EELS Acquisition using AutoFilter

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1 Introduction

The AutoFilter software provides an efficient and convenient means for technique-oriented control of the Gatan family of TEM post-column imaging filters. AutoFilter provides easy access to the key high-level functions required for conducting everyday EELS and EFTEM microanalysis, whilst conducting low-level functions such as microscope and filter control in an automated manner. Hence AutoFilter allows you to rapidly obtain directly interpretable results, without the inconvenience of the operational details.

The aim of this tutorial is to give the user an introduction to the spectroscopy-related aspects of the AutoFilter software, giving a practical overview to the features contained within the AutoFilter palette, and providing a step-by-step description of how to best use it to obtain EELS data with your imaging filter. After working through tutorial you should be able to run the spectrometer and acquire spectra. For customers that have the spectrum-imaging package a similar tutorial is provided as an introduction to EELS-spectrum imaging.

Detailed explanations of the software and how to do more complex operations can be found in other user guides specific to particular parts of the software.

- DigitalMicrograph for the imaging environment.
- EELS Analysis for the EELS specific processing.
- Spectrum-Imaging for the EELS spectrum-imaging software.

Depending on the exact configuration of your system other user guides may also be provided. Gatan also recommends a number of textbooks that users of the EELS analysis technique should find invaluable:


2 Hardware

The AutoFilter software requires a fully functioning and correctly configured energy-filtered TEM system; that is, a transmission electron microscope equipped with a GIF, its control electronics and a control computer on which the acquisition and analysis software is installed and through which the hardware communicates. The control electronics consist of four boxes; a Gatan Instrumentation Bin (or GIB) spectrometer control system for the lenses etc., a GIB power supply, an MSC camera controller and a TV-rate retractable camera controller. It is assumed that this equipment has been installed and correctly configured by your Gatan service engineer and is in correct working order. If not, please refer to the appropriate documentation or contact your local service representative for assistance.
3 Software

The software is split into two parts - a program that controls the hardware called FILTER CONTROL, and the viewing and processing software, DIGITALMICROGRAPH, within which AutoFilter is installed. Your desktop should have two icons for these programs. This tutorial covers spectral acquisition using the PC version of AutoFilter for DigitalMicrograph 3.6 or above.

4 Spectral Acquisition using AutoFilter

4.1 Starting Up

First turn on the computer, GIB, TV-rate camera control unit and TV-rate camera monitor. The order is not important. Start the FILTER CONTROL software followed by DIGITALMICROGRAPH (in that order). If the AUTOFILTER floating palette is not open in, launch it by selecting FLOATING WINDOWS / AUTOFILTER in DigitalMicrograph’s WINDOWS menu.

It is assumed that the camera is in its normal standby state i.e. cooled. The camera is cooled to minimize the detector readout noise. If for some reason the camera is not cooled, initialize the Peltier cooling using the switch on the GIF MSC camera controller. The temperature drops at a few °C per minute, so allow approximately 30 minutes for the system to fully stabilize. You should load your sample into the TEM, turn on the beam and align the TEM as for normal operation. For this tutorial it is assumed you use a sample of boron nitride on carbon film. This sample is provided with EVERY GIF system. Additional samples may be ordered through Gatan sales channels.

_Gatan supplies BN-C samples as they are reliable, give many areas than can be used for EELS and have three clear bright edges; the B K edge at 188eV, C K edge at 285eV and N K edges at 402eV._

4.2 The AutoFilter palette

All the commands you need to acquire spectra using a GIF are contained within the AUTOFILTER palette. The AUTOFILTER palette, with the analysis technique set to EELS, is shown in Figure 4-1. The layout of the AUTOFILTER palette reflects the flow of tasks one would normally follow when performing microanalysis, and its content changes dynamically with the specified analysis mode. It is comprised of four groups of commands. The first three groups of commands, TECHNIQUE, VIEW and ENERGY, enable you to set the mode and configure your instrumentation appropriately for acquisition of your EELS (or EFTEM) data. The last group of buttons, COMMANDS, allows you to acquire your data with a single click using the configured mode. The use of these groups of commands will be illustrated by example throughout this tutorial.
4.3 Locating the beam

It is advisable to locate the beam and align the GIF first in imaging mode using the automated alignment routines built into AUTOFILTER before proceeding to spectroscopy mode.

