tree

To export from the Data tree click with the right hand mouse button on the data label and select Export. This enables the user to convert and export the data in file formats such as JPG and TIF:

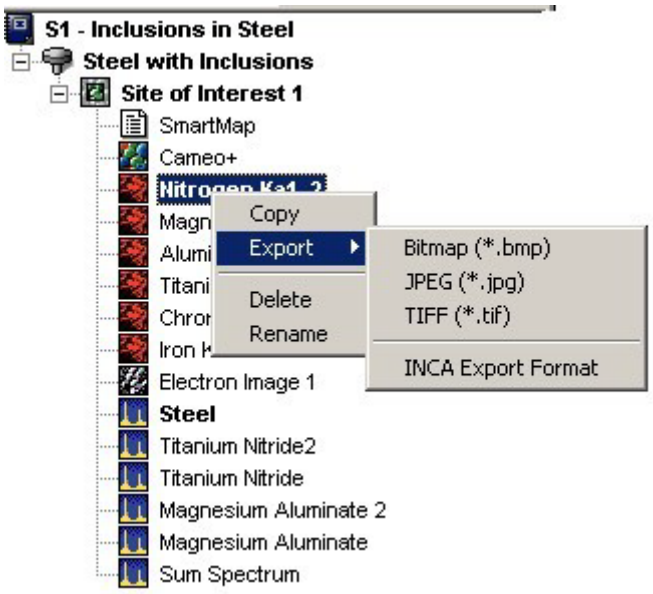
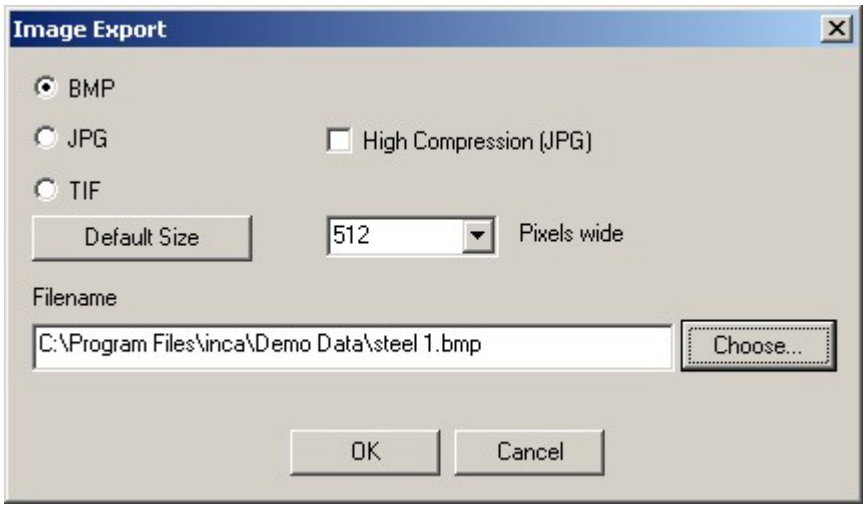


Image Viewer

When you click with the right hand mouse button on an image or map you have two options for exporting:

- **Export**

  This allows the data to be converted as a bitmap (BMP), TIFF (TIF), or JPEG (JPG, with optional high compression) file. You can select the image size from the drop-down list. The default size is set to the size of the original image. Enter the file name and select a folder in which you wish to save the image:



- **Export Data Only**

  This allows the raw data to be exported in suitable formats for further processing e.g., 8-bit or 16-bit TIF and TSV (Tab Separated Variable). Only raw data is exported without contrast enhancement or annotation. However, the TIFF Color option will save a color image with contrast enhancement. The scaling information is also included in the exported data.

  If you have analySIS® software (Soft Imaging Systems) installed on your computer you can export the data to the analySIS® database. There are two options for exporting data to the analySIS® database:

  - **analySIS® database:** Use this option if you wish to export the raw data.
  - **analySIS® database (color):** Use this option if you wish to export either a Cameo+ image or a contrast enhanced image.

Spectrum Viewer

When you click with the right hand mouse button on the spectrum you are allowed to convert and export the spectrum as BMP, TIF, JPG or WMF(Metafile). You can also export the spectrum in EMSA or ISIS format. If you have analySIS software installed on your computer you can export the data to the analySIS database.

Linescan Viewer

You can export a linescan in a variety of formats. When you click with the right hand mouse button on the linescan you are allowed to convert and export the linescan as BMP, JPG or TSV etc.

- Note that it is the data as currently displayed which is exported. Therefore, if the current image has been zoomed, the contrast or brightness, the color or the spectrum scaling has been changed, this will be included in the conversion.

- Note also that images exported to files include the scalemarker.

Exporting SmartMap data

You can export the current SmartMap data by selecting 'Export SmartMap' under File from the main menu.

Exporting data in an INCA format

Electron images, maps, Cameo+ images and energy dispersive (ED) spectra can be exported from INCA in an INCA format. These can be displayed within INCA Viewer.

Click with the right hand mouse button on the data label, select Export and then, INCA Export Format. This allows the data to be converted and exported in file formats: .iex for spectra and .mcv for images.

INCA Viewer

INCA Viewer is installed with INCA although INCA Viewer can be installed separately if desired

To start INCA Viewer either:

Double click on the exported file from Windows Explorer

Select INCA Viewer from Start/Programs/Oxford Instruments menu

Copying spectra, images, maps, linescans and Cameo+ data

Spectra, electron images, maps, linescans, Cameo+ images and spectra can be copied to the clipboard from INCA Energy in two ways:

1. By clicking with the right hand mouse button on the data label and selecting Copy.
2. By clicking with the right hand mouse button on the image, linescan etc and selecting Copy. Here you are allowed to choose the size of the image before you copy it to the clipboard:

- The data will be copied to the Clipboard. You can then use your desired application into which you wish to transfer the image etc from the Clipboard.
- Note that it is the data as currently displayed which is copied. Therefore, if the current image has been zoomed, the contrast or brightness, the color, or the spectrum scaling has been changed; this will be included in the copying.
- Note also that images copied to the clipboard include the scalemarker.

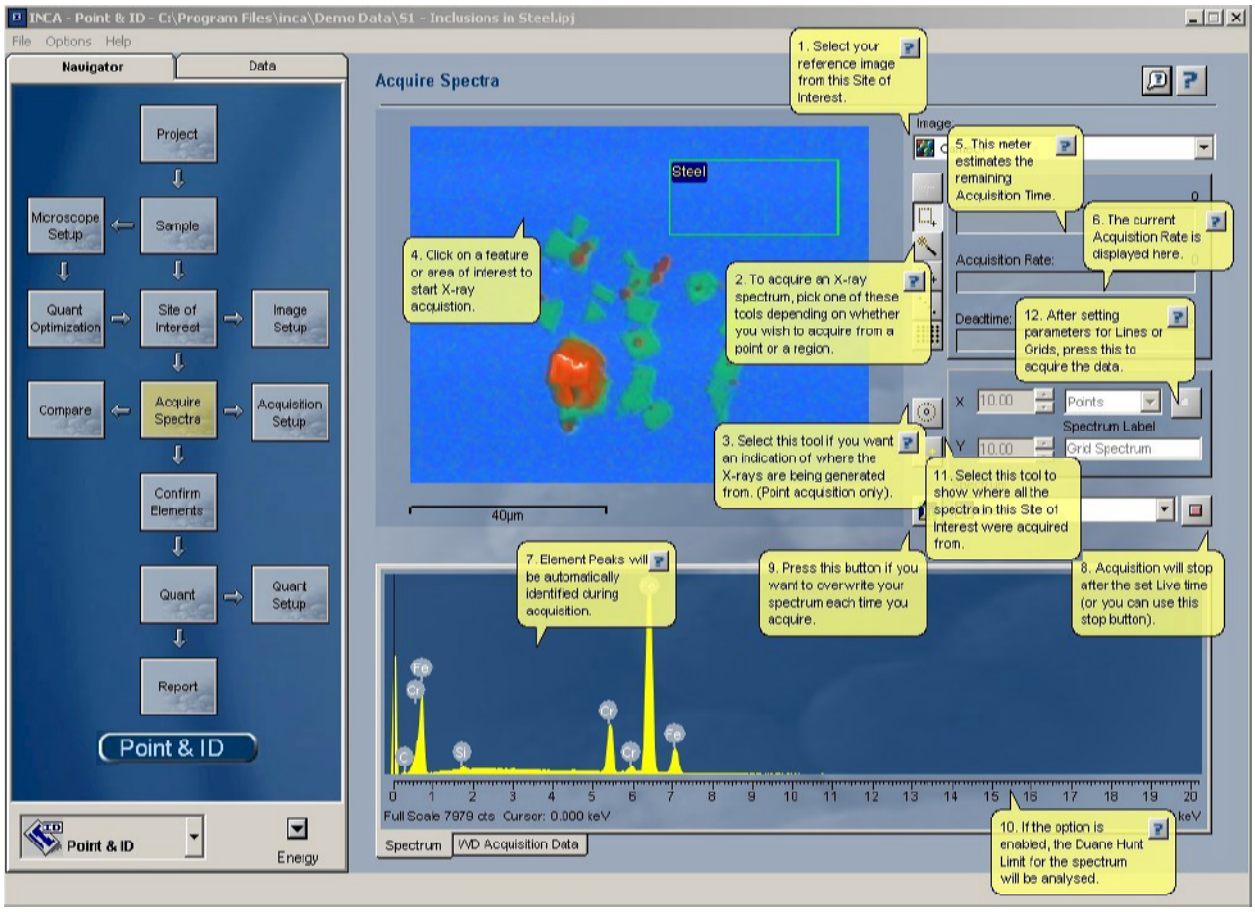
Deleting images, spectra, samples and Sites of Interest

- Spectra, electron images, maps, linescans, Cameo+ images and spectra can be deleted from INCA Energy by clicking with the right hand mouse button on the data label and selecting delete.
- Projects, Sites of Interest and Samples can also be deleted directly from the Data tree by clicking with the right hand mouse button on the appropriate icon. Note: You can not delete the last Site of Interest in a Sample and last Sample in a Project.

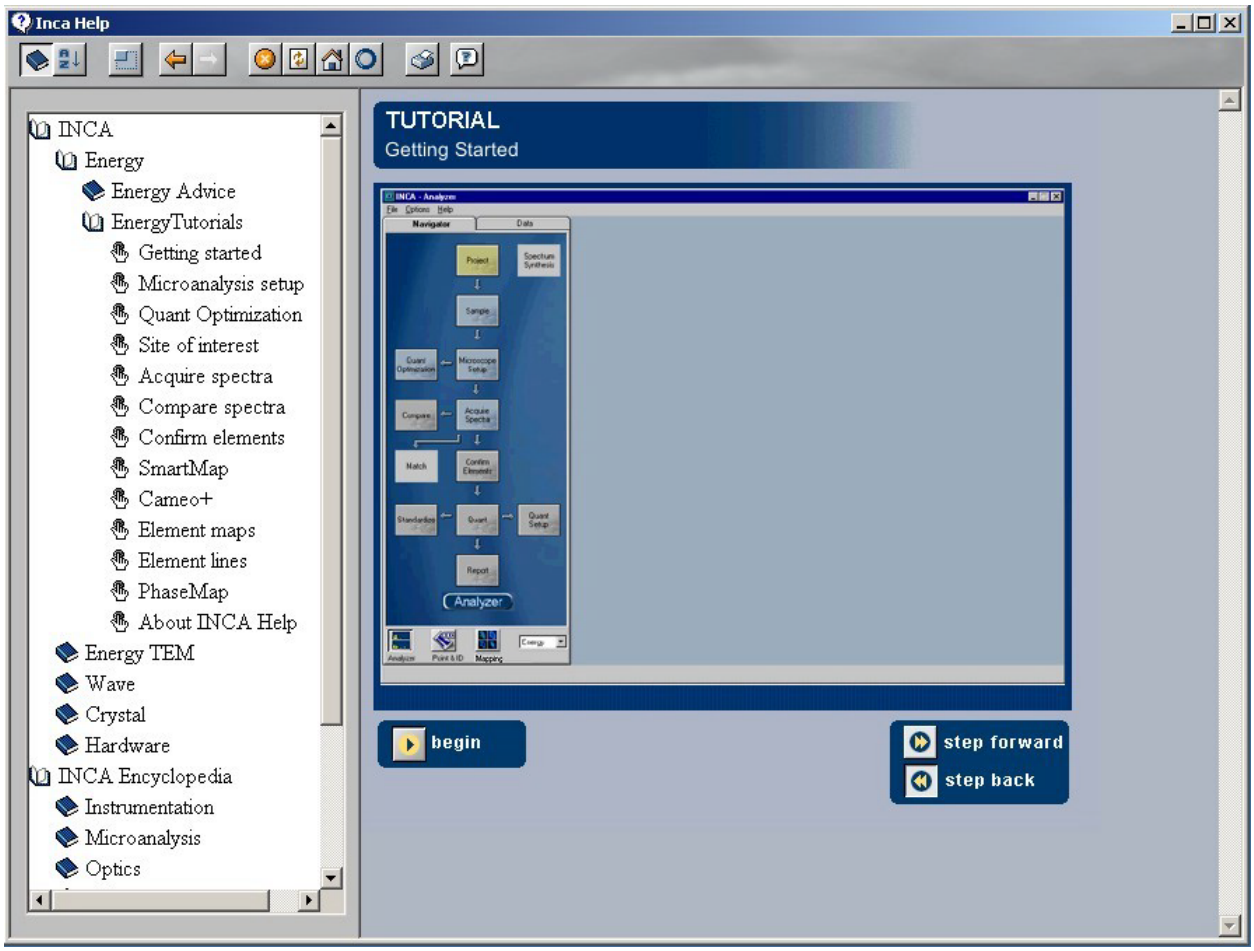
Help

There is an expert multimedia user Help system, which is fully integrated with the applications software. The Help has the following features:

- On-line Bubble help and tool tips for expert advice and guidance through each stage of the microanalysis process.
- A fully integrated and indexed microanalysis encyclopaedia that provides comprehensive description of key microanalysis terms, background, and theory.
- Multimedia training and background tutorials with movie based modules.
- 'Getting Started' multimedia tutorial for immediate training of new user.
- Also, advice on using the application to achieve good results.



INCA Bubble Help



INCA Tutorials

Energy Options

The basic packaged system has two navigators:

- Analyzer
- Mapping

The advanced packaged system has three navigators:

- Analyzer
- Point & ID
- Mapping

There are additional software options such as Cameo+, Spectrum Synthesis, PhaseMap, Matching, AutoMate, GSR, QuantMap and Feature. The basic system can be upgraded in steps by adding new software options easily.

Application Keys

Each software application is supplied as a key-enabled locked module. You need to have an appropriate key for unlocking each application.

Section 3 – Description of Main Steps

This manual describes the main steps to enable the users to carry out analysis using the INCA Energy system. Comprehensive INCA Energy Help is available within the software that users can easily access for further information. List of topics described in the manual are*:*

1. ***Optimum Microscope Conditions Setup for X-ray Microanalysis***
2. ***Quant Optimization***
3. ***Selecting Optimum Conditions for X-ray Acquisition***
4. ***Start/Stop/Resume Spectrum Acquisition***
5. ***AutoMatic Peak Identification (AutoID)***
6. ***Manually Identifying Peaks***
7. ***Spectrum Display and Manipulation***
8. ***Spectrum Export***
9. ***Four Modes of Spectrum Acquisition***
10. ***Acquiring Spectra from Lines & Grids of Beam Points***
11. ***Compare***
12. ***Quantitative Analysis***
13. ***SmartMap***
14. ***Element Maps***
15. ***Line scans***
16. ***Cameo+***
17. ***PhaseMap***
18. ***QuantMap***
19. ***AutoMate***
20. ***Montage***
21. ***Feature***
22. ***Report***

1. Optimum Microscope Conditions Setup for X-ray Microanalysis

The main purpose of this step is to enable you to set up an appropriate X-ray count rate from your sample to use for your analysis before you start collecting data into your Project.

If you are starting a new session with INCA Energy, there are a few things we recommend you optimize on your microscope, which will ensure that you get the best results for your analysis.

The main points to consider are listed below.

1.1 Are you working with tilted samples?

- If you are working with tilted samples and you are going to use the spectrum for quantitative analysis, it is important to enter the correct value of the sample tilt
- If you have purchased Microscope Control (the application for controlling and reading the electron beam and column parameters) and you have a motorized tilt stage, the current tilt angle will be automatically read by the software.
- More commonly, the stage tilt will not be motorized; in which case you will need to enter the value into Microscope Control.

1.2 What value of accelerating voltage should I use?

- Initially you need to choose a kV to get an image of your sample and then you can adjust the beam current such that you get sufficient X-rays in your spectrum. You may want to refine the kV once you have performed a more detailed analysis on your sample.
- 20kV is a good starting point, particularly if the sample is unknown. This kV will excite most X-ray lines from most elements and these will be automatically identified in the spectrum.

  Choose a lower kV if you are concerned about:

  1. Accuracy of quantification of light elements since the lower penetration into the sample will reduce the absorption correction.
  2. Analysis of a small particle, inclusion or a film less than 10mm in depth since a smaller excitation volume will enhance the contribution from these features.

- If you have purchased Microscope (column) control INCA Energy will automatically read the current microscope accelerating voltage.
- If you have not purchased Microscope (column) control and you are working in Point & ID, you will need to go to the 'Site of Interest step' in the Navigator and manually enter the current kV on your microscope into the space provided. Alternatively, select Microscope (column) control under Options from the main menu and enter the kV in the space provided.

1.3 Setting up the beam current

It is generally easiest to setup the microscope if you operate in secondary electron imaging mode.

