# Enfinium
# *Owner's Manual*

**Part Number: 976.MANUAL**
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**Gatan, Inc.**
5794 West Las Positas Blvd.
Pleasanton, CA 94588
Tel. (925) 463-0200
Fax. (925) 463-0204

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# Preface

## About this Manual

This owner's manual provides information about features and functions of the Enfinium family, along with procedures for setting up these systems for operation. Routine maintenance and service procedures are also included in this manual

The following typographical conventions are used for special comments:
**NOTE:** Used to highlight information directed at getting the best performance from the equipment.

The *Enfinium Product Family Owner's Manual* consists of the following chapters and appendices:

Chapter 1, "Overview," describes the Enfinium hardware and built-in software. The application software needed to operate the system is also described briefly. See the various EELS user guides installed with GMS for detailed software information.

Chapter 2, "Precautions and Warnings," describes important safety considerations for using the equipment, and should be read before using this equipment.

Chapter 3, "Theory of Operation," describes the concepts and principles underlying the use of the Enfinium.

Chapter 4, "Requirements and Specifications," provides detailed information about the Enfinium equipment and its operating specifications.

Chapter 5, "Installation," provides a brief description of the installation process. Installation of the Enfinium should not be performed by users. Contact a Gatan service representative to arrange for installation.

Chapter 6, "Getting Started," provides basic set-up and calibration instructions for first time use of the Enfinium.

Chapter 7, "Troubleshooting," lists typical problems that might occur and explains how to resolve them.

Chapter 8, "Care and Maintenance," provides procedures for routine maintenance to keep the Enfinium operating at peak efficiency.

Appendices

Appendix A "Enfinium Control palette access as a Power User," explains the additional controls available to a power user, similar to the controls that used to be available via FilterContol in earlier versions of GMS.

Appendix B “Enfinium ER HiVac Vacuum Pump palette (for HITACHI and NION dedicated STEMs),” explains the control of the camera low vacuum pump with high vacuum systems.

Appendix C “Baking an Enfinium on a HITACHI STEM,” explains the baking procedures for the high vacuum version on Hitachi dedicated STEMS.

Appendix D “References,” is useful additional reading

Appendix E “Glossary,” helps explain some of the abbreviations used in the manual

Appendix F “Size of Projector Crossover” explains how the S/TEM affects the resolution of the spectrometer.

Appendix G “Dispersion table,” helps understand how the effect of the prism changes with energy of the primary beam.

## Copyright and Trademarks



## Disclaimer



## X-Ray Disclaimer

The following product testing information is based solely on testing of sample products and does not constitute a guarantee or warranty concerning the performance of any particular product. It is provided solely for informational purposes.

Samples of this Gatan equipment (part numbers 976.2K and 977.2K) configured for 200 kV operation were tested on an electron microscope operating at an accelerating voltage of 200kV with electron beam current in the viewing chamber of 3 µA. The local dose rate at a distance of 0.1 m from the surface of the equipment did not exceed 1.0 µSv/h (micro Sievert per hour).

Additional samples of this Gatan equipment (part numbers 976.3K and 977.3K) configured for 300 kV operation were tested on an electron microscope operating at an accelerating voltage of 300 kV with electron beam current in the viewing chamber of 0.50 µA. The local dose rate at a distance of 0.1 m from the surface of the equipment did not exceed 1.0 µSv/h.

This information is provided with the understanding that all equipment should be fully tested and qualified by the end-user for each specific end-use application. Product performance and results are not guaranteed by Gatan. End-users bear all responsibility for the specific use of this equipment. Gatan does not enforce compliance with any laws or regulations governing the specific use of this equipment. It is the responsibility of the end-user to ensure that the end-user's specific use of any Gatan product does not violate any laws or regulations, including laws pertaining to occupational safety and radiation protection, or infringe on any patents or other third-party rights.

# Support

Gatan, Inc. provides technical support via phone, fax, and electronic mail. To reach Gatan technical support, contact the facility nearest you, or send electronic mail to service@gatan.com. Additional information is available on the company's web site, www.gatan.com.