To locate the beam

- On systems with both a GIF and a Gatan camera (i.e. MSC), first go to the CAMERA menu, go to the CAMERA sub-menu and select GIF.
- Locate a hole in the sample at between $2k\times$ and $200k\times$ magnification in the normal magnification mode.
- Retract anything that may obstruct the beam. For instance, an MSC below the screen and bright field / dark field detectors should be retracted to the dark field position.
- Illuminate the center 2 to 10 cm of the TEM screen, and then lift the TEM screen.
• Select the TEM button in the TECHNIQUE option group (see Figure 4-2).
• Insert the TV rate camera by clicking SEARCH in the VIEW palette, followed by clicking on the IDLE/ACTIVE button to activate the view (the red dot should turn green when active).
• Insertion of the camera should be accompanied by a pneumatic hiss. If the camera does not insert, check that the control electronics are switched on. Failing that check that TV is selected as the SEARCH VIEW MODE in the AUTOFILTER VIEW OPTIONS dialog (accessed by clicking SEARCH with the ALT key held down).
• Finally, click ALIGN ZLP to initiate the automated zero-loss alignment routine. Ensure the METHOD is set to AUTOMATIC in the ZERO LOSS ALIGNMENT dialog (activated by clicking ALIGN ZLP with the ALT key held down).

Figure 4-2. DigitalMicrograph with the AutoFilter palette in TEM mode

The routine should automatically find and center the zero-loss peak. Once finished, the TV-rate camera will be automatically reinserted. It is important to ensure that the beam is found and is incident on the TV-rate camera before proceeding further. If the illumination is very intense, spread the beam a little; conversely, if the illumination cannot be seen then condense the beam until it can. If a beam still cannot be found on the TV-rate camera, refer to ‘Finding the beam by hand’ in the Appendix.

Once the beam has been found, the GIF should be aligned while in the TEM (or EFTEM) imaging mode to ensure the basic alignments are correct.
To align the GIF

- Click the TUNE GIF button in the COMMANDS option group with the ALT key pressed to initiate the AUTOFILTER IMAGE RECORDING OPTIONS dialog (Figure 4-3). If the GIF hasn’t been aligned recently, check all the TUNING PROCEDURE boxes. Otherwise, correct just 1st ORDER ISOCHROMATICITY and ACHROMATICITY.
- Click on the TUNE GIF button to initiate the automated alignment routine. During the routine, you may be asked to adjust the incident illumination; spread or reduce the beam spot-size to reduce intensity, or vice-versa.

Figure 4-3. The GIF tuning procedure options in the AUTOFILTER IMAGE RECORDING options.

4.4 Preparing a gain reference

At this point it is recommended to take a gain reference image. This is an image of a flat-field beam and shows the variation in gain between pixels. After you have prepared the gain reference this it will be used to correct variations in all later images (whether acquired in spectroscopy or imaging mode).

The detector at the end of the spectrometer consists of a CCD, two fiber optics and a scintillator. When these are all combined the final system has slightly different response to electrons at different locations within the CCD array. A single primary electron gives a slightly different signal on the CCD depending upon the location it is incident and hence the corresponding pixel. By taking an image of flat illumination any variation can be normalized. Normally this variation is less than 5%.
Figure 4-4. AUTOFILTER VIEW OPTIONS floating window.

To prepare a gain reference

- In TEM TECHNIQUE mode, set up a view from the PREVIEW view with the parameters shown above in Figure 4-4 (ALT click on the PREVIEW button to open the dialog). Start it viewing by selecting PREVIEW and then clicking on the IDLE/ACTIVE button to set it to ACTIVE.
- Adjust the beam to ensure even illumination on the detector.
- Ensure that there are no sample features visible in the field of view.
- In the CAMERA menu, select PREPARE GAIN REFERENCE. Make sure the target intensity and frames to average are set to 8000 and 10 respectively (see Figure 4-5 below).
- Follow any on-screen prompts during the automated gain-reference acquisition procedure.