The next stage is to produce a stable beam of electrons for X-ray microanalysis. You may have a tungsten, LaB6 emitter or a field emission gun each requiring its own type of setup procedure.

The degree of electron emission can be monitored in more than one way, depending on your microscope.

If you have a tungsten or LaB_6 gun, it is good practice to increase the filament current slowly as this will preserve the lifetime of the filament. Initially, as you increase the filament or heating current, the beam

current increases to an initial maximum, before dropping and then rising again to a point above which the beam current no longer increases. This is known as the saturation point, and any further increase in heating current just reduces the lifetime of the filament.

With the filament operating at the saturation point, emission is stabilized and this is where you should operate if you wish the beam to be stable.

You can observe the saturation point for a filament by viewing the trace variation on the CRT, or waveform monitor, which represents the signal from the sample. Increase the filament current such that you no longer produce an increase in the height of the waveform above the baseline. At this point, you have reached filament saturation.

Another common way of monitoring the emission from a gun is by observing the so called 'emission image'.

1.4 Working distance

The working distance is defined as the distance between the lower pole piece of the objective lens and the plane at which electrons are focused.

1.4.1 Which value of working distance should I use for X-ray microanalysis?

Ensure that the working distance, as displayed in millimeters on your microscope monitor, is the desired value for performing X-ray microanalysis. This is specific to the geometry of the detector mount on the SEM chamber.

The recommended value for your instrument can be viewed by going to Microscope Control from the Options menu on the INCA menu bar.

If you have purchased Microscope (column) Control, the value of the working distance displayed on your microscope monitor will be automatically read by INCA Energy. If you do not have Microscope (column) control installed, you should manually enter the working distance by selecting Microscope (column) control under Options from the main menu.

1.4.2 How do I adjust the working distance?

Adjust the strength of the objective lens by adjusting the 'focus' on your microscope until your microscope monitor displays the recommended working distance. Note that your image may now appear out of focus.

Carefully adjust the sample height using the Z drive of your stage so that the image comes into focus. Check for excessive lens hysterisis.

If you move to another area of the sample or move to another sample completely, don't adjust the focus, but adjust the Z drive of the stage to bring the image into focus. This means that X-rays are always generated at the same position within the microscope, which gives the correct geometry for the detector.

Take care that if you have a large sample, or have many samples of different heights mounted on the stage, that any sample does not collide with any detector mounted directly below the pole piece of the objective lens (e.g. backscattered electron detector).

1.4.3 How to improve image quality

By now you should see a focused image on your microscope monitor.

The quality of your image will very much depend on how well your column has been aligned. Your image quality will be optimized if your filament has been exactly centered in the gun area, and the electron beam travels exactly down the optic axis of the column before it strikes the sample. There are a variety of methods provided on microscopes to achieve these optimum conditions.

Some microscopes have a ‘Wobbler’ button to check the alignment of the objective aperture which when activated fluctuates the objective lens current about a fixed value. Alternatively you can manually increase and decrease the focus about a fixed point.

If you do this, look at your secondary electron image, it will appear to go through focus but will distort if the objective aperture is not centered. When correctly centered, the image will appear to go in and out of focus, without moving or distorting.

You may further improve the quality of the image by adjusting the stigmators on your microscope. Astigmatism manifests itself in images by causing streaking of features as you change the focus about a fixed point. You can reduce any astigmatism by adjusting the stigmators in conjunction with the focus.

If you are going to perform any X-ray analysis, make sure that you re-adjust the focus to ensure that you are operating at your recommended working distance and bring your image into focus using the Z drive of the stage.

2. Quant optimization - Why do we need to do it?

Ambient temperature changes will alter the gain of the system and this will affect where peaks appear in the spectrum.

- The microscope beam current may vary with time
- The exact peak positions, and the resolution of the system are needed to precisely identify individual peak components in the spectrum.

If peaks overlap, the relative sizes of individual peaks can only be calculated accurately if the width and position of each peak is accurately known. By measuring the position of one known peak, the system can be optimized to determine the position of all other peaks.

If we want to measure absolute concentrations, we need to make a comparison of intensity of a peak with that from a known material. If we measure a known material, we can then make accurate intensity measurements on unknowns , provided the beam current doesn't alter after the optimization.

The necessary information is automatically measured by performing a Quant Optimization, which requires only the acquisition of a high quality spectrum, from which details of beam current and spectrometer gain are measured. The frequency with which this needs to be performed is discussed below.

2.1 How to perform a Quant optimization

Quant Optimization requires acquisition of a high quality spectrum from a suitable element from which details of the beam current and spectrometer gain are calculated and stored.

If you want accurate qualitative identification of peaks and there are no substantial overlaps in the spectrum, you should only be concerned with gain optimization.

If you need more than relative concentrations and cannot work with a normalized total, you will need to be more careful in your selection of a beam current correction standard.

2.2 How often should I perform the Quant Optimization?

At least one Quant Optimization should be performed. If one has not, a message will be displayed 'the spectrum has not been optimized for quantitation. You may process the spectrum by selecting Normalization and either All elements or Element by Stoichiometry'.

No message will be displayed if the Normalized option is checked.

'No Optimization has been performed' will appear in the Status section of the quantitative results.

If more than two hours have elapsed since the last Quant Optimization was carried out, the 'Quant Optimization performed' is replaced by 'Last Quant Optimization performed more than 2 hours ago'. This message is displayed in the Acquire Spectra step.

The electronics used in INCA are carefully designed to provide good temperature stability. Since a change of 10° C produces only a 1eV shift in peak position, most routine analysis can be performed without re-optimizing peak position. However, if you need the software to resolve very closely overlapped peaks, you should perform a Quant Optimization and reoptimize if the ambient temperature changes by a few degrees. With a good laboratory temperature control you may not need to Quant Optimize for many hours.

If you wish to calculate un-normalized totals, the frequency with which you perform the Quant Optimization will depend on the stability of the beam current. Repeated measurement of a known

standard will indicate whether beam current is varying. The variation in analysis total will be in direct proportion to the change in current since the last Quant Optimization.

If you change the Spectrum range or type of detector since the last Quant Optimization was performed, a message will be displayed ‘The Spectrum range or the detector type selected is different from the one used for the last Quant Optimization’.

3. Selecting Optimum Acquisition Conditions for X-ray Acquisition

3.1 Livetime

You can chose to terminate acquisition at the end of a preset livetime. Enter the required time in seconds into the text box.

This is the time for which the system is processing counts into the spectrum. The livetime clock runs slower than the real time clock so that the acquisition for '100' live seconds takes longer than 100 real seconds. This time is extended to compensate for the output rate being less than the input rate by the degree of Deadtime.

You can choose to terminate acquistion at the end of a **Preset integral.** The number of counts you set to acquire within the upper and lower energy limits refers to the gross integral - the sum of the counts in each channel in the window.

3.2 Process time

The Process time is the length of time spent reducing noise from the X-ray signal coming from the ED detector during processing. Process times 1 to 6 are available. By selecting different Process times it is possible to reduce differing amounts of noise. The longer the Process time, the lower the noise. If noise is minimized, the resolution of the peak displayed in the spectrum is improved, in other words, the peak is narrower and it becomes easier to separate or resolve, from another peak that may be close by in energy.

3.2.1 How do Process times differ?

There is a trade off between the Process time that is used, and the speed at which data can be acquired into the X-ray spectrum. Process time 1 is the shortest, and as such, gives the highest X-ray acquisition rates, but at some cost to resolution. Process time 6 is the longest, and gives the highest resolution, but at some cost to maximum acquisition rate**.** The longer the Process time, the slower data can be acquired, i.e. the higher the system Deadtime will be for a given input count rate. (The input rate is not affected by the pulse processor).

3.2.2 Which one should I use?

Process times 1 to 6 are available, select an appropriate one from the drop down list or type in the required value. The default Process Time is 5. The selectable Process time allows optimization of the analysis conditions for the current application.

For the first look at a specimen we recommend that you use a long process time (5 or 6) to start with in order that you do not miss any detail in your spectrum. For example, when identifying peaks particularly those closely spaced and overlapping, it is important to get good peak separation. Good resolution is also important for looking at a series of lines that are very closely spaced, like an L series and process times 4 to 6 should be chosen. Common overlaps include the Mo L and the S K lines.

If there are no closely spaced peaks then you can afford to use a shorter Process time such as 1-3, which will enable you to increase the acquisition rate by increasing the beam current. A compromise between acquisition speed and resolution should be found if there are peak overlaps.

When acquiring SmartMap data you should choose your Process time carefully.

- You may have been working on a sample in either Analyzer or Point & ID where you have setup your acquisition parameters to optimize your quantitative analysis. If you now wish to acquire SmartMap data and you think you may wish to reconstruct spectra from your SmartMap data and then quantify these spectra, you should maintain these acquisition parameters. This means that

you may have to acquire data with a long Process time to maximize resolution but limit the maximum acquisition rate.

- You may have been working in either Analyzer or Point & ID and you want to view the distribution of elements whose main peaks do not overlap as a map or a linescan or you want to acquire a Cameo image. In this case you should use a shorter Process time which will mean that you can work with higher acquisition rates and shorter acquisition times. The choice of Process time will very much depend on your sample and what you wish to do with your SmartMap data once it has been acquired.

- If you have started your Project in Mapping, and you are analyzing an unknown sample, we recommend that you use a long Process time in order that you do not miss any detail in your spectrum. However if you only wish to map certain elements whose main lines do not overlap, you can afford to shorten the Process time and increase the acquisition rate by increasing the beam current.

3.3 Spectrum Energy Range

Select a spectrum energy range from the drop down list. It is recommended that a new Quant optimization is performed with the same energy range as that used on the spectrum to be quantified. If you have performed a Quant optimization with a 10keV range, the range is changed to 20keV in this set up box and a spectrum is acquired, a message box will appear to warn you to repeat the Quant Optimization with the new range.

Spectrum range keV	***Number of Channels***	***eV/channel***
0-40	*2K*	*20*
0-40	*1K*	*40*
0-20	*2K*	*10*
0-20	*1K*	*20*
0-10	*2K*	*5*
0-10	*1K*	*10*

The appropriate energy range should be selected in conjunction with the current microscope accelerating voltage. If the accelerating voltage is above 10kV, in order to view lines, which may be excited above 10keV, the 20keV range should be chosen. Below 10kV, it may be more appropriate to choose the 10keV range since no lines above 10keV will be excited.

4. Start/Stop/Resume Spectrum Acquisition

The function keys are only enabled in the Analyzer navigator.

If you are working with Windows 2000 or later, the function keys are enabled. Press F9 to start, F10 to stop and F11 to resume acquisition.

If you are working with Windows NT4, the function keys are disabled. Resume acquisition can only be accessed by pressing the shift key and clicking on the start button.

5. Automatic Peak Identification (AutoID)

The peaks in the spectrum will be identified on termination of acquisition or during acquisition. Alternatively you can manually identify the peaks in the Confirm element step of the Navigator in both Analyzer and Point & ID.

5.1 To change when peaks are automatically identified

To set this option, select Analyzer Options/Point & ID Options under Options from the menu bar depending on which navigator you are currently working with.

Select During acquisition and on termination if you wish the peaks in the spectrum to be identified and labeled during spectrum acquisition and on termination.

Select On termination if you wish the peaks to be identified and labeled on termination only.

If you do not want the peaks to be automatically identified at this stage, do not select either of these options.

Press 'Ok' when you are happy with your selection.

5.1.1 Should I do a Quant Optimization?

A comprehensive library of profiles are fitted to your spectrum during the automatic peak identification process. This process will operate at its best when a Quant Optimization has been carried out. This will result in the most accurate matching of the profiles to your spectrum.

If you have not carried out a recent Quant Optimization, nominal calibration will then be used which could result in a less reliable result, since this will only be correct to within one channel. Best matching will also occur for peaks where the statistical scatter is low i.e. there are sufficient counts in the peak.

There are a few cases in which it is extremely difficult to separate overlaps. A well known case is Al K/Br L. In such cases, a little knowledge of the sample will help. To de-select certain elements from the list of elements used for identification, select Edit Sample Types under Options from the menu bar. A list of common overlaps are:

NaK / ZnL

SK / MoL

AlK / BrL

SiK / RbL

Details about how to edit a Sample Type can be found in the section of 'Sample Type' in the Help.

6. Manually Identifying Peaks

In the Confirm Element step you may wish to identify a peak in the spectrum or confirm the absence or presence of an element. For example:

6.1 Is a certain element e.g. Copper in my sample?

- Type in either:

Full element name	Copper
Element symbol	Cu
Atomic number of the element	29
First few letters of the element	Cop (C will show carbon, Co will show cobalt, Cop will show copper)

- And press 'Return'

Markers will be displayed for all the lines for this element. K lines are in red, L lines in green and M lines in purple.

Or, select the required element from the drop down list.

6.2 What is that peak?

Click on this button  in the Confirm Elements step and position the cursor at the center of the peak. Note, you may wish to expand the spectrum by holding down the control key and dragging the spectrum with the mouse. The list of elements corresponding to the energy at the cursor is displayed in the box in the top right hand corner of the spectrum viewer. By highlighting an element in this list, you will see the markers showing all the lines for this element.

Alternatively,if you have the 'Lock to peaks' box checked, pressing  and positioning the pointer anywhere in a peak will position the cursor at the center of that peak and display elements whose peaks correspond to that position.

If you wish this element to be added to the Confirmed element list, highlight it and press 

.

Or double click on the element in the manual ID list. The peaks will be labeled and the element added to the Confirmed element list.

6.3 Peak ID Confirmation – Overlay Spectrum Reconstruction

The purpose of this tool is to be able to quickly see that all the peaks in your spectrum have been identified. If the box, 'Overlay Spectrum Reconstruction' is checked, a spectrum is displayed which is reconstructed using peaks from the elements currently listed in the Confirmed element list.

This spectrum is overlaid on your spectrum in red. If you observe that a peak in your spectrum has no corresponding peak in the reconstructed spectrum, press the manual ID button either with the lock to peaks box checked or unchecked, position the pointer on the peak, and identify the peak from the list of elements in the drop down list.

6.4 Peak Label Editor

Peak Label Editor allows you to edit and select which peaks are labeled in your spectrum.

The element displayed corresponds to that of the closest peak labeled. Alternatively you can type in either:

Full element name

Element symbol

Atomic number of the element

First few letters of the element

And press 'Return'

The list of lines displayed corresponds to those in the X-ray database.Those checked correspond to those labeled in the spectrum. If you wish to edit the selection of lines, either check or uncheck the appropriate boxes and press OK.

The energy (in keV) of the highlighted line is displayed below.

Press Restore to undo any changes made after the form was last entered.

The peak label text may still be edited if desired. Either select a label from the drop-down list or type in text into the space provided. This text will appear as the peak label in your spectrum.

Additional notes

When editing peak labels, some labels may appear in the list highlighted in red. The software matches peak labels with corresponding lines in the X-ray database. However, there are occasions when a peak label may not exactly match a line energy and it is in these instances that the label is highlighted in red.

With ED spectra this may result from improvements to the energy values stored in the X-ray database. When the spectrum was originally acquired, peak labels were placed at known positions. However with an improved energy for a particular line, the peak label will no longer match its originally designated line. The new label will be located either directly above or below the highlighted peak in the list of available lines. This may be selected in place of the highlighted line in order to use the newer energy value.

With the continuing process of improving the X-ray database, users may notice other examples of this occurring with new releases of the microanalytical suite software when editing peak labels on existing data. This will only be noticed when editing peak labels on spectra acquired on suites prior to the current revision. Any newly acquired or Auto ID'd spectra will not see these additional peak labels.

For WD Spectra, peak to background label positions are dependent on the spectrometer and its calibration. These peak labels are extra to the set of lines available and are added in as red highlighted entries.

Unlike other peak labels, the entry for the label in the list shows the actual text to be used encapsulated in ( )s. Editing the text in the lower text box will directly change the text applied to the peak label. It should be noted that once the extra labels are removed they can not be added again (and will not show up in the peak label list).

7. Spectrum Display and Manipulation

- Full Screen Display — You can display a spectrum as Full Screen.
- Solid — You can choose to display a solid spectrum.
- Line Draw — You can display a line spectrum.
- Smart Peak Label — You can choose to display either ordinary labels or smart peak labels.
- Noise Peak — You can display the Noise peak. There are three options available.
- Details — You can view spectrum acquisition details such as kV, Process time and date and time etc.
- Log Scale — You can select Log Scale to display details of small intensities.
- Vertical Scalebar — You can switch Vertical Scalebar on or off

8. Spectrum Export

- Bitmap
- TIFF
- JPEG
- Metafile
- EMSA
- ISIS

9. Four Modes of Spectrum Acquisition

In the Point & ID navigator X-ray spectra may be acquired via four methods of beam control:

Click on this if you wish to acquire a spectrum from a point.

Click on this if you wish to acquire a spectrum from a rectangular region. By dragging the mouse, a rectangular region can be outlined on the image. A spectrum will acquire from this scanned area.

Click on this if you wish to acquire a spectrum from an automatically selected region. This region will be selected according to a grey level threshold.

Holding down the mouse and clicking around the feature you wish to acquire over. X-ray data will select the region for X-ray acquisition. Double-click with the mouse when you have completed dragging around the feature and this will automatically start X-ray acquisition from this region.

10. Acquiring Spectra from Lines and Grids of Beam Points

10.1 Setting up a Line of Points for a Beam Points Run

You can draw a line at any orientation on the current image (electron image, X-ray Map or Cameo+ image).

Press the line of points icon, click on the image and drag the mouse across the image in any orientation.

Enter the number of points or point spacing in the 'X' box provided and select your desired spacing method.