## USA, Canada, and Latin America

Gatan, Inc.
Corporate Headquarters
5794 West Las Positas Blvd.
Pleasanton, CA 94588
Tel: (925) 224-7360
Toll-free: (888) 887-3377
Fax. (925) 463-0204
Contact: service@gatan.com

Gatan, Inc.
780 Commonwealth Drive
Warrendale, PA 15086
Tel: (724) 779-2552
Toll-free (888) 778-7933
Fax: (724) 776-3360
Contact: service@gatan.com

## Asia and Pacific Rim

Nippon Gatan
3F Sakurai building
2-8-19 Fukagawa, Koto-ku
Tokyo 135-0033, Japan
Tel: +81 3 5639 2772
Fax: +81 3 5639 2763
Contact: nippongatan@gatan.com

Gatan Singapore
10 Eunos Road 8
#12-06
Singapore Post Centre
Singapore 408600
Tel: (65) 6408 6230
Fax: (65) 6293 3307
Contact: gatansingapore@gatan.com

## Europe

Gatan GmbH, München Germany
Ingolstadterstr. 12
D-80807 München
Germany
Tel: +49 89 358084-0
Fax: +49 89 358084-77
Contact: GatanGmbH@gatan.com

Gatan UK
25 Nuffield Way
Abingdon, OX14 1RL
United Kingdom
Tel +44 1235 540160
Fax +44 1235 540169
Contact: ukinfo@gatan.com

Gatan France
ROPER Scientific SAS – Division GATAN
8, rue du Forez – CE 1702
ZI Petite Montagne Sud
91017 EVRY Cedex
Tel : +33 1 34 94 44 07
Mobile : +33 1 69 11 03 69
Fax : + 33 1 64 97 19 67
Contact : gatanfrance@gatan.com

## Returns

If there is a need to return equipment to the factory, please call Gatan to obtain a Returned Merchandise Authorization Number (RMA #). This RMA number must appear on your shipping document, to help in tracking and to ensure that proper actions will be taken to repair or replace your equipment.

# 1. Overview

The Enfinium™ EELS spectrometer series applies the advanced features of the GIF Quantum™, Gatan's 4th and latest generation of post-column energy filters, to our dedicated EELS product line. Dodecapole-based electron optics combined with a blazingly fast CCD camera system yields a spectrometer that defines the new state-of-the-art in the capture of highly detailed EELS data sets with maximum throughput.

Gatan's patented sensor readout architecture supports ultra-fast spectroscopy recording on the CCD sensor. When used with advanced options such as a 1 µs electrostatic shutter, sub-ms readout times, high-speed 1000 spectra per second EELS acquisition is possible!

Aberration correction up to 5th order allows the use of a 5 mm entrance aperture (for ER model) for energy loss spectroscopy, supporting the use of high collection angles while preserving energy resolution.

The Enfinium™ comes in several models to optimally couple to the broad range of TEM configurations and applications available today. Whether you have a work-horse $LaB_6$ instrument, an extreme brightness aberration corrected STEM for probing interfaces at the atomic level, or a monochromated system for observing energy transitions at the meV level, there is an Enfinium™ spectrometer to fit your needs and all those in between. Advanced options including the 1 µs electrostatic shutter, high-speed camera, the 2 keV fast spectrum offset module, and DualEELS™ hardware and software, allow each Enfinium model to be tailored to the applications at hand.

## 1.1 Hardware

The Enfinium™ series EELS system is designed for optimal performance for beam energies from 60 keV up to 300 keV and can be attached to the bottom flange of nearly any modern TEM allowing outstanding energy-loss spectral acquisition. The post-column design provides maximum flexibility in the choice of electron beam source (W, $LaB_6$, Schottky, cold-FEG, or monochromated) and optics (objective lens, correctors, TEM and STEM mode). Furthermore, Gatan's exclusive quadrupole projection optics ensure the spectra are always aligned with the detector, can be captured with a broad range of dispersions, and are optimally projected to a rectangular area of the CCD detector to guarantee the best dynamic range, sensitivity and readout speed possible.

The electron optics of the Enfinium™ system fully corrects focusing aberrations through third order, allowing the use of a 3 mm entrance aperture. The ER model also corrects the fourth and fifth order aberrations permitting the use of a large 5 mm aperture, yielding excellent collection angle and high efficiency. These advancements in aberration correction and new implementations of Gatan's patented tuning algorithms, allow the Enfinium™ to achieve the most accurate spectrometer alignment of any commercial spectrometer. Spectrometer performance is further optimized by automatically storing fine-tuned settings for each major mode of the TEM imaging column ensuring peak performance under all operating conditions.

The Enfinium™ series EELS systems include an advanced version UltraScanXP 1000 FT (Frame Transfer) imaging system optimized for spectroscopy. Capable of running at both 1MHz and 10MHz, as well as the all new pipeline spectrum mode, this single camera system provides both high quality single spectra, and the speed and duty cycle of a real-time viewing detector with up to 1000 spectra per second EELS performance.