Figure 4-5. The PREPARE GAIN REFERENCE window

If, at any later point, fixed pattern noise appears in the image or the detector is temperature cycled you need to repeat the process of obtaining a gain reference.

The selection of the parameters in the PREPARE GAIN REFERENCE dialog depends on the experiments that you intend to perform later. All later images are divided by the images taken at this point. If the noise in this gain reference is greater than the noise in a subsequent image then the noise in the gain reference will dominate the data acquired. As a simple rule the total number of counts in the gain reference should be more than in the final data. If you intend to take 10 frames averaged later then make sure the gain reference has 10 frames averaged. The target intensity is set at a level below the saturation so that the detector is unlikely to saturate in a single frame. On the GIF
saturation varies from CCD to CCD but is normally between 11,000 and 14,000 counts so selecting 8,000-10,000 counts is safe.

4.4 Focusing the system in spectroscopy mode

Now the basic GIF alignments have been performed, we now want to focus the system in spectroscopy mode. To enter spectroscopy mode, click on the EELS button in the TECHNIQUES option group. A prompt will warn you to reduce the incident intensity if necessary; respond accordingly and click PROCEED.

Figure 4-6. The AUTOFILTER TECHNIQUE OPTIONS dialog

Select ALT + EELS to open the AUTOFILTER TECHNIQUE OPTIONS dialog, where the spectrometer dispersion (in eV/channel) and entrance aperture size can be determined (see Figure 4-1 above). For performing alignment select a high dispersion (that is, a low eV/channel value, such as 0.1 eV/channel) and the medium size aperture, then hit OK.

Figure 4-7. The SPECTROSCOPY SET-UP dialog.

You are now ready to view the zero-loss peak. On the ENERGY group of options on the AUTOFILTER palette, select ZERO LOSS to specify the zero-loss region as energy-loss region of interest. The best way to get a ‘live’ view of the spectrum is to use the TURBO VIEW mode, as indicated in Figure 4-7 above. Clicking ALT + TURBO will display the
SPECTROSCOPY SET-UP dialog where acquisition parameters for all the VIEW modes can be set; ensure your set-up matches the values below for the TURBO mode, and hit OK. You can now click on the IDLE/ACTIVE button to start the acquisition and view the spectrum. The viewed unaligned spectrum should look something like the spectrum shown in Figure 4-8.

**Figure 4-8.** Zero-loss before focusing

![Figure 4-8](image)

If the displayed spectrum is red or yellow, and/or looks like the spectrum in Figure 4-9 below then the incident intensity is too high; reduce this by spreading the beam or by reducing the spot size. If the peak appears to be dominated by noise then increase the incident intensity.

**Figure 4-9.** Saturated zero-loss peak (ZLP)

![Figure 4-9](image)

The acquisition time can be varied to increase or decrease the CCD exposure and hence the recorded intensity. The up arrow key doubles the exposure while the down key halves
it. Beware, though, too slow an exposure will make it difficult to focus the peak, and too fast can introduce unwanted artifacts as a consequence of the finite beam blanking time; between 0.05 and 0.5 s is a suitable range.

If the peak is not visible at all, then it could be out of the field of view since we aligned the zero-loss peak in imaging-mode, not spectroscopy-mode, and the two can be misaligned. Alternatively, the zero-loss peak might be visible but not well centered on the 0 eV channel. In either case we wish to shift the spectrum to align the zero-loss peak:

**To align the zero-loss peak in spectroscopy mode**

- Clicking **ALT + ALIGN ZLP** to launch the **AUTOFILTER IMAGE RECORDING OPTIONS** dialog (Figure 4-10). Select fixed step as the **MANUAL** step method, and a fixed step size appropriate to the amount you wish to shift the spectrum (10’s of eV to search for the zero-loss, or a small value for fine adjustment). Hit OK.
- Clicking on the **ALIGN ZLP** button in spectroscopy mode will shift the spectrum by the specified fixed-step size in the positive direction. The same action with **SHIFT** or **CTRL** pressed will perform the shift in the opposite direction.
- Shift the spectrum, altering the fixed step size if necessary, until the spectrum is aligned at 0 eV.
- If the zero-peak still cannot be found, refer to “Finding the beam by hand” in Appendix A.