Once this information has been added, INCA Energy will then calculate the co-ordinates of points for analysis on this line and add them to a beam list.

Press the green 'Go' icon to start acquisition.

10.2 Setting up a Pattern or Grid of Points

You can generate a grid or pattern of points on the current image (electron image, X-ray Map or Cameo+ image).

Press the grid of points icon, click on the image and drag the mouse across the image in such a way that you define a rectangle/square.

Enter the number of points or point spacing in the 'X' and 'Y' boxes provided and select your desired spacing method.

Once this information has been added, INCA Energy will then calculate the co-ordinates of points for analysis on this grid and add them to a beam list.

Press the green 'Go' icon to start acquisition.

10.3 Spectra Labels

You can define a name for the spectra to be acquired into your beam list from a line or grid of points by entering a spectrum label into the space provided. For example if you set the label as 'Point', the spectrum's label will be as follows:

Line spectrum: Point(1), Point(2)...

Grid Spectrum: Point(1,2), Point(2,2).....

When the spectra have been acquired, they will have this label in the spectrum drop down box.

10.4 Lines and grids from SmartMap data

It is possible to reconstruct spectra from a line or grid of points from SmartMap data such as an X-ray Map or Cameo+ image.

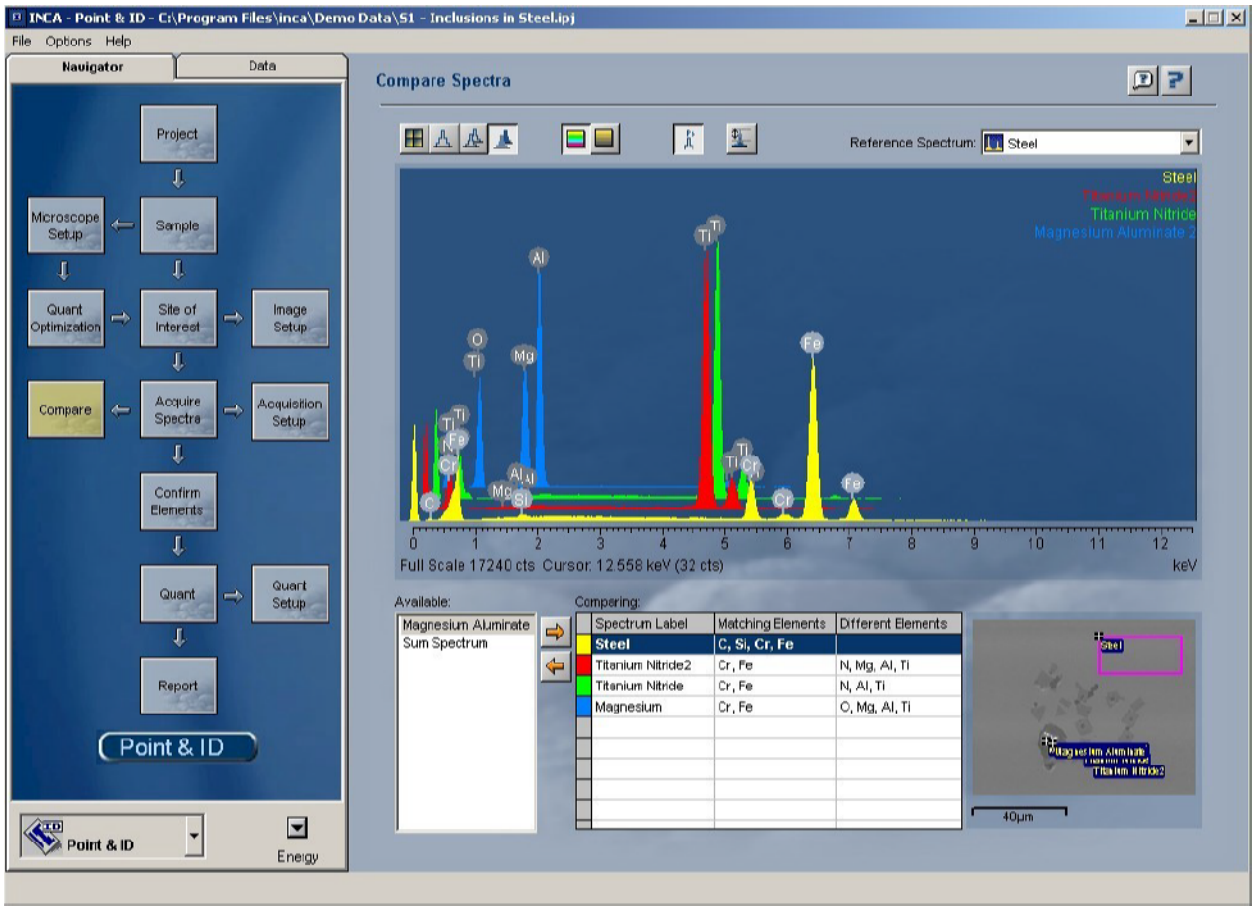
The procedure to follow is identical to that outlined above except that the spectra are reconstructed from areas rather than point acquisition. These areas are indicated by rectangles drawn on the image.

10.5 Acquisition order

When acquiring spectra using a number of different tools (e.g. point, area and feature), acquisition from a set of lines or grids will always take priority over acquisition using tools, which generate a ' To-do ' list. For example, if you setup to acquire spectra from 5 points using the point acquisition tool, acquisition will automatically commence. If you subsequently setup a grid of points and press the go button, the next point to acquire will be from the grid in preference to acquisition from the point setup using the point acquisition tool.

11. Compare

This step in both Analyzer and Point & ID allows you to compare spectra acquired from different Sites of Interest and Samples from any of the Projects currently open.



The spectra in the ‘Available' list are all those associated with the current Site of Interest. If you have acquired the spectra in Point & ID, the positions of all the spectra associated with the current Site of Interest are displayed on the image. You can add to this list of available spectra by holding down the control key and dragging spectra from any Project, Sample and Site of Interest from the Data tree into this list.

Select which spectra you wish to compare by selecting them individually from the available list, and pressing the upper arrow. This will add all the spectra you wish to compare into the ‘Comparing’ table. If you want to remove any spectra from this table, highlight the spectrum and press the lower arrow.

You can choose which of the available spectra you wish all the others to be compared to by selecting your reference spectrum from the drop down list at the top right of the work area.

In the comparison table you can view both the matching and different elements between the reference spectrum and all the other spectra being compared to it. The elements listed in the 'matching elements' list correspond to those in both the reference spectrum and all the spectra being compared to it, whereas the elements in the 'different element' list correspond to those which are in any of the spectra used for comparison but not in the reference spectrum.

11.1 Display modes for comparing spectra

You can view your spectra in a variety of ways by selecting the appropriate button at the top of the work area. You can simultaneously view all the spectra selected for comparison. They can be tiled, viewed in different colors as two dimensional lines, offset from each other, as solid lines or in shades of yellow. Spectra can be expanded and contracted, exported, copied and viewed as lines in the same manner as described in the Acquire Spectra step. You can view spectra either with the peaks labeled or unlabelled.

If you want to scale your spectra on a particular channel press the scale spectra button, place the cursor on the channel and click with the mouse. This will scale the intensities of all the spectra at the cursor position.

12. Quantitative Analysis

Quantitative analysis of elements in any sample requires an accurate measure of the peak intensities, before the concentration of elements in a sample can be calculated. In determining peak areas in spectra, two problems arise

1. A typical spectrum contains characteristic peaks, which are superimposed on a slowly varying background, which is 'noisy' because of statistical variations. This background contribution needs to be carefully subtracted from the spectrum.

2. The energy resolution of the detector imposes a limit on the separation of peaks. Identification of peaks is generally not a problem, but overlapping peaks require deconvolution, before the true peak intensities for each element can be extracted.

Once these intensities have been determined, a comparison is then made with standards of known composition, followed by application of matrix corrections, before the concentration of each element can be determined.

The simple method of linear interpolation of the background beneath a peak is not appropriate, since the background is non linear, both locally in the vicinity of peaks, and over the entire energy range. Different methods can be used to remove the background and separate the peaks from each other:

1) The method used in the INCA Energy software suppresses the background, using a filtering method which avoids any specific shape calculation. The peak intensities are then obtained by using a least squares fit of standard peaks, in which the background has also been suppressed. A symmetric, zero area 'top-hat' function is used to filter both the unknown spectrum and standard peak shapes or profiles, which are subsequently used in the peak deconvolution. The filter suppresses any background component which is linear over the width of the top-hat, and is approximately equivalent to taking the negative second derivative of the smoothed spectrum.

A profile can be considered as a fingerprint for an element. It can be either for K, L or M series lines and is calibrated both in energy and resolution. Profiles are filtered in the same way as the unknown spectrum and are subsequently fitted to the filtered spectrum using a least squares routine. The filtering and fitting technique is collectively known as Filtered Least Squares or FLS.

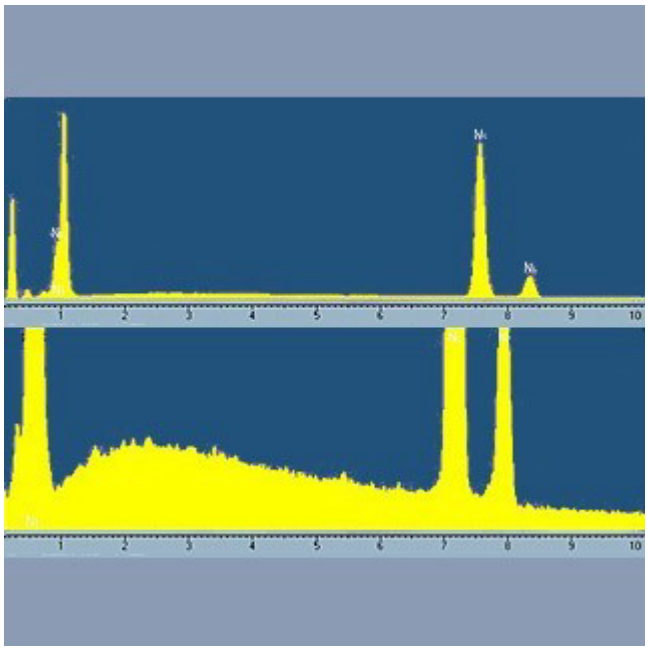
2) Another method relies on the fitting of a theoretical background to the spectrum background, and then using a least squares fitting technique to obtain peak intensities. However, construction of a model to fit the background requires an accurate knowledge of a number of physical parameters, which are often difficult to determine precisely. The strength of this method is that it attempts to model the curvature and absorption steps in the spectrum. However, systematic errors, uncertainties in the absorption correction and detector efficiency, often combine to produce a poor fit to the true background, particularly at low energies. Background modelling is only effective when the sample geometry is accurately known, and there are no spurious background contributions, otherwise the theoretical formulae will not be appropriate.

12.1 Spectrum Processing

This section describes the different events when the spectrum is processed for quantitative analysis.

12.1.1 Background

Continuum X-rays or bremsstrahlung (literally translated as braking radiation) are generated when electrons interact with matter. The emission of these X-ray photons is a result of the incident electrons decelerating in the field associated with the atoms in the interaction volume. This results in counts in the X-ray spectrum in channels between zero energy up to an energy which corresponds to the value of the incident electron beam energy.



The above figure shows the X-ray spectrum acquired from a pure Nickel sample, showing both the continuous and characteristic components of the X-ray spectrum. The most energetic X-ray that is seen in the spectrum, corresponds to an incident electron having lost its entire energy, as a result of the interaction with the field of the atom, and has an energy whose value is known as the Duane-Hunt limit. If the sample is not charging, the Duane-Hunt limit has the same value as the incident beam energy.

12.1.2 Peak deconvolution

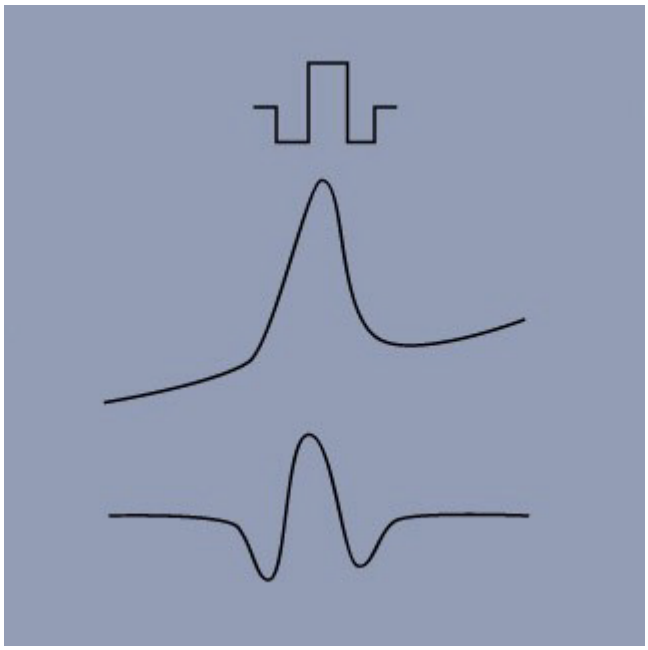
The energy resolution of EDS imposes a limit on the separation of peaks. There are several examples of peak overlap which commonly occur: the K_α peaks for Be, B, C, O, N and F, are sufficiently close for the tails of one peak to overlap into the neighbouring peak. The K_β line of one element often overlaps with the $K\alpha$ line of another. Examples include the V K_β and Cr K_α (15eV apart). Overlaps between lines from different shells e.g. between Mo $L\alpha$ and S K_α, can also occur.

When peaks overlap, it is still possible to extract individual peak areas, provided that the corresponding peak shapes are accurately known. These peak shapes or profiles are fitted to the spectrum using the method of least squares. Clearly the accuracy of the fitting depends on the similarity of the peaks in the unknown spectrum, to those of the profiles, and this is reflected in the accuracy of the peak intensities. This is particularly important when small peaks and larger peaks overlap. The fitting of profiles to the unknown spectrum requires that the gain and the system resolution should be matched between the two. Optimization of spectra, in terms of gain and resolution, can be taken into account by using an optimization standard which produces suitable known peaks. In INCA, this is done by carrying out a Quant optimization.

12.1.3 Digital filtering

A typical spectrum consists of three components: a slowly varying curved background, rapidly varying peaks and statistical noise.

The role of the top hat filter is to suppress the high frequency noise component in the spectrum and the slowly varying background, while emphasizing the characteristic peaks. Suppression is achieved by deconvolving the spectrum with a 'zero area' top-hat function. This is shown diagrammatically in the figure below. The top-hat filter is shown at the top of the figure, the central curve represents the experimental spectrum (made up of a characteristic peak which is Gaussian in form superimposed on a slowly varying background), and the curve at the bottom of the figure represents the spectrum after filtering.



In order to obtain the filtered spectrum, the filter function is calculated for every channel position in the spectrum. If the original spectrum has no curvature across the span of the filter, then the average will be zero, however any high frequency noise on the spectrum will be suppressed.

> Note that although filtering results in a severe distortion of the peak shapes, the results will not be affected provided the shapes or profiles are also subject to the same filtering before the least squares fitting is carried out. Once filtering has been applied to both the profiles and the unknown spectrum, the profiles are fitted to the sample spectrum using a least squares routine.

12.1.4 Matrix corrections

The number of X-rays generated at any one point is governed by a number of factors.

As well as the energy and number of electrons incident upon the sample the detected X-ray intensity is also dependent on several properties of the sample, the so called matrix effects. These include the efficiency of X-ray generation and the extent of absorption within the sample, the fraction of the incident electron beam which remains within the sample and is not backscattered, and the contribution to the signal from X-ray fluorescence as the X-rays traverse the sample.

Matrix corrections are employed to convert the measured intensity from the sample relative to that from a standard to the actual concentration. Standards are materials in which the concentrations of all the elements are accurately known and may be pure elements or compounds.

The uncorrected or apparent concentration of each particular element, Capp is given by the following expression:

Capp sam = Cx std (Ix sam / Ix std)

where Cx std refers to the weight percent concentration of the element in the standard, Ix sam and Ix std are the sample and standard intensities. This assumes that measurements were made from the standard and material with identical operating conditions (beam current, geometry, kV and livetime). In addition, it assumes that the detector acceptance solid angle and transmission efficiencies are identical. If any of these conditions are not met it is generally possible to apply an additional correction, k, for example to account for the difference in Livetime.

Capp will only be an approximation to elemental concentration and must be corrected for the matrix effects. Since Ix, the X-ray intensity can be described by

Ix = k Cx Fx

where Fx represents the matrix corrections in the sample. We therefore have

Ix sam = k Cx sam Fx sam

And similarly for a standard:

Ix std =k Cx std Fx std

By dividing these two equations an expression for the concentration of the sample can be extracted:

Cx sam = (Ix sam / Ix std * Cx std) * Fx std / Fx sam

Since the term in brackets is our first approximation to the composition, Capp, we have

Cx sam = Capp sam * Fx std / Fx sam

12.1.5 Absorption correction

Not all the X-rays generated, as described by the $\varphi(\rho z)$ curve (see online help), will be detected by the X-ray detector due to absorption of the X-rays as they traverse the sample. Therefore if we describe the measured intensity as I and the generated intensity at a particular depth z as I_0 then the relationship between the two is described by the following expression

$I = I_0 \exp(-\mu\rho t)$

where μ is the mass absorption coefficient, ρ is the density and t is the path length the X-rays have to traverse before they can exit the surface.

The value of the mass absorption coefficient is a function of the energy of the X-rays and the composition of the material that the X-rays have to traverse. Therefore, the degree of absorption will be different for different X-ray lines. X-ray absorption is generally the most significant effect which must be accounted for in quantitative analysis.