### 1.1.1 Major components

The major components of the Enfinium are shown in figure 1.

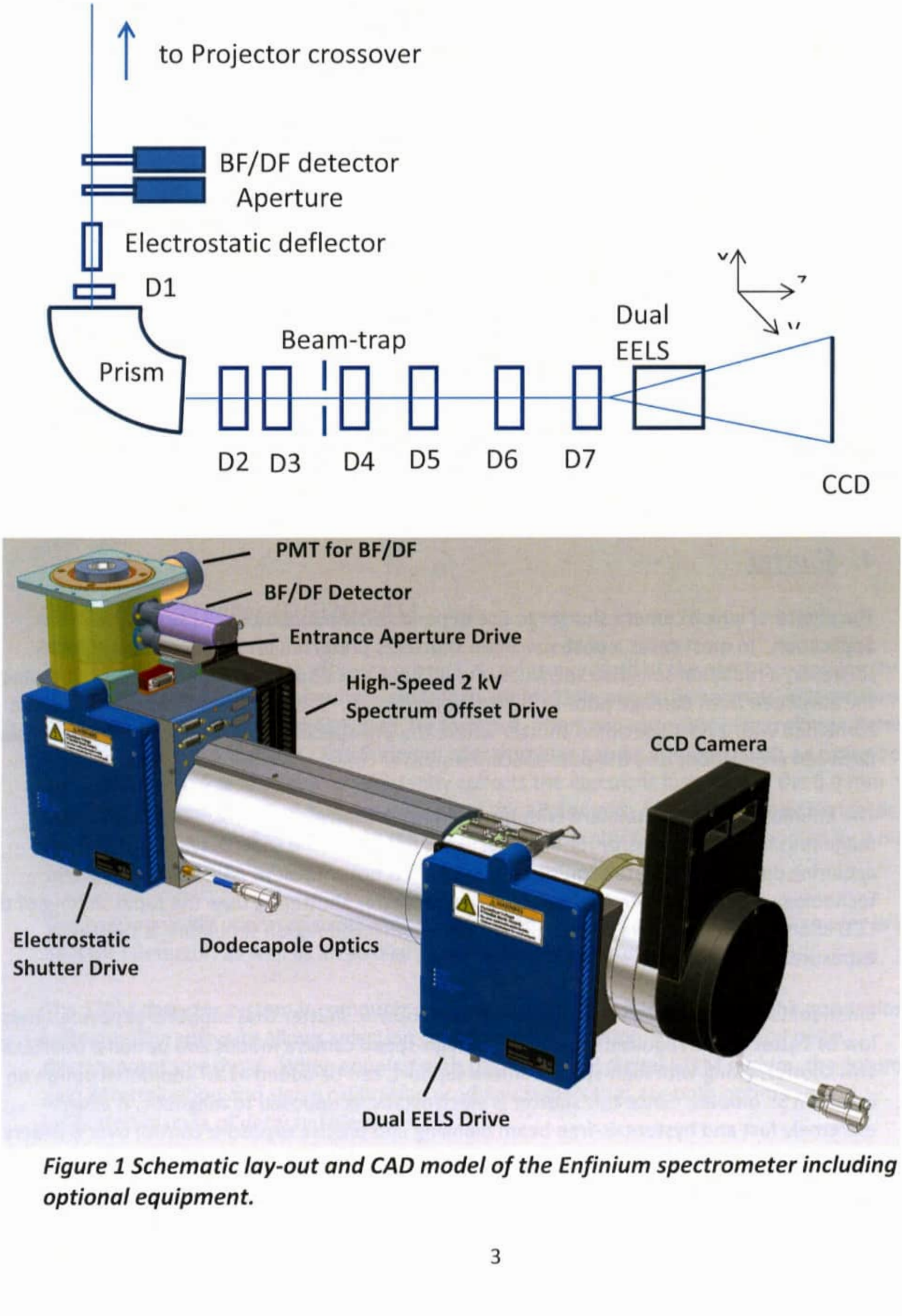



***Figure 1 Schematic lay-out and CAD model of the Enfinium spectrometer including optional equipment.***

## 1. Entrance aperture

At the entrance to the Enfinium ER (977 model), there is a pneumatically controlled, 2 position aperture rod containing round apertures. The two aperture positions are denoted as 2.5 mm and 5 mm, and typically used for defining the collection angle and to limit spectrum aberrations. The Enfinium SE (976 model) is equipped with a fixed 3 mm aperture.