**Figure 4-10.** The **AUTOFILTER IMAGE RECORDING OPTIONS** dialog.

The next step is to focus the zero-loss peak, which is performed through the FilterControl software. Open and position the FilterControl window such that you can still view the TURBO spectrum acquisition in DigitalMicrograph and alter the FilterControl settings simultaneously.

If the adjustments menu as shown in **Figure 4-11** below is not open then select **ADJUSTMENT...** under the **WINDOWS** menu. To change a value, double-click on the spectrum property of interest to launch a value adjustment dialog as shown below for **FOCUS X**. Moving the mouse left or right will alter the value, and the adjustment strength can be incremented using the + and – keys. Left-click the mouse to set the adjusted
value, or to exit the dialog and restore the original value hit ESC. Now focus the zero-loss peak using the TURBO spectrum view following the procedure below:

**Figure 4-11.** FilterControl with the ADJUSTMENTS window open; FOCUS X is being adjusted.

![FilterControl with ADJUSTMENTS window open](image1.jpg)

**To focus the zero-loss peak**

- Select the small (0.6 mm) entrance aperture
- Make the spectrum as sharp as possible by adjusting FX and FY.
- Select the medium (2 mm) entrance aperture
- Focus with SX and SY
- Go to the aperture you intend to work with.
- Refocus FX and FY.
- Realign the zero-loss peak with the 0 eV channel.

**Figure 4-12.** Zero-loss peak after focusing

![Zero-loss peak after focusing](image2.jpg)
When correctly aligned, the zero-loss peak should be sharp and symmetrical as shown in Figure 4-12 above. If a shoulder remains on the low energy-side of the peak, the TURBO view exposure is most likely too fast; adjust accordingly and repeat the focus procedure. If during focusing the system the zero-loss peak looks saturated or is yellow/red, the incident intensity is too high and the detector is saturated. Reduce the beam current and repeat the procedure.

4.5 Acquiring a spectrum

In order to acquire a spectrum we must first locate a suitable area of interest on the sample. You may wish to do this in normal TEM mode (i.e. using the TEM phosphor screen) or using the GIF CCD / TV-rate camera by selecting TEM TECHNIQUE in the AUTOFILTER palette. To obtain good EELS data it is important that the area you look at conforms to the following criteria.

It should be:

a) Thin
b) Clean
c) Of scientific interest.

In Figure 4-13 below a typical low magnification view of a BN-C sample is shown.

Figure 4-13 Low magnification view of BN-C film.

Select a thin area for analysis from a smaller grain rather than a collection of grains, as illustrated in the higher magnification image in Figure 4-14 below.
Reduce the spot size and condenser aperture to typical values for microanalysis (refer to your TEM user’s guide for details). Put the beam on the edge of a small grain. For this tutorial do not focus it to a very small spot as this tends to contaminate some samples. See the section on contamination in the Additional Tips section at the end of this chapter. Select the diffraction mode on the TEM and set the camera length to about between 5 and 20cm. Make sure the center spot (disc) is in the center or the screen. Ensure EELS is the selected TECHNIQUE in the AUTOFILTER palette. Select 0.1 eV/ch dispersion, 0.05 s acquisition time and the medium-size entrance aperture in the AUTOFILTER TECHNIQUE OPTION dialog (ALT + EELS). Lift the screen, click TURBO VIEW and then set the IDLE/ACTIVE button to ACTIVE to start acquisition. Your spectrum should look like the one below.

Figure 4-15. Typical first spectrum.

Congratulations on your FIRST spectrum!
Adjust the beam intensity or acquisition time if the spectrum is saturated or too noisy. Now move the TEM’s diffraction shift settings a small amount to optimize the signal entering the detector. On most microscopes changing the magnification or mode causes small changes in the focus and apparent energy of the beam so repeating the alignment of the zero-loss and focusing should improve the spectrum. Once you are happy with the acquired spectrum and the turbo-acquisition parameters, try acquiring a spectrum in the CORRECTED VIEW mode. This acquisition mode is activated and set-up in a similar way to that described for the TURBO mode. Ensure similar set-up parameters are used as in Figure 4-16 below.