The absorption correction can be calculated using knowledge of the depth distribution of the generated X-rays from the $\varphi(\rho z)$ curve combined with the path length to the detector for each incremental depth. The path length at a depth z is given by z cosec(ψ)

where ψ is the take off angle. The overall absorption correction is commonly referred to as $f(\chi)$ written as

$$f(\chi) = \int \varphi(\rho z) \exp(-\chi\rho z)\, d(\rho z) / \int \varphi(\rho z)\, d(\rho z)$$

where $\chi = \mu$ cosec(ψ). The denominator in the above represents the area under the $\varphi(\rho z)$ curve which is equivalent to the total number of X-rays produced and serves to normalize the expression.

The above correction formula assumes that this material is of the same composition as the material in which the X-rays are produced. The amount of extra material though which the X-rays pass outside the electron interaction volume will depend on the take off angle of the detector.

12.1.6 XPP correction method

A number of different approaches to matrix correction procedures are present in the literature with ZAF and Phi-Rho-Z being the two most widespread. INCA Energy uses the XPP matrix correction scheme developed by Pouchou and Pichoir. This is a Phi-Rho-Z approach which uses exponentials to describe

the shape of the $\varphi(\rho z)$ curve. The atomic number and absorption effects are included by integrating a model for $\varphi(\rho z)$. XPP has been chosen as the correction scheme because of its favourable performance in particular for situations of severe absorption such as the analysis of light elements in a heavy matrix. In addition, XPP enables the calculation of the matrix correction terms and therefore quantitative analysis for samples that are tilted with respect to the incident electron beam.

Since the correction factors are dependent on the composition of the sample (which is what we are trying to determine), the true concentrations have to be derived using an iterative procedure. The apparent concentrations are used to calculate correction factors which are then used to derive more precise 'estimates' for the concentrations and so on until after successive iterations, a self-consistent set of concentrations and correction factors are obtained.

12.2 What Happens When You Press 'Quant'?

After spectrum acquisition when you go to 'Quant' step, the quant results are calculated and displayed straight away. It is important to setup the system correctly before you proceed to perform accurate quantitative analysis. You have to decide about the geometry, kV, process time and quant optimization etc.

12.2.1 Basic quantitative analysis

When you press Quant:

1) Using the strobe zero the sample spectrum is corrected for zero drift.

2) Using the Quant optimization data the sample spectrum is corrected for

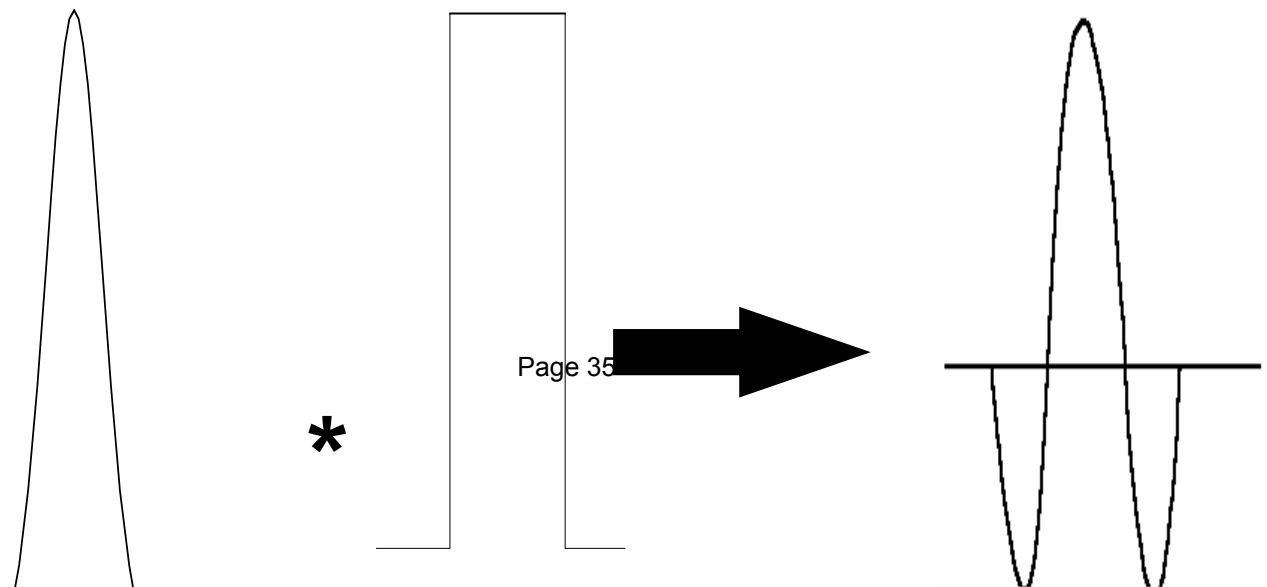
a) small, short term gain change

b) beam current

3) The resolutions of the sample spectrum and all the standard profiles (peak shapes) are compared and the worst one found; the sample spectrum and all of the profiles, including the worst one, are then broadened to the same resolution (4 eV more than the worst one).

4) Escape peaks are removed and restored to their parent peaks in the sample spectrum. Refer to online help for details of Escape peaks.

5) The background contribution is removed from the sample spectrum and standard profiles using a digital top hat filter.



6) The filtered standard profiles are fitted to the sample spectrum using a least squares routine and the peaks deconvolved from the spectrum. Due to the energy resolution of an EDS spectra there is a limit on the separation of peaks which overlap e.g. between Mo L_α and S K_α.

7) The apparent concentration is calculated directly from sample to standard intensity ratios determined by least squares fitting of background filtered (removed) standard profiles to the sample spectrum.

8) The fit index for a profile is a figure of merit indicating the magnitude of systematic errors in the fit in relation to random statistical errors. The systematic errors may be due to errors in the profiles or spectrometer drift. A lower fit index value indicates a better fit between the spectrum and the standard peak shape. For **INCA** if the total number of counts in the spectrum is around 250,000 then a fit index between 5 and 20 is considered acceptable. The spectrum reconstruction overlay should be used to ensure that there are no unidentified peaks in the spectrum and hence the element identification is correct. If the number of counts in a peak is high then the fit index may be higher, reflecting the dependence of the systematic error on the number of counts. If profile optimisation has been used the fit index may be used to verify that the optimisation has been successful.

9) The apparent concentration is put into the XPP correction routine:

- The Apparent concentration is used to calculate a first estimate of the XPP correction.
- The first estimate of XPP correction is applied to the apparent concentration to give a first estimate of element weight%.
- The first estimate of the element weight% is used to calculate a second estimate of XPP correction.
- The second estimate of XPP correction is applied to the apparent concentration to give a second estimate of element weight%.
- This iterative routine continues until successive estimates in weight % differ by less than 0.1%.

10) The results are now displayed. They also contain diagnostic information to help you to assess the quality of the result such as Fit Index and omitted peaks.

You can view the results in a variety of ways using the tabs provided in the Quant step.

12.2.2 Setups

You can change the setup parameters in the Quant Setup step before you proceed to perform quantitative analysis.

12.2.2.1 Quant Configuration

This step allows you to select alternative X-ray lines to use for your quantitative analysis.

The automatic choice of X-ray line for quantitation is based on overvoltage. For a given element, if the K line exists in the current energy range of the spectrum and Ec (the energy required to excite the particular line) for the K line is less than Eo (the energy of the incident electrons) and the overvoltage U (U=Eo/Ec) is greater than a certain value, the K line is used. If not, the L line is considered and then the M line.

Depending on the other elements in your sample, an X-ray line that has been automatically chosen may overlap with another X-ray line. In this case you may wish to use an alternative line.

To choose an alternative X-ray line for a given element, either highlight an element in the Element list or select an element from the drop down list and then select the line for quantitation. Make sure ‘Automatic selection of all ED X-ray lines’ is de-selected.

Note that all changes to the selection of X-ray lines will be automatically saved when you exit from this form. These changes will be saved under the current setup in Quant setup.

12.2.2.2 Default X-ray Lines

If you have chosen to work with a Fixed Element List and you check the 'Show selected elements only' box in the 'Select Lines' step, the elements displayed will be those displayed in the element list in Quant setup.

Pressing the 'Defaults' button will restore the default choice of X-ray lines to all elements whether they have been selected or not. These defaults are based on an accelerating voltage of 20kV and a 20keV energy range. From Boron to Gallium, the K line is used, from Germanium to Lutetium, the L line is used and from Hafnium to Uranium, the M line is used.

12.2.2.3 Automatic selection of X-ray Lines

Checking the 'Automatic selection' box will ensure that appropriate X-ray lines for elements are applied to all spectra regardless of their energy range and the kV they were acquired with. For example you may be quantifying spectra which were acquired with different energy ranges and kVs within the same Site of Interest. Appropriate X-ray lines will be assigned based on overvoltages for these different spectra.

The automatic choice of X-ray line for quantitation is based on overvoltage. For a given element, if the K line exists in the current energy range of the spectrum and Ec (the energy required to excite the particular line) for the K line is less than Eo (the energy of the incident electrons) and the overvoltage U (U=Eo/Ec) is greater than a certain value, the K line is used. If not, the L line is considered and then the M line.

12.2.2.4 All spectra tab

Note that the results in the All spectra tab will reflect the type of element list selected.

12.2.2.5 Which X-ray lines are used?

The X-ray lines associated with any elements selected for quantitation will be those last specified in the 'Select lines' step under Configure. As you exit either of these steps, the selections you have last made for X-ray lines will be used during your analysis unless you change this selection. This can be achieved by going into the individual steps or alternatively by selecting a different setup from the drop down list which may contain different selections for X-ray lines.

12.3 Confirm Element List

If the peaks in the spectrum have been automatically identified in the previous stage of the flow, the list of probable elements will be automatically displayed in the Confirm element list. The list of elements displayed here will be the ones used for quantitation. The elements that you may have selected for deconvolution only such as coating elements will not be included in the quant results.

You may manually add and remove elements from this list.

Clicking on one of the elements in the confirmed element list puts up the markers for that element. Lines series are colour coded: K series lines are marked in red, L series lines are marked in green and M series lines are marked in purple.

12.4 Element List For Quant

You can select a different type of element list depending on how you want your spectra/spectrum to be quantified:

12.4.1 Current spectrum

If this option is selected, each spectrum will be quantified using the elements confirmed in the Confirm Elements step for that spectrum.

12.4.2 Combined

Select the combined element list if you wish the ‘All Spectra' tab in the Quant step to display average elemental concentrations from the spectra within a Site of interest together with maximum and minimum. If this option is selected, an element list will be built from the confirmed elements of each spectrum within the Site of Interest. All of the spectra will then be analyzed using this new element list and the processing option currently selected. Note that the current spectrum will also be analyzed using this combined element list.

As an example, consider the case where we are analyzing a stainless steel and wish to gather statistical information about its homogeneity. If we collect a number of spectra and use the Confirm Elements step to identify elements within the sample we can then process the sample as All Elements with a combined element list.

Therefore if Spectrum 1 has the confirmed elements Fe, Cr and Ni, Spectrum 2 has Fe, Cr and Ni and Spectrum 3 has Fe, Cr, Ni and Mo the combined element list will have Fe, Cr, Ni and Mo and the All Spectra tab will show the results of the analysis of these three spectra along with a mean, maximum and minimum for each of the elements.

12.4.3 Fixed list

Select this option if you wish to define a list of elements with which to quantify your spectra. For example you may only wish to quantify your spectra using certain elements if you are regularly quantifying similar spectra. You can either create this list of elements within Quant Setup step by selecting elements from the element drop down list or you can transfer the current element list in Confirm Elements step by pressing 'Transfer' in the Quant Setup step. Once this list has been transferred it can be modified.

Pressing Save on this form will save your list of elements under the currently selected setup name. If you wish to save the selection of elements under a new name, type in a new name and then press Save.

12.5 Processing Options

To make the correct selection, a little knowledge of the sample is required. For example, can all elements in the sample be detected and analyzed using your INCA Energy system, or are you analyzing a mineral where it is more usual to calculate the oxygen present?

12.5.1 All elements

This is used when processing spectra from samples in which all elements yield X-rays, which can be detected e.g. steels, alloys and other materials with insignificant amounts of elements lighter than sodium.

12.5.2 Element by Difference

This option can be used if you can analyze all elements except one in the sample. The omitted element is called the combined element. The concentration of the element selected as Combined element is not measured, but is calculated assuming that the difference between the analyzed total and 100% is due only to the presence of this element. Intensity corrections are calculated assuming the presence of this element. The total from this type of analysis is always 100%.

This can be used when analyzing a sample in which a significant quantity of a light element which cannot be detected is known to be present. It can also be used in cases where an element for which no standard is available is present.

12.5.3 Element by Stoichiometry

Use this option if you want the concentration of one of the elements to be calculated assuming that it is bound by predefined stoichiometry to all the other analyzable elements.

When analyzing mineral samples, oxygen may be calculated in this manner, i.e. the corrected weight % of measurable elements is determined and the compound % for each of these elements is calculated. Generally, intensity corrections are calculated assuming that all the analyzed elements are bound to the combined element.

There are some cases where a measured element cannot be combined with the unanalyzed element, (e.g. Fluorine in a mineral). In such cases, the % compound is not calculated for those elements. Some unanalyzed element may be present in a form not bound to measured elements, e.g. water of crystallization etc., when it might be better to analyze oxygen by difference although in this case, the compound % and formula will not be calculated.

12.5.4 Normalize results

When this is selected the analytical total of an analysis carried out using All elements or Combined element by Stoichiometry is forced to 100%.

Use of this needs care in interpreting the final result since if, for example, the element list is incomplete, there will be serious errors in the result, even though the total is 100%.

This is often used as an expedient where the beam current is unstable or the sample is unpolished.

12.5.5 Saving Setups

Setups can be selected from the drop down list at the bottom of Quant Setup window. You can select alternative setups from the drop down list. A number of settings can be saved under this setup name such as the processing option, fixed element list and the k Factors (in the case of EnergyTEM) and X-ray lines last selected when the Select lines/k Factor steps were exited. If you wish to create a new setup, enter a new name in the entry box and the settings last chosen will be saved under this name.

Recommended way of working

1. Create a new setup
2. Select processing option
3. Select Element List
4. Select the X-ray lines step

If 'Automatic selection of All X-ray lines' is not selected either restore defaults or use the currently selected X-ray line for quantitation. Note that in the case of EnergyTEM, clicking on an element will show what type of k Factor will be used in the calculation.

12.6 How to improve the accuracy of quant results

You can improve the accuracy of your quantitative results by:

- Using your own standards **Standardize**

- Customizing the spectrum processing **Profile Optimization**
- Combined ED/WD analysis **Energy+**

12.6.1 Standardize

In Energy, quantitative analysis can be carried out without the need to measure standard materials since your Energy system is supplied with a complete list of default standardizations.

However, in specific cases, using your own standards will lead to an improvement in your quantitative results. In the Analyzer navigator the Standardize step is available to enable the user to set up their own standards for quantification.. If you use your INCA Energy system for the first time and click on the standardize step, a standards database will be created. At this stage the database will contain only the default standardizations. Once you have added your own standardized entries, the standards database will be modified accordingly.

12.6.1.1 What does standardization do?

In order to make a direct comparison between intensity and concentration, a standard sample is referred to in which the relationship between Istd and Cstd is accurately known where Istd is the intensity from the standard and Cstd is the concentration of the standard . Once this is known, this ratio can be used to determine the concentration of that element in an unknown sample (Csam) since the the intensity of the element in the sample (Isam) can be measured. Let Cxsam be the concentration of element X in the unknown sample and Ixsam be the intensity of the relevent peak from element X in the unknown sample. All intensities are assumed to have been corrected for background.

The concentration of element X in the unknown sample can be approximated as:

$$Cxsam=\{Cxstd(Ixsam/Ixstd)\}$$

and is often referred to as the 'Apparent concentration' or the 'uncorrected concentration'. Once these Apparent concentrations have been determined, the element weight percents are then calculated by applying a matrix correction to the measured intensity ratios. These corrections attempt to account and correct for the side effects of X-rays traversing the sample matrix such as absorption of X-rays in the material.

The ratio in value between the Apparent concentration and the true concentration is a measure of the matrix corrections which need to be included in the calculation. However, the need for the correction is minimized if the composition of the standard sample is as similar as possible to the composition of the unknown sample. This simply means that the effect on the X-ray intensity of X-rays traversing the sample and standard is similar. This only accounts for the absorption. There are also backscatter and fluorescence effects to be considered. Since these matrix corrections can be calculated with only a certain degree of accuracy, the choice of standard material is very important if the quantification is to be as accurate as possible.

12.6.1.2 When to standardize

The need to standardize depends very much on the level of accuracy you require from your analysis on any one material. As a rule of thumb:

If you require accuracy such that the relative errors are less than 2%, you should standardize.

If you are quantifying elements whose X-ray lines are in the low energy region of the spectrum (which may be the case if you are using an accelerating voltage of less than 15kV, or the element you are quantifying has an atomic number less than 11), standardization will improve your quantitative results.

If the matrix corrections are high such as would be in the case of quantifying Al in a Pd matrix, (e.g. light element in heavy matrix or vice-versa), where your intensity correction Ic is either >1.25 or < 0.8, you should standardize. Note: The intensity correction is given in the quantitative results.

12.6.2 Profile Optimization

This step allows you to improve accuracy of your quantitative results by customizing the spectrum processing for your particular system.

This is particularly beneficial when there are extreme overlap situations e.g. AlK/BrL, SiK/WM/TaM. In addition, profile optimization can also minimize the effects of chemical shifts on your quantitative results.

In order to correct for peak overlap and background effects, INCA Energy has to calculate the peak shape for all element lines present in the spectrum. An empirical model is used to synthesize peak shapes, that has been matched to the characteristics of the detector and pulse processor provided with your INCA Energy system. However peak shape is influenced by detector to detector variations and chemical shifts. These effects can be minimized by profile optimization.