**Figure 4-16.** The CORRECTED VIEW set-up parameters in the SPECTROSCOPY SETUP dialog.

You may have noticed that the CORRECTED mode is set-up by default with only $1 \times$ hardware (on chip) binning, while the TURBO mode used $16 \times$. This increases the effective dynamic range of the CORRECTED view acquisitions (at the expense of read-out time), increasing the maximum intensity before saturation at a given exposure, allowing spectra to be acquired with more counts and hence increased signal-to-noise.

### 4.6 More detailed analysis of a spectrum region

Now we have acquired a spectrum it is a good idea to think about what your aim is. For this exercise we will take a spectrum over a wide energy range, a narrow energy range and over a large range of intensities. The start energy is set using the ENERGY LOSS value in the ENERGY options group of the AUTOFILTER palette, and the range is set by the dispersion. For example, a start-energy of 0eV and dispersion of 0.5 eV/ch would give you -50eV to 462eV (the spectrum starts at -50eV since there is a 100 channel offset applied to accommodate the zero-loss tails). You will start by taking a spectrum from about 100eV to over 500eV.

*The reason for looking from 100 to 500eV is that we are interested in the core loss edges and although the low loss region may contain some edges, these are hard to interpret. A second reason is the low loss region is very bright so, if we take the spectrum with the zero loss and this is not saturated, then the intensity in the spectrum at 500eV is very*
small (this will be demonstrated in our last example). By starting and 50eV or 100eV we avoid the brightest part of the spectrum and then can optimize the exposure on the part of the spectrum we are actually interested in. If we want to take a spectrum to include the low loss as well we can try optimizing the parameters to do this (refer to the later exercise on how to do this.).

Set the dispersion to 0.5 eV/ch and the start energy to 150 eV. To set the start energy, select CUSTOM in the ENERGY options group of the AUTOFILTER palette then click on the ENERGY LOSS value; enter 150 eV as the energy. Next set the CORRECTED VIEW mode to 0.1 second exposure and click the IDLE/ACTIVE button to start acquisition. The spectrum may look like this:

**Figure 4-17.** First B-N-C spectrum

![Image](image1.png)

If so, move the TEM’s diffraction shift setting a small amount to optimize the signal, as noted previously, until the spectrum should look like this:

**Figure 4-18.** Improved B-N-C spectrum.

![Image](image2.png)
Click ALT + ACQUIRE to initiate the AUTOFILTER SPECTRUM RECORDING OPTIONS dialog as shown in Figure 4-19 below. Enter the same values, check PROMPT EVERY TIME and hit OK.

**Figure 4-19.** The ACQUIRE preferences in the AUTOFILTER SPECTRUM RECORDING OPTIONS dialog.

Now click on ACQUIRE, specify 1 second acquisition and 3 frames, then press OK. The acquired spectrum should look something like this:

**Figure 4-20.** Acquired B-N-C spectrum

If the acquired spectrum is noisy or saturated (i.e. clipped at high counts, or red / yellow in color), adjust the incident beam intensity and/or the exposure time accordingly and reacquire. Three edges are visible starting at 190eV, 285eV and 400eV.

N.B. These edges can be identified using the EELS analysis software; please refer to the EELS Analysis User’s Guide for details on the identification and quantification of edges within the spectrum. The EELS analysis software should suggest the edges are boron K at 188eV, carbon K at 285eV and nitrogen K at 401eV. This is consistent with our a priori knowledge of the sample’s constituent elements.
To continue this exercise we will try to take a spectrum containing only the boron edge at higher spectral sampling. The boron edge is an interesting example as it contains plenty of near edge structure. To increase the spectral sampling we need to increase the dispersion; go to the AUTOFILTER TECHNIQUE OPTIONS dialog and select 0.1 eV/ch. The spatial resolution of the spectrometer is improved by using a smaller spectrometer entrance aperture; however, this will also result in a reduction of intensity. Select the small aperture for this exercise. Before you start to acquire your data you may wish to check the zero-loss peak is still focused centered on the 0 eV channel; often changing dispersion can cause a small change in alignment. We wish to get as much of the post-edge spectral detail of the boron edge within the field of view of the detector. Additionally, it is also desirable to get a few tens of eV pre-edge background within the acquisition. The selected dispersion gives us about 100eV range across the detector; select an offset energy of 180 eV to give a range from 170-270 eV (again, the 100 channel offset results in a start energy of 170 eV and not 180 eV). Using the CORRECTED view mode, adjust the beam intensity and signal integration time to maximize the spectrum counts without saturating the detector. Once you have determined the optimal acquisition parameters, set up the CUMULATIVE acquisition with the same parameters as the CORRECTED view; this is done through the SPECTROSCOPY SETUP dialog, initiated by ALT-clicking within the View. Once set up, select the CUMULATIVE view followed by the IDLE/ACTIVE button to start acquisition. The CUMULATIVE view will sum successive acquisitions, resulting in a spectrum with an overall intensity that can exceed the saturation level for a single acquisition. When you are happy with your spectrum, halt the acquisition by selecting the IDLE/ACTIVE button.

The last spectrum that we will try to acquire is of the whole spectrum from the zero loss to higher than the nitrogen edge. The zero loss is very bright compared to the edges so it is important to maximize the dynamic range of the system. Set a start energy of 0eV and a dispersion of 0.5eV/ch. Make sure the X and Y hardware (on-chip) binning is set to 1 for the CORRECTED and CUMULATIVE views in the SPECTROSCOPY SETUP dialog. Single binning gives the maximum dynamic range. Next set the CORRECTED exposure to the minimum value where the shutter speed does not cause deterioration of the spectrum; this is typically about 0.05s. Select CORRECTED view followed by the IDLE/ACTIVE button to observe the spectrum. The CCD detector is very sensitive so it is easy to saturate the detector with the zero-loss peak. If the spectrum is saturated, the spectrum appears red. Reduce the beam brightness on the TEM, either reducing the spot size, reducing the condenser aperture or reduce the emission. It may be necessary to use a combination of these steps. Once you have a spectrum that appears unsaturated, has returned to its normal color and is focused, select the CUMULATIVE view and set the same exposure used in the CORRECTED mode, and then click the IDLE/ACTIVE button to start the CUMULATIVE acquisition. Zooming into the region where the core-loss edges are will show them. Their signal should increase with additional acquisitions. Hit the IDLE/ACTIVE button to halt the CUMULATIVE view once you can see no visible improvement in the edge signal with additional acquisitions.
4.7 Optimizing the spectrum

You now have some spectra from your sample. However, you may wish to improve the quality further by improving the resolution of the spectral details and reducing noise. To reduce noise we need to increase the number of counts. To make the spectrum sharper we need to make sure the focus is perfect, the entrance aperture is suitable and the TEM is setup for good energy resolution. Unfortunately two of these parameters work against each other. When the TEM has a lot of beam current the energy resolution gets worse. At installation you engineer should have made a chart of resolution vs. various parameters on the TEM, and of resolution vs. aperture size. This allows you to understand how these parameters affect the resolution.

A number of simple things can be done to increase the signal to noise ration of the spectrum. Try the following and see the effects:

- Move the diffraction pattern a small amount using the projection shift or diffraction shift to maximize the intensity. This just makes sure the beam is aligned to the exact center of the spectrometer.
- Increase the integration time. A higher number of counts make better signal to noise ratio. Beyond a certain point you cannot increase the integration time without the detector saturating. After this point you can integrate over multiple frames.
- The noise in a spectrum is a combination of the noise in the gain reference and the noise in the image. This means you need as many counts or more in the gain reference as in the spectrum. Simply if you try to average 10 frames in your spectrum you need to make sure the gain reference was taken with 10 frames.
- The spectrum “looks” brighter at lower dispersions, so if you wish to see a small edge try the lowest dispersion you can. At 0.05eV/ch the signal is spread over 10x number of pixels than at 0.5eV/ch. If the primary aim is to see a trace amount of a material then use a lower dispersion.