Synthesised profiles from INCA Energy may also be optimized. which will help to refine the profile shape and position to match more closely the peaks acquired on your system.

You should ensure that any spectrum you use to optimize a synthesized profile has a sufficient counts in the energy range you require, since the number of counts will affect the overall peak shape. This is particularly important when using spectra acquired on a TEM. Ideally, the element peak series to be optimized should not be overlapped by neighbouring peaks. However, if an overlap is unavoidable, a correction will be made if the elements with potentially interfering peaks (within 600eV of any peaks) are included in the element list.

To minimize the effects of chemical shifts, the spectrum you use for optimization should be collected from a material with elements in similar state to your intended sample.

Note that:

- Optimized profiles will be used in both the spectrum processing for quant and the Auto-ID.
- The Profile optimization process functions in both INCA Energy TEM and INCA Energy.
- Profile optimization works for different energy ranges and number of channels in the spectrum.
- If you wish to undo a particular profile optimization, press the Undo button in the Profile Optimization step.

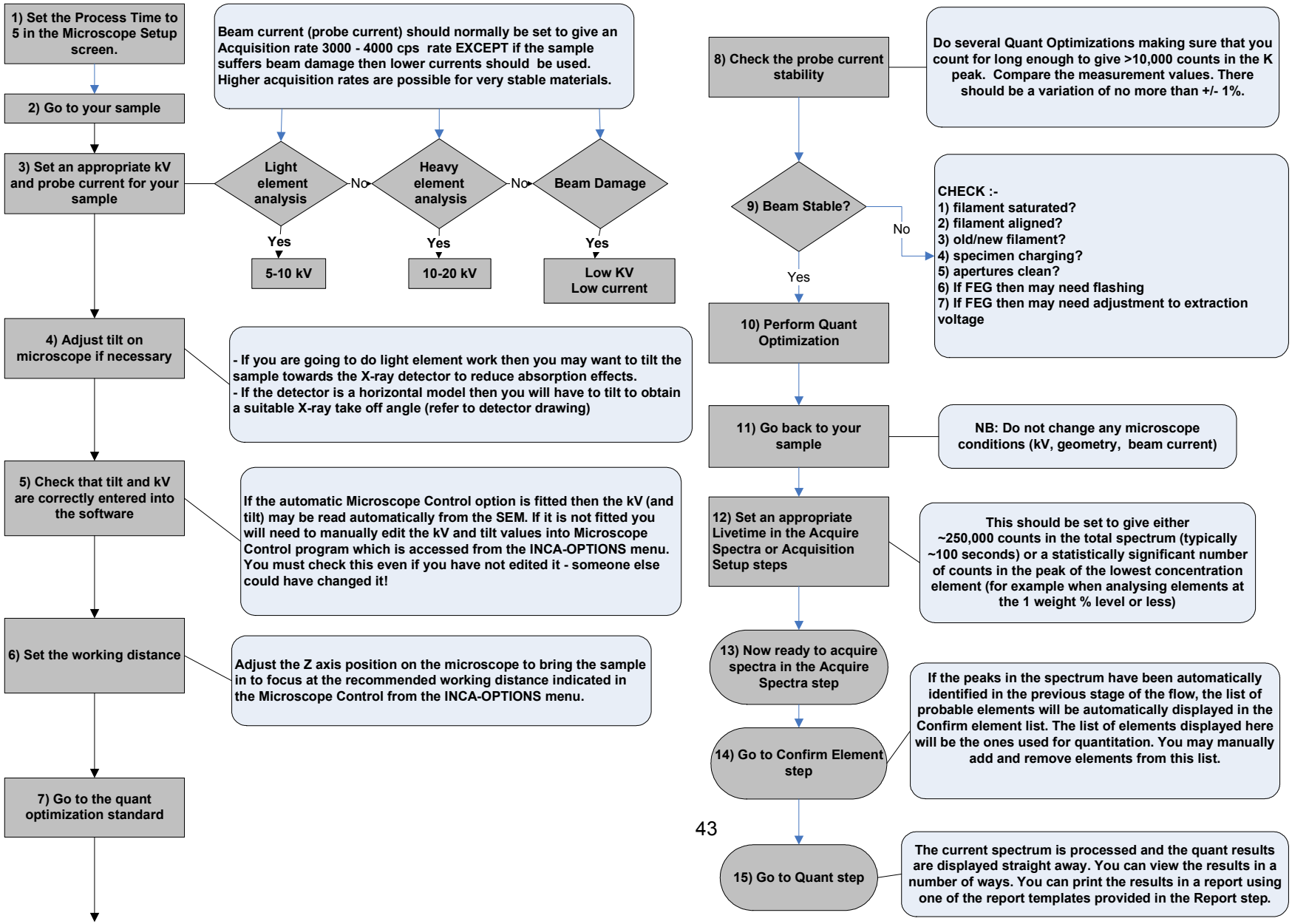
12.6.3 Energy+

As part of the Confirm Elements step you can enhance the spectrum resolution and sensitivity with a WD spectrometer scan across an appropriate energy range(s). The combined ED/WD spectrum is saved in the Data tree with a different icon to distinguish it from ED spectra.

12.6.3.1 When should you use Energy+?

- When you see inconsistencies in the fit using 'Overlay spectrum reconstruction' use Energy+ to confirm that a peak in the ED spectrum contains the contribution of another element.
- When you suspect that the sample contains particular elements that are present at trace levels or below the minimum detectable limit for ED spectroscopy (~2,000 ppm / 0.2 weight %)
- When the sample is an unknown material that may contain elements below the detection limit for ED spectroscopy (typically less than ~1 weight %)

12.7 Quantitative Analysis Flowchart


1) Set the Process Time to 5 in the Microscope Setup screen.
2) Go to your sample
3) Set an appropriate kV and probe current for your sample
4) Adjust tilt on microscope if necessary
5) Check that tilt and kV are correctly entered into the software
6) Set the working distance
7) Go to the quant optimization standard
Beam current (probe current) should normally be set to give an Acquisition rate 3000 - 4000 cps rate EXCEPT if the sample suffers beam damage then lower currents should be used. Higher acquisition rates are possible for very stable materials.
Light element analysis
No
Heavy element analysis
No
Beam Damage
Yes
5-10 kV
Yes
10-20 kV
Yes
Low KV
Low current
- If you are going to do light element work then you may want to tilt the sample towards the X-ray detector to reduce absorption effects.
- If the detector is a horizontal model then you will have to tilt to obtain a suitable X-ray take off angle (refer to detector drawing)
If the automatic Microscope Control option is fitted then the kV (and tilt) may be read automatically from the SEM. If it is not fitted you will need to manually edit the kV and tilt values into Microscope Control program which is accessed from the INCA-OPTIONS menu. You must check this even if you have not edited it - someone else could have changed it!
Adjust the Z axis position on the microscope to bring the sample in to focus at the recommended working distance indicated in the Microscope Control from the INCA-OPTIONS menu.
8) Check the probe current stability
Do several Quant Optimizations making sure that you count for long enough to give >10,000 counts in the K peak. Compare the measurement values. There should be a variation of no more than +/- 1%.
9) Beam Stable?
No
CHECK :-
1) filament saturated?
2) filament aligned?
3) old/new filament?
4) specimen charging?
5) apertures clean?
6) If FEG then may need flashing
7) If FEG then may need adjustment to extraction voltage
Yes
10) Perform Quant Optimization
11) Go back to your sample
NB: Do not change any microscope conditions (kV, geometry, beam current)
12) Set an appropriate Livetime in the Acquire Spectra or Acquisition Setup steps
This should be set to give either ~250,000 counts in the total spectrum (typically ~100 seconds) or a statistically significant number of counts in the peak of the lowest concentration element (for example when analysing elements at the 1 weight % level or less)
13) Now ready to acquire spectra in the Acquire Spectra step
14) Go to Confirm Element step
If the peaks in the spectrum have been automatically identified in the previous stage of the flow, the list of probable elements will be automatically displayed in the Confirm element list. The list of elements displayed here will be the ones used for quantitation. You may manually add and remove elements from this list.
15) Go to Quant step
The current spectrum is processed and the quant results are displayed straight away. You can view the results in a number of ways. You can print the results in a report using one of the report templates provided in the Report step.

13. SmartMap

SmartMap performs the simultaneous acquisition of X-ray data for all possible elements from each pixel on a user defined area of an image.

The main advantage of analyzing samples in this way is that minimal knowledge of the sample composition is required prior to analysis since all of the X-ray data is collected. This allows a high degree of flexibility in subsequent reprocessing.

SmartMap data can be viewed and processed during or after acquisition in a number of ways including spectra from points or areas which can be quantified, elemental X-ray maps, elemental line profiles or linescans and Cameo+ data.

For example, if you wish to view the distribution of certain elements over a particular area, you simply select the elements that you want to view map data from. These maps will be automatically updated as acquisition proceeds or on termination. You can also change the selection of elements on-line whilst acquisition is in progress. Alternatively, SmartMap data can be visited and processed at a later date.

- You can set the resolution of your SmartMap by choosing the number of pixels in the X and Y dimension over which the beam scans. The actual dimensions will depend on the aspect ratio of your microscope monitor. For a square monitor, the options are 128 x 128, 256 x 256 or 512 x 512 pixels.

If you are collecting X-ray data from the entire area shown in the image using the full field tool  the number of pixels used will be the number set in the SmartMap resolution option.

If you have defined an area using the rectangular area tool  X-ray data will be collected from only the defined area with a proportional number of pixels.

13.1 Choosing an Appropriate SmartMap Resolution

When selecting SmartMap resolution, you should consider the relative size of the X-ray generation volume, the features of interest and the image pixel size. This is particularly important at high magnifications where it may be possible to see features in the secondary electron image which are smaller than the X-ray generation volume and therefore would be difficult to see in the corresponding X-ray map. In this case, no additional information will be obtained by acquiring at high resolution.

Since the dwell time is fixed per pixel, the 'frame' time for higher resolution maps is longer than for lower resolution maps. This means a longer map refresh time.

It is important to consider the total number of counts you expect in your SmartMap and choose the resolution accordingly. For example if you wish to acquire for a long time or with a high count rate, then you should choose either 128 or 256 resolution.

13.2 Choosing an Appropriate Process time for SmartMap Acquisition

Refer to 'Process time' - item 3.2 under Section 3, 'Selecting X-ray Acquisition Conditions'.

13.3 How long should I Acquire My SmartMap for?

A) If you are primarily interested in mapping then:

While acquiring, view the X-ray maps and scatter diagrams for the elements of interest at your chosen, or reduced, resolution.

This will enable you to decide when you have sufficient contrast to discern the regions or phases of interest, or how much longer you must acquire to obtain the same level of contrast at a higher resolution.

B) If you are interested in reconstructing spectra from features:

While acquiring, view the reconstructed spectra from the features of interest in the Element Setup step.

Look at the maximum number of counts in each spectrum and ensure you have sufficient counts to perform quantification.

13.4 The Advantages of Viewing Maps at Lower Resolutions

The number of counts per pixel will increase if you view your maps at a lower resolution. This will improve image contrast and the separation of clusters within the PhaseMap scatter diagram.

X-ray maps can be viewed at lower resolution using the reduce resolution selection in the element maps step of the Mapping navigator.

Cameo images can also be smoothed to reduce color noise and improve cluster separation in the PhaseMap scatter diagram.

If you view a map at half the resolution you can immediately see how much better the contrast and signal-to-noise would be if you acquired for four times longer.

13.4.1 Why choose 512?

The advantage of acquiring a SmartMap with 512 resolution is that by using the highest number of sampling points, the maximum spatial definition will be achieved. Therefore, if you are mapping an unknown sample, selecting 512 will ensure that you don't miss any features.

However you should be aware that if you choose 512 resolution and either use a high count rate or acquire for a long time, the memory allocation of your INCA Project file may become quite large.

13.4.2 Why choose 256 or 128?

Choose 256 or 128 resolution if you expect the total number of counts in your SmartMap to be large.

If your interaction volume is large compared to the image pixel size then choose 256 or 128 resolution. These options will also give faster frame rates.

13.5 Choosing an Appropriate Linescan Resolution

The Linescan resolution simply defines the number of sampling points along your chosen line. You should take into account similar considerations such as the size of the interaction volume.

13.6 Modes of SmartMap Acquisition

Click on  if you wish to acquire SmartMap data from a rectangular region. By dragging the mouse, a rectangular region can be outlined on the image. SmartMap data will acquire from this scanned area.

Click on  and then press Start if you wish to scan over the full field of view.

To define a line, click on  , then define the starting point of the line on the image. Drag the mouse with the left button held down until you reach the point at which you wish the line to end.

13.7 SmartMap Acquisition Status

Memory

The status bar gives an indication of how much of the available memory is in use. If the indicator goes into the red, we recommend you to stop the acquisition and save your Project. Acquisition will stop automatically if the indicator goes to the end of the bar.

Time

This displays the time, in seconds, elapsed since the start of acquisition.

Counts

This shows the total number of counts in the spectrum.

Frames

This shows the number of times the beam has scanned over the selected region.

14. Element Maps

The distribution of elements over a particular area of the sample can be viewed using Element Maps. Element Mapping utilizes the X-ray signal from a specified energy range in order to show the elemental distribution.

Qualitative mapping gives information on the X-ray intensity distribution over the selected region of the sample. The gray scale value for a given pixel in any digital map corresponds simply to the number of X-rays which enter the X-ray detector within the energy range and, therefore, gives an idea of the distribution of the elements.

If you have entered this stage directly from SmartMap, the element maps displayed correspond to those elements automatically identified in the sum spectrum from the region over which SmartMap data was/ or is being acquired.

The size of the displayed maps depends on the number displayed.

The secondary or backscattered electron image of the 'Site of Interest' is also displayed.

If you have changed the element selection in the Element Setup stage, the selection of maps currently displayed will reflect this.

14.1 Subtracting Element maps

You can subtract one image from the current group. It is often useful to subtract an X-ray map collected from a background window from an element X-ray map. To access the window for subtracting maps:

- Click on the image you wish to subtract and drag it to the image from which you wish to subtract it. (You need not align the two images accurately).

Note that the widths of the energy windows used to generate the two images you are using for subtraction should be the same. You can modify the widths of X-ray maps and setup new windows for backgrounds in the Element setup step. In addition you can view details of all the energy windows for the currently selected elements and associated backgrounds.

- Select the scaling factor you wish to apply to the image you are subtracting.
- Press Subtract. The first image is then subtracted from the second.
- The processed map is now denoted by a star (*) in the map title.

To restore the subtracted image to its original state, exit the element map step and re-enter it again. All images are reset to their original state.

14.2 Mixing Maps

Pressing the 'Mix' button in the Element Maps step allows you to mix up to three currently selected maps using red, green and blue separately for each map.

Before mixing the maps, you might find it useful to threshold each of the images separately so that edges around an object or boundary appear sharper. Adjust the contrast and brightness of the images individually or together as a selected group.

Select upto three images you wish to mix and press 'Mix'. The first image is then mixed with the second and/or third image. Possible color combinations, assuming that the 3 maps are from 3 different elements, are:

Elements	1	2	3	1&2	1&3	2&3	1,2&3
Red	x						
Green		x					
Yellow				x			
Blue			x				
Cyan						x	
Magenta					x		
White							x

15. LineScan

You may examine the distribution of elements along any line over which SmartMap data is being or has been acquired. Element Linescanning utilizes the X-ray signal from a specified energy range in order to show the elements distribution.

15.1 Which Linescans are displayed?

If you have just acquired or are acquiring SmartMap data, then the selection of element linescans displayed will automatically correspond to the AutoID of the Sum Spectrum along the line.

You can change the selection of viewed linescans by going to the Element Setup step in the analytical flow.

15.2 Display of Linescans

The window to the top left of the work area allows you to view your selection of linescans superimposed on the electron image. This image corresponds to that used to setup the SmartMap data acquisition along a line.

The window to the bottom right of the work area contains all the linescans. You can view these in a number of ways: stacked, as black patterned lines or as colored lines. Windows can be zoomed and viewed in a variety of ways in your report.

15.3 Linescan scaling

You can adjust the vertical scaling of individual linescans; the default setting is auto scaling. Using the right hand mouse button, click on the linescan to view the context sensitive menu from which you can reset the scaling.

15.4 Export facilities

Various functions such as changing the contrast and brightness, exporting and copying the image and zooming in and out of the image can be accessed by clicking with the right mouse button.

You can export Linescans in a variety of formats. Select the required format from the selection displayed in the context sensitive menu accessed by clicking with the right mouse button.

15.5 Selecting Lines for Mapping/Linescanning

It is possible to change the default X-ray line used for reconstructing maps/linescans from your SmartMap data for any given element. In order to do this press Configure in the Element Setup step. A window will appear which allows you to select alternative X-ray lines for mapping/linescanning.

The elements you wish to show maps and linescans for are highlighted in yellow on the periodic table. If you wish to change this selection, double click on the element symbol or click on the symbol and press select. If you check the Auto line selection box, X-ray lines will be automatically associated with the elements depending on the accelerating voltage and energy range currently being used.

Note that if there are any overlaps between any of the peaks associated with the lines automatically selected, you will be alerted to this in the window above the Auto line selection box. You may choose an alternative X-ray line for mapping as follows:

Click an element symbol in the periodic table and select the X-ray line you wish to use from the drop down list. To aid you in the choice of X-ray line for a particular element that may be overlapping with another element in your sample, a chart that displays elements against X-ray line energies is available in on-line Help. You may find it easier to print this chart. Once you have chosen an alternative X-ray line for a particular element, use the slide rule to check that it does not overlap with any other lines associated with other elements in your sample.