- The TEM’s camera length affects the final signal. This and the entrance aperture give the collection angle to the spectrometer:

  \[
  \text{collection angle (mrad)} = \frac{\text{entrance aperture radius (mm)}}{\text{camera length at aperture (m)}}
  \]

  When the beam is focused on the TEM sample a CBED pattern is formed with discs rather than spots in the diffraction plane. If the camera length is very long so that is excludes some of the bright field disc from the spectrometer most of the signal is rejected. To collect the maximum inelastic signal with minimum background contribution, a rule of thumb is to make the collection angle 2 or 3 times the energy-loss’ characteristic angle.
For a typical TEM the characteristic scattering semi-angle $\theta_c$ is about (see Figure 4-21):

$$\theta_c \text{ (mrads)} \approx \frac{E \text{ (eV)}}{2E_0 \text{ (keV)}},$$

Increasing the collection angle over 2-3 times $\theta_c$ just increases the background contribution. For a more detailed discussion about scattering angle refer to the textbook by Egerton in referred to in Section 1. For example, for the silicon L$_{23}$ edge at 99 eV a 1 mrad collection angle is more than enough; however, for the K edge at 1836 eV a 10 to 15 mrad angle is more suitable.

5 Additional tips

5.1 Contamination

On many samples, when the beam is condensed on the specimen a small dark spot will begin to appear at the beam position. This is particularly noticeable in FEG TEM’s as the beam brightness is very much higher. The dark spot is carbon formed from the intense beam cracking residual organic matter on the specimen surface. In comparison to the majority of elements, carbon is light so it gives little contrast in the TEM. When it is very thick it becomes electron opaque. If you get a build up of carbon at your probe position then the EELS spectrum will deteriorate. Spending effort removing the hydrocarbon contamination sources will give better results when working on the microscope.

Three common techniques to avoid hydrocarbon contamination in the TEM are:
• Heat the sample on the surface of a light bulb to remove any surface hydrocarbons before putting it in the TEM.
• Use a liquid nitrogen holder to bind the hydrocarbons in place.
• Use a plasma-cleaner to remove the hydrocarbons before putting it into the TEM.

5.2 Image and diffraction coupling

The TEM has two modes of operation:
• image mode and
• diffraction mode

When the TEM is in diffraction mode, the spectrometer is focused on a crossover located at the projection lens; this is actually a very small image. In this mode of operation the spectrometer is said to be, rather confusingly, “image-coupled” which means the TEM is in diffraction mode. Conversely, when the TEM is in imaging mode there is a diffraction pattern at the crossover and it is termed as being “diffraction coupled”.

Performing spectroscopy in a diffraction-coupled configuration can be VERY misleading and should be avoided. It is tempting to put an image at the entrance of the spectrometer and select a particle with the entrance aperture. This will often get the WRONG result. What you are doing is putting the zero-loss image at the entrance aperture. The image at the energy-loss of interest will be very far out of focus and so will partially (or even completely) miss the aperture. Whenever trying to take a spectrum from a small area, isolate the area of interest using a focused probe; do not select an area with the entrance of the spectrometer. The only time using diffraction coupling is “safe” is when the TEM’s high-tension can be adjusted and the energy range of the spectrum is short. This is the situation used in energy-filtered imaging mode; the energy range is the slit size, normally below 50eV, and the loss image is kept in focus by increasing the TEM high tension by the corresponding value.
Appendix A

Finding the beam by hand

- Insert the TV-rate camera and put the spectrometer into EELS mode.
- Ensure the largest entrance aperture is inserted and the energy-selecting slit is retracted.
- Adjust the value of the lens marked ADJUST, found in the IMAGE FILTER CONTROL floating window, from $-2000$ to $+2000$ eV. The beam should be in this range; typically it is within a few hundred volts.
- When the spectrum is found, position the zero-loss peak to the left hand side of the monitor and select NULL ENERGY ADJUST to zero the ADJUST value.