You can save a selection of elements and their associated X-ray lines by entering a name in the text entry box and pressing Save.

16. Cameo+

Cameo+ allows you to see the chemical composition and topography in one image. The fine detail of the electron image is shown together with a full color overlay showing variation in the X-ray spectrum, which can indicate compositional changes. Individual X-ray photons are assigned a color, which depends on their energy. A color range for Cameo+ is selected by entering a value for the lowest energy which defines where the red part of the color scale starts, and the higher energy which defines the end of the blue part of the scale. Some experimentation may be necessary to find the range which emphasizes the compositional variation in your samples. The effect of changing the color range on the image is shown interactively in the section on Cameo+ color range of the on-line Help.

- You can adjust the lower energy end of the color scale by typing the value in the minimum box or by using the upper slider bar. Similarly, you can adjust the upper end of the color scale by typing the value in the maximum box or by using the lower slider bar. You can also double click with the mouse on the spectrum itself. This will change the energy limits directly on the spectrum.

- The corresponding energy range on your spectrum is highlighted automatically in bright yellow.

- Press **Refresh** to update the Cameo+ image for this energy range.

- The Cameo+ image can be viewed without the underlying electron image by clearing the Background box.

- The brightness and contrast of the underlying electron image can be adjusted by pressing the Brightness/Contrast button.

17. PhaseMap

Some samples have quite complicated compositional variations. Applications such as Cameo+ and Element mapping aid the user to locate regions of similar composition over an area of the sample. However, with mapping applications, this may entail simultaneously viewing a number of elemental maps making it awkward to identify areas of similar composition.

PhaseMap makes the location of areas of similar composition easier by allowing a number of 'phases' to be visualized using a scatter plot. A phase is a region of material containing a defined ratio of elements throughout. For example, a sample can contain both elements A and B. This can be segregated into different phases with different compositions of A and B. Therefore element maps of A and B will show different intensities where the phase is different. Furthermore, the spectrum emitted by the sample will be different for different phases, which will result in the Cameo+ color being different for each phase. Either Cameo+ or elemental X-ray map data can be used to provide PhaseMap data.

For Cameo+, the scatter plot shows the differences in spectral content over all regions of the field. PhaseMap gives useful information about the spatial distribution of distinct phases and their relative areas.

17.1 Source Data for Scatter Plot

Both Cameo+ and Element Map data can be used to generate phase map information.

If you use Cameo+ data you will not exclude any information when generating phase data. However, some phases may overlap in the scatter plot.

If you use Element Maps as the data source, the phase data that you generate will depend on your choice of Element Maps and phases may therefore be missed if the wrong combination is chosen. However the separation of phases may be superior to those generated from Cameo+ data.

17.2 Using Cameo+ data

Select Cameo+ as Source data for the scatter plot. This will use the Cameo+ data as currently displayed in the Cameo+ window.

17.3 Using Elemental map data

Select Maps as source data for the scatter plot.

Select 3 Element Maps from the drop-down Element Map list.

17.4 Generating a Scatter plot

Press 'Scatter Plot' to display phases as clusters of pixels in the scatter plot.

17.4.1 What is a scatter plot?

Presenting data in the form of a scatter plot is a means of being able to rapidly identify the relationships between different parameters. In this case, consider a scatter plot being generated from three element maps. The apices of the plot correspond to each of the elements. Each X, Y position in the field of the maps corresponds to a count somewhere in the scatter plot. Those counts that appear at the center of the scatter plot, correspond to X, Y positions in the maps that have the same intensity in all three maps. If a count appears in one of the apices of the scatter plot, this means that there are X, Y positions where one

map has intensity but the other two maps are zero at these X, Y positions. Every X, Y position in the field where the relative intensities in the three maps are the same will produce an additional count at the same point in the scatter diagram. In practice, slight variations in composition and statistical noise in the maps results in a scattering of points forming a cluster. Maps with higher counts produce tighter clusters, which are easier to delineate. Thus, by encircling a region in the scatter plot you can find all X, Y positions that correspond to a particular composition or phase.

17.4.2 Scatter Plot tools

Select an appropriate tool from the selection to isolate the phase you want. This will record an entry for this phase into the table opposite and simultaneously display that phase on the electron image which is displayed under the PhaseMap tab on the top right form of your work space.

17.4.3 Selection Tools for Scatter Plot

Click on this if you wish to acquire PhaseMap data from a rectangular region. By dragging the mouse, a rectangular region can be outlined on the Scatter Plot.

Click on this if you wish to acquire PhaseMap data from an automatically selected region. This region will be selected according to a grey level threshold. This threshold value can be changed by selecting Mapping Options under Options from the main menu.

Click on this if you wish to acquire PhaseMap data from a region selected using this freehand tool.

17.4.4 Scatter Plot Scale

There are three different scales for displaying the data in the scatter plot, Logarithmic, Grayscale and Color. The Logarithmic scale is the default scale for displaying the data.

Logarithmic scale

Data is displayed as colored clusters. This scale is particularly useful in revealing fine detail in the plot since the intensity of the pixels is automatically adjusted to display the maximum information in the scatter plot. The lowest pixel intensity is scaled up and the highest pixel intensity (255) is scaled down so that the fine details of the data are displayed. The table below shows the color versus intensity of the logarithmic color scale.

Color	*Intensity*
black	*0*
dark blue	*1*
blue	*2*
turquoise	*3-4*
green	*5-8*
light green	*9-15*
yellow	*16-30*
orange	*31-60*
red	*61-120*
light red	*121-238*
pink	*239 and above*

Gray scale

Data is displayed as black and white clusters in the scatter plot. The brightness of the clusters can be adjusted by using the scroll bar present at the bottom of the scatter plot.

Color scale

Data is displayed as red, green and blue clusters in the scatter plot. The brightness of the clusters can be adjusted by using the scroll bar present at the bottom of the scatter plot. This is particularly useful for identifying any detail within clusters. Note that the color of the clusters corresponds to the Cameo+ color.

17.5 PhaseMap colors

Note that the colors used in the scatter plot, PhaseMap and phase table are self consistent. You can optionally chose other colors to be used in the phase table and PhaseMap by double clicking on the color in the phase table.

If you are using Cameo+ as your source data you may wish to use the Cameo+ color as your Phase color. This option can be selected by going to Mapping Options under Options from the main menu.

17.6 Displaying Phase Information

You can display phase information in a number of ways. Click in the box adjacent to the particular phase. This will make that phase active.

If you wish to see the phase displayed on the electron image select the ‘PhaseMap’ tab on the top right of your workspace. You can uncheck the ‘background box’ if you do not wish to see the PhaseMap superimposed on the electron image. The location of the pixels on the scatter plot from which the phase information was derived is also indicated.

The percentage area this phase occupies is also indicated in the table.

The spectrum derived from any active phase can be displayed by clicking the ‘spectrum tab’ on the top right part of your workspace.

If you wish to delete a particular phase, make it active, press delete and this will delete it from the list.

If you want to see the phases in the currently displayed percentage areas of the PhaseMap table press the pie chart or bar chart tab on the PhaseMap table.

If you want to see all the phases currently in the PhaseMap table displayed at once, press ‘display’.

You can select which phases you want to exclude from view by clicking in the far right hand box adjacent to the particular phase in the PhaseMap table. This will mark a cross next to the entry.

Note

If you have selected Maps as your data source and created a scatter plot, you will observe a tab marked 'mix' on the window to the top right of the work area. If you select this tab, the RGB image you see is the superposition of the three maps (Red, Blue and Green) you have selected.

18. QuantMap

QuantMap is a software application designed to generate quantitative Element maps from SmartMap data. Quantitative data is calculated from spectral data stored at each pixel of the SmartMap according to the current quantitative conditions and then displayed in the form of quantitative maps.

Note that no WD data will be quantified.

18.1 QuantMap Resolution

You can set the resolution of the QuantMap created from your SmartMap data by choosing the number of pixels in the X and Y dimension. Note that for a square monitor, the options are 32 x 32, 64 x 64, 128 x 128, 256 x 256 or 512 x 512 pixels.

The resolution options available will depend on the resolution the SmartMap was collected with. Note that QuantMap data cannot be created with a resolution greater than that of the original SmartMap data.

18.2 Generating a QuantMap

Once you have selected the required resolution with which to generate your QuantMap, press Start. Note that the time taken to generate a QuantMap will depend on a number of factors including the resolution selected and the number of elements in the fixed list.

When 'Start' is pressed, QuantMap builds X-ray maps from the spectrum data at each pixel which is deconvolved and intensity corrected according to the current Quantitative setup. Note that you can select any processing option but the Element list must be fixed for QuantMap.

The progress bar gives an indication of how long to go before spectrum processing is complete. You can press 'Stop' if you wish to stop the QuantMap generation before completion.

The distribution of your quantitative data in the form of element maps can be viewed in the Element Map step.

If a problem is encountered at this stage, useful diagnostic information may be obtained by quantifying the sum spectrum of the SmartMap in a standard application such as Point and ID.

18.3 Threshold Level

You can set the sigma level you wish to apply to your quantitative data from the drop down list. The element maps are automatically re-displayed with this new threshold applied.

18.4 Reviewing QuantMap Data

The Element Maps step allows you to review your QuantMap data in the form of element maps. Quantitative maps for all the elements in the current quantitative setup will be displayed in this step. Once you have generated a QuantMap in the QuantMap step, and you enter the Element Map step, a quantitative map for every element will be automatically saved in the data tree, together with a fit index image.

18.5 Combining Color Information

This functionality allows you to combine several quantitative maps together and display the combined data superimposed on the electron image. It can help you to visualize regions where more than one element is present or absent by assigning a color to each element distribution.

Select the maps you wish to combine by holding down the shift key and clicking on the maps. Note that you can select any number of maps. Press the color mix button to create the color mix image known as a QuantMap Mix image. Each element is then assigned a color when superimposed on the electron image - this color corresponds to the map label. The resultant color distribution in the final QuantMap Mix image will depend on the distribution of the selected elements.

The QuantMap Mix image will automatically be saved in the data tree.

Note that in some cases it may be helpful to adjust the contrast of the individual maps before they are combined in order to see features more clearly.

Quantitative information on the area occupied by a particular phase can be obtained using PhaseMap in the Mapping navigator. Select the 'QuantMap Mix image to generate the scatter plot' option, press Scatter plot and select the QuantMap mix tab to display the resultant PhaseMap.

We recommend that you refer to the help on the PhaseMap step for details on the display and generation of phase information.

Note that the spectrum derived from any active phase can be displayed by clicking the ‘spectrum tab’ on the top right part of your work space.

18.6 Smoothing Data

Pressing this button will apply a 3x3 smoothing function to the your quantitative element maps, whose weighting function is:

1 2 1

2 4 2

1 2 1

19. AutoMate

Introduction

You can set up lines and grids of stage points and specify areas to be covered so that the same data acquisition is done at every stage point.

Context menus are provided in the Stage Layout step for adding or deleting items such as Projects, Samples, Sites of Interest, Areas and Data Tasks. You can edit the layout of stage points or fields on areas.

The same Task can be used across several Sites of Interest. Changing the acquisition parameters of a shared Task in one Site of Interest will change the acquisition parameters everywhere else this Task is used.

You can acquire Analyzer X-ray spectra using the microscope scan at a specified magnification from either a point at the center of the field or over a rectangular area with a specified width and height at the centre of the field.

A Stage mimic display is provided in the Stage Layout and Run steps.

Default Data Acquisition Task Instructions are defined and are added automatically when a new task is started. These Default tasks can be modified as required to suit the particular user's requirements.

19.1 Context Menus

The context menus are available in the Stage Layout step to create and modify Tasks to collect data from related stage points . You can access the available Context menus by right clicking on an item on the Task tree on the right hand side of the Stage Layout screen. The items in each Context Menu will vary depending on what items are valid for that point in the Task:

Project

Add Project

Add Sample

Delete (Project)

Rename (Project)

Sample

Add Site of Interest

Delete (Sample)

Rename (Sample)

Site of Interest

Add Area

Add Data Task

Delete (Site of Interest)

Rename (Site of Interest)

Area

Edit Area

Set Layout

Delete (Area)

Rename (Area)

Spectrum Acquisition

Delete (Spectrum Acquisition Task)

Rename (Spectrum Acquisition Task)

Spectrum

Beam control for Spectrum

Delete (Spectrum)

Image Acquisition

Delete (Image Acquisition Task)

Rename (Image Acquisition Task)

SmartMap Acquisition

Delete (SmartMap Acquisition Task)

Rename (SmartMap Acquisition Task)

19.2 How to record a new Task from one Sample

You can record a Task to collect data from user selected (unrelated) points on a sample.

- Press the Start button to record a new task.
- A new Project and Sample are assigned to this task automatically: Project 1 and Sample 1.
- Note that the background of the work area has changed to reflect that the system is in recording mode.
- You are now in a position to setup a series of instructions such as acquiring a grid of spectra on a particular Site of Interest, followed by acquiring a SmartMap at another Site of Interest which may be acquired with different column and stage positions. How to setup this example will now be outlined in detail below.
- Click on the Point and ID navigator button.
- Note that you are still in recording mode in the steps in Point and ID.
- You may enter information relevant to this Project and Sample in the Sample and Project steps and, if required, check the microscope setup and Quant optimization.
- Go to the Site of Interest step and acquire a Site of Interest with the required setup conditions in Image Setup.
- Next go to the Acquire Spectra step and select the regions/points/grid or line of points on your image. If you wish to acquire a grid of spectra (or a line of spectra), remember to press the green 'Go' button to save these beam points in a list ready for acquisition.
- Once you are happy with this, press the Mapping navigator button if required.
- Acquire a new Site of Interest.
- Select a region/whole area of your image to acquire SmartMap data from and press the 'Start' button.
- Now press the AutoMate navigator icon and return to the Record step.
- Press the Stop button to stop recording your Task.

Note that you can also record information in your Task using the Analyzer navigator.

1. Press the Analyze navigator button
2. Press the new 'Site of Interest' button - the default label is 'Group of spectra' when you are in record mode.
3. Select the area of interest on your microscope - note that spot mode is not supported in Analyzer when using AutoMate.
4. We recommend that you increase the magnification so that the beam rasters over a small region.
5. Press the start button.
6. Select another region on your sample using the microscope.
7. Press the start button.

8. These regions for acquisition will be stored under the same 'Group of spectra' until the new 'Site of Interest' button is pressed or the task is run.

19.3 How to record a new Task from multiple Samples

1. Record a Task from a Sample as explained in 18.2.
2. Go to the Sample step and add a new Sample.
3. Acquire a new Site of Interest.
4. Select a region/whole area of your image to acquire SmartMap data from and press the 'Start' button.
5. Now press the AutoMate navigator icon and return to the Record step.
6. Press the Stop button to stop recording your Task.

19.4 Tasks containing combined ED/WD data

You can automate the acquisition of combined ED/WD data. This data can be acquired in both Point and ID and Analyzer. Once you are in record mode, the setup to acquire combined ED/WD data is the same as if you were to acquire the data live. However, note that spot mode is not supported in Analyzer when using Automate.

Ensure that you have chosen the appropriate method of beam current measurement in Automate setup. You have the option to use separate measurements for ED and WD, beam current measurement for ED and WD or element optimization for ED and WD. Once you have decided on the mode of measurement, you can chose the time interval between beam current measurements.

19.5 Stage repositioning during an AutoMate run

There are a variety of mechanisms used to provide automated stage control on electron microscopes. Each mechanism will perform differently. For example, some stages move very fast and some stages are designed for high precision positioning. Some microscopes offer a choice of stages to suit different applications.

If a stage is moved, it is not always possible to move it back exactly to the same position because of tolerances in the mechanism. The biggest source of error is so-called "backlash" and the effects of backlash are usually minimized automatically by the controller taking up the slack and always approaching the target position from the same direction. Even with a backlash correction to aid repositioning, a typical stage may still end up a few microns away from the original position.

If the stage is moved during an automated run it may not return to exactly the same position where the original Site of Interest image was acquired for point selection. When the Task is run, a new Site of Interest image is acquired at each stage position and the points selected for analysis may appear to have moved relative to their intended positions on the sample. It is worth experimenting at a suitable magnification (e.g. 10,000 X), selecting points on well defined features, to determine how good your particular stage is at returning to the same position.

Once you have determined how many microns uncertainty there is after stage repositioning, you can use this figure to make a sensible selection of points for analysis. When you record a new task, the micron marker bar on the Site of Interest image will help you decide if stage repositioning error is likely to move your analysis points too far away from their intended positions.

19.6 AutoMate Setup

19.6.1 Settings for each Site of Interest

- ***Recorded column conditions***

Check the 'Use Recorded Column conditions' box if you wish to use the column conditions, which were recorded during the Record stage. If this box is not checked, the current column conditions will be used.

- ***Auto contrast and brightness***

  Check the 'Set Auto Contrast/Brightness' box to ensure that you optimize these parameters for each Site of Interest you acquire. This option will only be active if your microscope supports this functionality; otherwise the check box will be grayed out.

- ***Auto Focus***

  Check the 'Auto Focus' box to ensure that you optimize this parameter for each Site of Interest you acquire. This option will only be active if your microscope supports this functionality; otherwise the check box will be grayed out.

- ***Acquire image data only***

19.6.2 Settings for End of Run

- Beam off
- Filament off

19.6.3 Settings for Quant Optimization

- Set the time interval for Quant Optimization
- Selection of Livetime
- Halt the run if Quant Optimization fails

19.6.4 Settings for Beam current measurement

- Select the time interval for Beam current measurement
- Select Measurement Method

  Select the measurement method you wish to use.

  Separate measurements

Refer to the on-line Help for full details of Beam measurement methods.

19.7 Edit Task

This step allows you to edit the current Task shown in the Task viewer. For example you may wish to edit the acquisition conditions of spectra, electron images or SmartMap data.

Note that the same acquisition Task instructions can be shared across multiple Sites of Interest. Changing the acquisition parameters of a shared Task in one Site of Interest will change the acquisition parameters every where else this Task is used.

To edit any parameters associated with a Site of Interest or instruction below it, select the item on the Task tree. These parameters can then be viewed and edited in the forms below.

You can also edit the column and stage values of the currently selected instruction. If you wish to set the parameters to the current microscope values, press the 'Read Microscope' button.

19.8 Run

This step allows you to run the Task currently shown in the Record step.

1. Press Start to commence the Run.
2. As the Run progresses, the current Site of Interest is shown in the image viewer, if appropriate.
3. All associated information such as which Project and Sample the Site of Interest is associated with is shown to the right hand side of the image viewer.
4. As spectra are acquired during the AutoMate Run, they are displayed in the Spectrum viewer in the lower part of the work area.
5. Information about the current status of the Task is shown to the right hand side of the screen.
6. During a Run, you can select to view a previously acquired spectrum in the current Site of Interest or Group of spectra from the spectrum drop down list. To revert back to view the current spectrum, select 'Current Spectrum' from the list.
7. If you are acquiring SmartMap data, the image displayed will be in the form of a Cameo image, provided that you have Cameo available.
8. The sum spectrum for the SmartMap acquisition is shown in the spectrum viewer.

AutoMate data storage

During a Run, AutoMate data is automatically written to disk as soon as it is collected. When the run stops or is stopped, then the AutoMate project is added to the data tree. It is also added into the MRU (most recently used bits for convenient retrieval).

20. Montage

Montage allows you to image and map over large areas of samples in the SEM by combining the data from multiple Sites of Interest into a single Site of Interest.

In normal operation using SEM and EDS, you can only view data i.e., an image and element maps from individual Sites of Interest. Montage enables you examine large areas of samples in the SEM. In application such as the diffusion profile of an element in a sample, you may like to view the diffusion across a large area that extends across many fields. Montage can be extremely useful for this. You can collect data from large number of fields from a sample using the AutoMate software. Montage combines the image and SmartMap data from a sample and gives you a single high resolution image and SmartMap data across the montaged image. You can also include WD maps in the montaged SmartMap.

20.1 How to create a montage including SmartMap

1. Select 'Montage' step from the Automate' navigator.
2. Click 

in the Montage step. The 'Load Source Data' dialog box is displayed.
3. Select the project file that you wish to use for creating the montage.
4. Click 

in the 'Load Source Data' dialog box. A montage of images of a sample is displayed in the Montage Viewer. If you have more than one sample in your Project, select a Sample from the drop-down list to display the montage.
5. Check for any misalignment of fields and make changes using 'Adjust' mode.
6. Select the resolution for the output image.
7. Select an area on the montage by clicking and drawing out a rectangle.
8. Check the 'Include SmartMap' check box.
9. Click 

.
10. A new Project, the selected Sample, a new Site of interest and a SmartMap are added to the Data tree.
11. Now you can view the SmartMap data in the 'Mapping' navigator.

20.2 Montage Viewer

The Montage Viewer is specifically designed for viewing and manipulating montages of images.

In the Montage Viewer, you can perform the following operations:

- Selecting an area to montage.
- Zooming in and out of a montage.

- Manually aligning fields in a montaged image to correct for any misalignment. Individual fields can be moved one pixel at a time.
- Adjusting the brightness and contrast of the montage.

You can use the keyboard and wheel mouse to control some of the functions provided in the Montage Viewer. The details of the controls are described below:

Brightness/Contrast

You can control the Brightness/Contrast of the image using the following keys:

Insert - Increase Brightness

Delete - Decrease Brightness

Page Up - Increase Contrast

Page Down - Decrease Contrast

Zoom

You can zoom in on an image and zoom out using the following keys:

Home - Reset Zoom

+ - Zoom in

- - Zoom Out

Mouse Wheel - Zoom In/Out

Middle Button - Reset Zoom

CTRL - Allows you to use the left mouse button to drag the image around to see different areas on the image. If the left mouse button is already pressed, the original operation will continue when CTRL is released.

'Select Area' Mode

This mode allows you to select an area on the montage. The normal operation mode (no modifiers) allows you to drag a rectangle out using the left mouse button. Use CTRL to drag the view.

'Adjust Fields' Mode

This mode allows you to adjust the position of individual fields (Sites of Interest) within the montage. Use CTRL to drag the view.

Left button:

- Double-click selects or deselects an individual field.
- Drags the selected fields if there are any.

- 'Click' deselects all fields if not over a selected field. Override this behaviour with ALT.
- SHIFT + Drag allows you to select multiple fields using 'rubber-band' box.

ALT in combination with other operations will allow you to add fields to existing selection.

Arrow Keys - Move selected fields in the corresponding direction, one pixel at a time.

'Bring to Front' or 'Send to Back' selected fields using Context menu or Space Bar on the keyboard.

Adjust Alignment

In Adjust Fields mode, you can correct for misalignment between the images, maps or TopMap before creating a montage. Select **Adjust Alignment** from the context menu in the Montage Viewer. The Adjust Alignment dialog opens. You can use the interactive sliders to align images, maps or TopMaps displayed in the Montage Viewer along X, Y and Beam rotation axes. All images are manipulated together.

21. Feature

INCA Feature allows unattended collection of data from features detected in one or more samples. A feature may be a small particle of dust, mineral, GSR (Gunshot residue)or inclusions in steel. The data collected using INCA Feature provide the following information about the features:

Morphology

- Area - Area of whole feature in square microns
- Length - maximum Feret
- Breadth - minimum Feret
- Perimeter - perimeter of whole feature in microns
- AspectRatio - lenghth/breadth
- Direction - of maximum Feret. This is the angle from the horizontal in degrees.
- Shape - sq(Perimeter)/4Pi x Area
- ECD - square root of (4 x Area)/p
- Mean gray level - mean image gray level over an individual feature.

Chemistry

- Spectrum area - number of counts in the whole spectrum (except the noise peak) for an individual feature
- % - weight% of each element

Position

- Beam X - of longest chord
- Beam Y - of longest chord
- Stage X - calculated stage position of the center of the longest chord.
- Stage Y - calculated stage position of the center of the longest chord.
- Stage Z - calculated stage position of the center of the longest chord.

21.1 Area layout

This step allows you to define areas over which the feature scan will be done. A sample can contain a number of areas and each area can contain a number of fields.

The area layout contains the area locations (rectangles, lines, points and references for quant optimize and/or beam current compensation) in addition to the field layout within areas.

Area layouts are saved as part of a feature database but not part of a Recipe. You can therefore re-use area layouts with different samples and recipes. If you wish to move an area layout to another system, the .adb file needs to be copied from the INCA\Data\Feature folder. The area layout is specific to one microscope type and may not work correctly on a different microscope or stage.

You may wish to perform a Feature run over a single field or multiple fields.

21.2 Single field analysis

The Feature run will proceed at the current stage position.

21.3 Multiple field analysis

A Feature run can be performed over multiple fields, areas and samples. These can be selected in the Area layout step. You should note that this step will not become active until you have defined a Feature database in the Recipe step.

Select the areas to be used with the current sample by clicking on them in the Select Areas list or on the stage mimic directly. You can use the ctrl key to select multiple areas.

21.4 Define a new Area layout

Press the new area layout button to the left of the drop down list to start a new area layout.

Enter a name in the text box.

Existing area layouts can be selected from the drop down list.

Press the Area layout tab.

Add, remove or edit an area by clicking on the appropriate button.

21.4.1 Area layout tab - Add

Specify the shape of the area to be added from the drop down list and press the Next button.

A number of areas can be defined over which the feature scan will be performed. These areas are:

Single stage point.

This 'area' is defined as a stage position and it can only have one field defined on it. It is equivalent to the Site of Interest used in Point and ID and AutoMate.

Move the stage to the single point that defines the area, wait for the stage to finish moving and press the Next button to specify the point.

Specify the name for the point and press the Finish button to add this point to the list of areas displayed on the Select Area tab.

Line

This is defined with two stage positions, the start and the end of the line. Fields for data acquisition are defined along the line as specified in the field layout.

Note that if Z is different at the two ends of the line, Z will be adjusted for fields along the line.

Move the stage to the first point that defines the area, wait for the stage to finish moving and press the Next button to specify the point.

Move the stage to the last point that defines the area, wait for the stage to finish moving and press the Next button to specify the point.

Specify the name for the line and press the Finish button to add this line to the list of areas displayed on the Select Area tab.

Rectangle

This is defined by specifying four stage positions, one for each vertex of the rectangle.

Note that the rectangle can be irregular and need not be aligned with the stage movement axes.

Note that the rectangle must not have any concave sides. If it does, then the rectangle will be reduced to a triangle.

Note that if Z is different for each vertex then Z is adjusted so that fields lie on the plane defined by three of the vertexes.

Move the stage to the points that define the rectangle, wait for the stage to finish moving and press the Next button to specify the points.

Specify the name for the rectangle and press the Finish button to add this area to the list of areas displayed on the Select Area tab.

Circle

A circular area, such as a circular stub is defined by specifying three points on the edge of the circle.

Note that if Z is set differently for the three points, then a tilted circle will be defined and field positions on the circle will be adjusted so that they lie on the surface of this tilted circle.

The points must be separated sufficiently that the circle can be defined accurately. When defining a circle, try to arrange the three points 120 degrees apart round the circle. While two points can be close together if the other is diametrically opposite small errors in the position of the two points can cause circle definition errors.

21.4.2 Area layout tab - Edit

The position or name of an area can be edited in the area layout tab by selecting the area and selecting "edit". The Stage will move to the first position that is currently recorded as defining the area. The stage position can then be changed.

When 'Next' (or 'Finish') is pressed, the stage position is recorded and the stage will move automatically to the next point that defines the area.

The area name can then also be changed. The modified area position is placed in the area layout and the display updated when 'Finish' is pressed. If 'Cancel' is pressed then no changes are made. ·

Note

The mechanical stage may have backlash that can cause problems with accurately determining the position of areas. Often for automatic stage moves, a backlash correction is implemented so that the stage always approaches the final position from the same direction. Points that define an area will be

more accurate if the final move to the position is made from the same direction as the backlash correction.

The stage may also be subject to mechanical positioning errors that can lead to inaccuracies in positioning and repositioning of fields and features. Note that backlash correction will help but cannot correct for all errors.

The points that define an area must all have the same tilt and rotate. If tilt or rotate changes then you will be prompted and given the opportunity to set the same tilt and/or rotate.

21.4.3 Defining the Field layout

For a point, a single field is defined as centered on the stage position of that point.

For other area types the fields can be laid out in a regular grid or randomly. The coverage of the area can be selected in two ways: the percentage of the area to be covered or the maximum number of fields to be used.

If All the Area/Line is selected, then the area is tiled with touching fields arranged to cover the whole area (or line). If a guard zone is selected then the fields will overlap by the guard zone distance. The percentage area is 100% when the whole area is tiled with touching fields (or overlapping if a guard zone is specified).

If 'Random fields' are selected, then a random selection of the tiled field arrangement is defined to cover the required percentage or number of fields.

If 'Regular fields' are selected, then a grid (or line for a line) of fields are defined. The grid spacing is selected so the required percentage area or number of fields is covered.

Note that it may not be possible to use the exact number of fields as the specified number may not fit into the required area given the Y and X dimensions.

21.4.4 To define a new Area layout

Press the new area layout button to the left of the drop down list to start a new area layout.

Enter a name in the text box.

Existing area layouts can be selected from the drop down list.

Press the Area layout tab.

Add, remove or edit an area by clicking on the appropriate button.

21.5 Stage mimic

The stage mimic offers a powerful way to visualize the layout of your stage in terms of the relationship between samples, incident electron beam and EDX detector. It is also a tool with which to drive the stage to a defined position.

Double click on an area on the stage mimic to drive the stage to that area.

Single click to select an area and use ctrl-click to select multiple areas.

You can change the view direction of the stage mimic by clicking and dragging.

The track ball controls the stage mimic tilt and rotate on screen.

Zoom using the + and - controls are displayed underneath the stage mimic.

21.6 Recipe step

All parameters which define the detection, acquisition, measurement and classification of features are stored and saved in a Recipe. Recipes (.rdb files) are stored globally and are available to all users of the system to use with different samples. A Recipe contains a number of parameters including:

Recipe setup (e.g. termination limits, saving options)

Detection setup parameters (e.g. resolution, speed, binary and gray level processes)

Spectrum setup (e.g. number of ED passes, livetime, energy range, process time)

Quant optimization (e.g. optimization element, frequency of quant optimization)

Class setup (e.g. setting up classes and criteria)

Quant setup (e.g. element list).

Note that the quant setup and class setup are stored separately and can be reused in different recipes.

21.6.1 Locking recipes

You can protect a Recipe by locking it. This means that you cannot edit the Recipe settings and then save the recipe under the same filename. If you wish to edit a locked Recipe, open it, edit it and save it under a new name.

21.6.2 Creating Databases

Within INCA Feature, data is managed within projects and each project can contain many samples. Feature data is stored in a database file which can be either embedded or not embedded in the project file. For an every new project you will need to create a new database.

When not embedding the database

Feature needs to know the location of the project so that it can store the database file relative to it. Data is written directly to the database file location during a run. This way of storing data is the most speed efficient.

- Enter a name for your project
- Enter a name for your sample
- Save the project
- Press create database
- Feature database will appear as an entry in the data tree

In the unlikely event of your system crashing during a run, data up until the time of the crash will be present in the database file.

If you wish to move and view your data to view files on another computer, you will need to transfer both the project (.ipj) file and the file folder containing the database together.

When embedding the database

In this case, data is written to a temporary file until the moment that you save your project. At this point, data gets transferred from the temporary file to the project file.

- Check the 'Embed database option'
- Press create database
- Proceed

Note that the name of this temporary file is given at the top of the form when the embedded database is created.

In the unlikely event of your system crashing during a run, you can recover the temporary database file by pressing Recover Database.

If you wish to move and view your data on another computer, you need only transfer the project (.ipj) file.

Delete database

Press this to permanently remove the database from the system.

Recover Database

- This really only applies if you have embedded your database.
- Restart INCA.
- Press Recover Database.

Save the project with the database either embedded or not. Note that you should not shut down INCA before you have recovered the temporary file, as INCA will delete all temporary files on a normal exit.

21.7 FeatureMap

Check this box in the ‘Recipe’ step if you wish the full feature detection algorithm to be applied to existing SmartMap data. Existing SmartMap data can be used to setup classification schemes.

Procedure

Ensure that your current Site of Interest has SmartMap data.

When the box labeled, 'Use SmartMap data from current SOI', is checked, a single area is automatically created.

Thresholds can be set and processes applied to the electron image and subsequent binary image to ensure optimum feature detection.

You can then process your SmartMap data in the ‘Run’ step.

Note that the magnification at which the SmartMap data was acquired will be used, not the magnification specified in the current recipe.

21.8 Recipe Setup step

You can select from the following options for conditions for the 'End of the Run':

21.8.1 Turn beam off

Check this box to turn the microscope beam off at the end of a feature run. The exact way in which the beam is turned off will depend on what control is provided by the microscope manufacturer.

21.8.2 Turn Filament off

Check this box to turn the filament off at the end of a feature run.

21.8.3 Saving options

For each feature you can:

Save the spectrum

Save the diagram/binary image/chord diagram.

Additionally, you can save the image for each field.

We recommend that you save the spectra for each feature in case you wish to reprocess and /or reclassify using a different element list in the Quant setup.

The overall file size should be considered and sufficient disk space available if you wish to save the above items. Saving these items will also marginally increase the time for a run to complete.

21.8.4 Termination Limits

When the termination condition is met, Feature will stop analyzing the current field, area or sample and move onto the next field, area or sample. This can avoid collecting excessively large amounts of data, reduce the time spent analyzing samples and minimize disk storage space.

Field termination:

Total number of features in a field - the stage will move to the next field once it has detected the number of features specified.

Total features limit in a rank - the stage will move to the next field once it has detected the number of features specified of a given rank.

Total time spent in a field - the stage will move to the next field once the number of minutes specified has elapsed scanning the field.

Area termination

Total number of features in an area - the stage will move to the next area once it has detected the number of features specified.

Total features limit in a rank - the stage will move to the next area once it has detected the number of features specified of a given rank.

Total time spent in an area - the stage will move to the next area once the number of minutes specified has elapsed scanning the area.

Sample termination

Total number of features in a sample - the stage will move to the next sample once it has detected the number of features specified.

Total features limit in a rank - the stage will move to the next sample once it has detected the number of features specified of a given rank.

Total time spent in a field - the stage will move to the next sample once the number of minutes specified has elapsed scanning the sample.

Example

For example, you may only want to acquire data from 20 Rank 1 features. Note that these features may belong to more than one class. To do this you would select the required termination condition (field, area or sample), enter 20 into the number of features box and select Rank 1 from the drop down list.

During the run, all features will be analyzed and sorted into appropriate classes (assuming that a classification scheme exists). When a total of 20 features of Rank 1 are found, the termination condition is met and the appropriate action will be taken. If the termination condition is not met data acquisition continues until all the fields are analyzed.

21.9 Detection Setup

This step allows you to set up parameters associated with detecting features such as image source, resolution, magnification, minimum feature size, gray and binary image processing.

21.9.1 Field setup

There are a number of parameters that can be set to optimize feature detection performance using Feature.

Type of signal

The choice of signal is important and can make a considerable difference to whether features are reliably detected. Backscattered electron images (BSE) are generally used in preference to secondary electron (SE) images as the contrast in BSE images is heavily dependent on the atomic number of the material which helps to differentiate between different phases.

The many mechanisms such as topography that influence the contrast in secondary electron images make it difficult to produce a meaningful binary image from which features can be easily differentiated.

Resolution

A higher resolution will allow a lower magnification to be used for the same pixel size. This will reduce the time taken for acquisition over an area because fewer stage movements are required. Changing from a resolution of 1024 to 2048 should allow the magnification to be halved for the same pixel size and the number of fields to be reduced by a factor of 4.

First pass image

The first pass image speed can be set to values between 1 and 10 microseconds per pixel. Shorter times will reduce the scan speed but there will be two effects:

- The data will be noisier. This can mean that small features are missed. It can also cause spurious detection of particles which can mean that a second pass scan is made when it should not be. This can reduce the feature detection efficiency and can also increase the time taken.

- Some types of backscattered detector have a slow response to changes in brightness. This can cause small features to be missed if the scan speed is too high.

Second pass image

The second pass is used to locate the features accurately and to eliminate spurious noise features. This is only done where a feature has been detected by the first pass and therefore minimizes the effect on the overall feature detection time. The second pass dwell time should be:

– Long enough such that any scan lag is eliminated in order that the feature can be detected accurately. The value will depend on the microscope, values of between 20 and 100 microseconds dwell are generally suitable. However, if you find that there is no contribution in the spectrum present from the feature, you should increase this second pass image time.

– Long enough that a good low noise signal can be detected and an accurate threshold determination can be made.

Advanced options

Using Advanced options you can fine tune the feature detection setup.

Leading Edge

The start of a feature is detected by the first pass at a position A. The leading edge is the number of pixels before this position A. The second pass scan will start at this leading edge position.

Trailing Edge

The end of a feature is detected by the first pass at a position B. The trailing edge is the number of pixels after this position B. The second pass scan will finish at this trailing edge position.

These options allow for scan lag during the second pass.

During the first, fast pass, delays in the electronics and in the scan generator, will tend to cause the observed position of the feature to appear to be behind where it actually is. This can vary depending on the microscope and detector. The effect of scan lag will be greater with short dwell times. Typical values can be between 5 and 20 microseconds, corresponding to between 5 and 20 pixels at the shortest dwell time. In some cases it can be greater on the left hand edge of the field. If small features tend not to be detected, particularly on the left hand edge of the field then increasing the leading edge may help. Increasing these values will slightly increase the particle location time because a greater number of pixels are scanned at the second pass dwell time.

Magnification

As the magnification is reduced, the area scanned for each field increases and therefore fewer fields need to be scanned to cover the same area. This reduction in the number of fields means that less time is spent moving the stage and that the overall speed of the run will be increase. A small reduction in magnification has a large effect because both the width and height of the field are increased.

21.9.2 Feature setup

Magnification

Enter a magnification here and the smallest expected feature width will be automatically calculated. If you enter a value for the smallest expected feature width, the magnification will be calculated from this.

Smallest expected feature width

This is the smallest feature width which will reliably be detected. Some smaller features may also be detected. The magnification displayed is calculated for the detection of features of the specified width.

Ignore features smaller than area

Features smaller in area than the values (in pixels) entered will be ignored and not detected. The length and ECD in microns are calculated using the magnification entered and reported.

The Equivalent Circle Diameter (ECD) of a feature is determined from the following equation:

ECD = square root of (4 x Area)/p

Guard Zone

The guard zone prevents double counting of features. The guard zone is a special area within a field of view setup in a particular way to enable feature counting across tiled fields. Any features which lie across the guard zone are included in the feature count, whilst those that are wholly contained within the guard zone and or touch the edge are excluded. In this way, each feature will be counted only once. The guard zone is aligned to the right and lower edges of the field and its width should correspond to the largest expected feature.

An individual feature should not extend from the measurement region through the guard zone and touch the edge.

The guard zone is displayed on the image. Setting a guard zone is only appropriate if the expected features are of broadly similar size. It cannot be reliably set if the range of feature sizes is too large.

Read magnification

Press this to read the current microscope magnification. This value will be used for all areas for a given sample during a run.

Image processing parameters

Adjusting the image processing parameters may improve feature detection efficiency and speed. If the image from which the features are extracted is noisy then instead of detecting one large feature, many small features may be detected. In addition to giving incorrect results, these multiple features will have to be scanned slowly and X-ray data acquired from them. The following image processing options available may be able to help:

The gray scale median and smooth filters may improve the image quality and so give better detection at the expense of a slight reduction in resolution.

The binary filters, particularly erode, dilate and hole fill can be used to eliminate or combine particles that are on the limit of detection.

Strategy for optimizing feature detection speed

- Select a representative sample area that has a typical distribution of features, especially including small and low contrast ones. Set the microscope conditions to give a good, low noise image with good contrast.
- Setup a conservative scan, where the minimum feature size is equivalent to 10 pixels. Setup a first pass speed of about 8µsecs, a second pass speed of 50µsec and a resolution of 1024.
- Scan over the area, detecting features. This is the base run where the detection efficiency can be assumed to be about 100% ·
- Increase the first pass scan speed, to 4, then 2 then 1µsec. Reacquire data and note the point at which the number of small features starts to fall off. Reduce the scan speed until this effect just goes away. This gives the optimum first pass scan speed. ·
- Reduce the smallest feature width, in steps, to about 4 pixels and adjust the magnification to the new value suggested and acquire. The number of features should be about the same. It will not be identical because the different field size and field positions will mean that the area acquired will not be identical. Use this data to determine a satisfactory minimum feature size.
- Try the high-resolution scan; again the magnification will need to be reduced. Acquire data and check that features are not lost. You may increase the first scan speed or the number of pixels as this may give you better detection efficiency at the higher resolution.
- If larger features tend to break up into smaller ones then try the gray scale and/or binary image processing options to reduce this.

Other factors affecting particle detection

There are a number of other things that can affect the feature detection efficiency and speed. ·

Noise

A noisy image may produce a lot of spurious feature detections, particularly in the fast feature detect scan or first pass scan. These spurious features then have to be scanned slowly in order that they can be rejected. Using a slower first pass scan speed may help because this will reduce the noise and so reduce the number of spurious features.

Increasing the beam current will also reduce noise but, in some cases, at the risk of damaging the sample.

Optimizing for feature detection reliability

Speed and reliability can have an inverse relationship. Operations that increase speed will tend to reduce the reliability of feature detection.

However, settings that may improve the reliability of feature detection are:

- Increasing the magnification. This will make the features appear larger and so they will be detected more easily. If small features are not required to be analyzed, then the minimum feature size can be increased to eliminate them.
- Reducing the first scan speed will give a lower noise signal and so will improve the reliability of detecting small features.

- If the highest resolution is used then noise in the scan generation circuits can mean that small features are not seen clearly.
- Increasing the contrast of the features against the background will allow the feature threshold to be set more accurately.

Additional note

The feature detection setup so far assumes that morphological measures of small features do not need to be accurate. The pixellation of small features will make their morphological measures increasingly inaccurate as they get smaller.

If accurate morphological measures are required then a higher magnification should be used such that the pixel size is sufficiently smaller than the minimum feature size. The minimum area in pixels can be increased in order to eliminate small features for which no data is required.

21.10 Feature Detection step

This step allows you to optimize the detection of features by calibrating the system against a standard and setting thresholds.

21.10.1 Calibration tab - setting up the backscattered detector

The points below describe how the backscattered electron detector signal is used to set the upper and lower gray level thresholds. All features which give a signal within these thresholds are set to white and all others are set to black.

It is important to correctly set the upper and lower threshold or time may be wasted detecting unwanted features. For given detector contrast and brightness settings, lower than optimum threshold settings may increase the number of background features detected and unnecessary time will be spent on each field. Higher than optimum threshold settings may increase the chances of missing features.

Select the area defined for the calibration and beam compensation check from the drop down list. To move the microscope stage to this area, press 'Move to Area'. The type of sample you use as a reference is important. Ideally it should have the same dynamic range over a given area as that over a typical area of your sample on which you wish to detect features.

Turn off the auto brightness and contrast on your backscattered detector. Press the start button to start continuous image acquisition. A live backscattered image of the area will be displayed and acquisition will continue until the stop button is pressed.

A line is automatically drawn on the image. Click and drag to move the ends of the line across a feature or features of interest. The adjacent graph shows the distribution of signal intensity across the linescan.

Adjust the backscattered detector gain and offset in order to optimize the signal level. Adjusting the gain (contrast) will increase or decrease the separation of the plateau of the waveform. Adjusting the offset (brightness) will move the waveform up and down.

You should then set the calibration levels on the graph to appropriate values.

21.10.2 Thresholds tab

This tab allows you to setup up to six pairs of thresholds on an image. It also allows you to switch between gray and threshold images to check the feature detection.

Method

Select the position you wish to move to from the drop down list and press 'Move to Reference'.

Switch off the auto brightness and contrast on your backscattered detector.

Press the start button to collect an electron image. If you have set any gray image processes, these will be applied to the electron image.

Select the number of thresholds you wish to set from this drop down list.

The histogram shows the frequency distribution of the gray level (where gray level represents the signal intensity). To set levels for the currently selected threshold, you can enter minimum and maximum gray level values directly or slide the markers beneath the image or move them directly on the histogram.

Press 'Feature Detect' to apply the thresholds and image processes defined in detection setup to the current image. The features detected are colored on the electron image and the total number in each threshold band displayed.

You can display either the gray image, threshold image or feature image by selecting the appropriate button above the image viewer. You may need to check the feature detection by zooming in (from right mouse click) and swapping between gray and threshold images to see that feature edges are correct. Several zoom steps may be needed for very small features. Thresholds can also be set from a calibration standard which may be useful if the features you are looking for are rare and not found in the setup field.

Note - right mouse click to show the various options available such as export facilities, full screen image display and image details.

21.10.3 Beam compensation checks

Beam current drift will affect image threshold. INCA Feature allows you to automatically compensate for beam current drift during a run by periodically returning to a calibration standard. The thresholds are then reset to compensate for drift such that features are reproducibly detected.

Method

Select the area defined for the calibration and beam compensation check from the drop down list.

Select the time interval between beam compensation checks from the drop down list. This will depend on the length of the run, the predicted beam current drift and the effect on the thresholding of the image.

During a run, the stage will move to this reference position, set the magnification and acquire an image.

The beam rasters over a small area on the standard (green square) and the mean threshold is determined. This value is monitored and if it changes, the thresholds are adjusted accordingly such that beam drift is compensated for. You can change the maximum deviation above and minimum deviation below starting value on the Calibration form from the Feature options accessed via 'Options' on the main menu. On the calibration form, you can adjust the sampled area width in pixels.

21.11 Spectrum Setup step

This step allows setting up spectrum acquisition parameters such as:

- Process time
- Spectrum range

- Number channels

You can select where on the Feature you wish to collect ED data from i.e., Whole feature or Center of longest chord. You should consider the interaction volume and homogeneity of the feature composition with respect to the size of the feature.

If you decide to make morphological measurements only by checking the tick box, all the ED parameters will be grayed out.

You can select the number of ED passes you wish to perform. Two pass acquisition is only relevant if you have an ED filter.

A simplified summary is given below:

- Feature locate
- Measure feature
- Passes Morph filter?
- Pass 1 ED spectrum acquisition
- Passes ED filter?
- Pass 2 ED spectrum
- Measure feature
- Classify feature

If you have an ED filter set for any of your classes, you should use two ED passes. Note that the final classification is based on the two ED spectra added together.

If you have no ED filter set for any of your classes, one pass is sufficient. However if you do set two ED passes, the 'Passes ED filter' step in the above sequence is ignored and pass 1 and pass 2 are carried out. Note that the final classification is based on the two ED spectra added together.

If an ED filter is set

Setting an ED filter allows features to be rejected based upon Pass 1 ED analysis. Features deemed 'interesting' after the first pass can be analyzed for much longer (Pass 2). In this situation, you should set the number of ED passes to 2.

If 'Use in ED filter' is marked Yes for a class, the class will be evaluated after a short ED acquisition step (Pass 1). If a feature is found to be a member of this class, then further ED analysis of the feature may be performed (Pass 2)

Note that a feature, its associated morphological results and its preliminary quant results based on Pass 1 spectrum are stored in the database even if it is rejected by the ED filter. In this case, features are stored in the 'Rejected ED' class.

21.12 Batch Setup step

This step allows you to review and edit the samples that have been selected for the batch run. Details of the recipe associated with each sample are shown. These include details of the setup conditions and areas to be analyzed.

Note that any discrepancies will be marked in red.

21.13 Run step

In this step you can start an automated run for feature detection in the areas that you have selected to analyze. Once the run is complete you should proceed to the review steps to view your data

21.13.1 Live review of data during acquisition

The user is allowed to review data for a selected feature on the current area during live acquisition. Select a feature of interest from the list to display its chemistry, classes, morphology and spectrum while the system is acquiring data.

21.13.2 Move to next area

When sufficient data has been acquired on an area you are allowed to move to the next area by clicking Move to Next Area button in the Run step.

21.14 Review Classes step

This step allows you to review the classification of features in a number of ways. You can reclassify your data using another class setup from the drop down list or go into class setup to edit or create a new classification scheme.

You will need to reprocess the data before you reclassify if you wish to use a different Quant setup than the one saved with your current recipe.

21.15 Review Features step

This step allows you to review individual features and relocate features of interest to reacquire data.

21.15.1 Reacquisition

Feature reacquisition

This step allows you to reacquire data from features of interest using different acquisition conditions from the ones that you used during an automated run. It can helps to improve the quality of data and give better characterization of features for further investigation.

The reacquisition of data from features involves the following steps:

- Setting up a run to acquire feature data from multiple samples using shorter acquisition conditions. This helps to get a quick overview of the samples' morphology and chemistry.
- Manual examination of the data in the Review Features step to find features of interest such as GSR specific particles.
- Relocating a selected particle and zooming in on it by increasing the microscope magnification.

- Going into the Reacquire step to **collect a new image** of the relocated particle.
- **Collecting a new spectrum** either by scanning the whole particle or from a user selected point on the particle.
- Acquiring an image and a spectrum several times before you decide to save the feature.
- Saving the reacquired feature by pressing **'Save Feature'.** The reacquired feature is added to the database with a new feature identification and its new morphological and chemical measurements.
- Choosing to either **delete the original feature** or to keep it. If you decide to delete the original feature it is deleted from the database and you can not restore it. If you decide to keep the original feature it is removed from the feature list and placed in the Rejected (Manual) class.
- Saving the conditions for feature reacquisition in the current recipe by pressing the Save Recipe button in the top left of the Reacquire screen.
- Going back into the Review Features step to select the next feature for reacquisition of data from it.
- Viewing all the reacquired features and manually marked features in the Review Features step by selecting 'Marked' features from the drop-down list.

21.16 Review Data step

This step allows you review all features data as a spreadsheet. Each row represents a feature, with columns representing class membership and measurement values.

22. Report

Reports can be arranged in various formats. You should first decide which report format/template suits you or your customer.

Select the way you wish your report to be arranged from the drop down list of available report templates presented as a tree view. These templates include those dedicated to reporting Sample and Project details.

If you wish to change the current image displayed, select the image/map type from the drop down list, which contains all images from the current Site of Interest.

If you wish to change the current spectrum displayed, select the spectrum you want in the report from the drop down list, which contains all spectra from the current Site of Interest.

The individual components can be manipulated in the usual way or you can use the right mouse button to access the available options in the context menu.

Note that you can zoom on any of the text boxes (e.g. Comment box) on the report page, by clicking with the right mouse button.

Press the 'print' button to print your report.

22.1 Creating Word documents

You can create a complete Word document, using the currently selected INCA report template by pressing the 'Create Word document' button, which will start the Word program.

22.2 Publishing your report as a Web page

You can publish all your data stored in the current project as a web page by going to the main menu, under File and selecting 'Publish as web page'.

22.3 Exporting your report as an HTML document

You can export your current report as an HTML document by pressing the appropriate button in the report step.

22.4 Producing your own Report

You can produce your own report by either exporting or copying data to other applications such as Word, PowerPoint etc or by creating your own Report Template.

22.4.1 Exporting spectra, images, maps, linescans and Cameo+ data

BSE and SE images, maps, linescans, Cameo+ images and spectra can be exported from INCA Energy in two ways thus allowing them to be read by appropriate software packages:

- By clicking with the right hand mouse button on the data label in the Data tree and selecting export. This allows the data to be converted and exported in file formats such as jpg and GIF.

- By clicking with the right hand mouse button on the image, linescan etc and selecting export. This allows the data to be converted into a bitmap (bmp), TIFF (tif), Jpeg (jpeg), Jpeg (compressed) file.

Note that it is the data as currently displayed which is exported. Therefore, if the current image has been zoomed, the contrast or brightness or the color has been changed or the spectrum scaling has been changed, this will be included in the conversion.

22.4.2 Copying spectra, images, maps, linescans and Cameo+ data

Spectra, BSE and SE images, maps, linescans, Cameo+ images and spectra can be copied to the clipboard from Energy in two ways:

- By clicking with the right hand mouse button on the data label and selecting copy.
- By clicking with the right hand mouse button on the image, linescan etc and selecting copy.

The data will be copied to the Clipboard. You can then use the application to which you wish to transfer the image etc to read the Clipboard.

Note that it is the data as currently displayed, which is copied. Therefore, if the current image has been zoomed, the contrast or brightness or the color has been changed or the spectrum scaling has been changed, this will be included in the copying.