## 2. Magnetic Prism

A key element of the Enfinium is its 90-degree gradient sector magnet, which bends the primary beam over a 7.5 cm radius and thereby disperses it in energy. The prism has inclined pole faces that give it additional first order focusing properties.

## 3. Dodecapoles

The Enfinium uses 7 dodecapole (12 pole) lenses rather than a series of individual multipoles featured in earlier generation systems. The poles of the dodecapoles are individually excited in order to generate any combination of dipole, quadrupole, sextupole, octupole, decapole, and dodecapole lens, enabling control of optical properties up to 5th order.

## 4. Shutter

The choice of which camera shutter to use depends on the minimum requirements of the application. In most cases a post-specimen shutter is preferred for better sample stability. However, a radiation sensitive specimen should have a pre-specimen beam blanker to protect the specimen from damage prior to capturing the image. This pre-specimen blanker is often combined with a post-specimen shutter where the pre-specimen blanker protects the sample in between acquisitions and the post-specimen shutter controls the exposure.

The Enfinium SE comes standard with frame transfer exposure control. Here, an internal, magnetic shutter is used only for blanking beam to protect the camera when not actively acquiring data. During data acquisition, the beam is not shutter at all and frame transfer technology is used to control exposure. Frame transfer shuttering uses the rapid shifting of the CCD charge from the active region of the CCD to a masked region simulating a shuttered exposure.

Enfinium ER models come with an internal electrostatic shutter that supports exposure times as low as 1 μsec and is required for support of high-speed camera modes and optional DualEELS™. This shutter, along with high-speed camera support, can be added as an additional option on Enfinium SE models. Since this shutter is electrostatic as opposed to magnetic, it allows extremely fast and hysteresis-free beam blanking and precise exposure control over 8-orders of magnitude

## 5. Drift tube spectrum offset

The Enfinium, like earlier post-column filters, uses an electrically isolated drift tube through the magnetic prism to offset the energy-loss spectrum by 1 kV. An optional high-speed, 2 kV spectrum offset module is available for the Enfinium. This module supports a larger 2 kV offset range with a fast 10 μs switching to 100 mV accuracy and with better than 30 mV RMS noise. This option is particularly useful when running in DualEELS™ mode.

## 6. DualEELS™ (OPTIONAL)

The DualEELS mode of operation simultaneously acquires a core-loss spectrum and corresponding low-loss spectrum with independent exposure times. The DualEELS hardware includes an electrostatic non-dispersive deflector in front of the CCD camera that alternately deflects the spectrum perpendicular to the dispersive direction to opposite halves of the CCD camera. The voltage on the prism drift tube is changed between the individual exposures so that one spectrum contains the core-loss region of interest, and the other the low-loss spectrum. Careful orchestration of the timing of the drift tube, electrostatic non-dispersive deflector, and electrostatic shutter allows DualEELS to be performed up to 500 spectrum pairs per second. Since the low-loss intensity is typically several orders of magnitude greater than that of the core-loss, it is acquired at a fraction of the exposure time and hence incurs a negligible increase in the total exposure time per spectrum. When coupled with the high-speed 2 kV spectrum offset module, the chemical shift of edges up to 2 keV energy loss can be measured with sub-eV precision.

## 7. BF/DF detector (OPTIONAL)

STEM imaging can be a very efficient process allowing nearly all of the electrons passing through the sample to be acquired simultaneously through multiple acquisition signals. The synchronous collection of STEM-EELS and ADF data, for instance, offers accurate spatial correlation between spectral and image features. The Enfinium spectrometer can be equipped with an optimized annular dark-field (ADF) detector efficiently collects the electrons just outside the 5.0 mm spectroscopy aperture. The ADF detector has a scintillator with an effective inner diameter of 5.3 mm and an outer diameter of 12.0 mm. The light from the scintillator is coupled by a mirror to a PMT based amplifier chain.

The pneumatically operated scintillator drive rod also includes a 5.7 mm outer diameter bright-field (BF) detector, as well as an auxiliary position allowing future additions.

The STEM detector system is computer controlled and is fully integrated into the acquisition software. The software allows selection of the detector position, PMT gain, and pulse discriminator threshold. When coupled with the DigiScan II digital STEM system, the automatic gain determination and signal calibration produces signals that are both optimized and calibrated in units of detected electrons.

## 8. CCD Camera

The Enfinium uses a 4 port, 2k scientific grade CCD. The sensor readout electronics has both a 1 MHz and an optional 10 MHz read-out rate (per output). The 1 MHz mode is used for the highest quality data acquisition; the 10 MHz mode is used for searching, focusing, and high-speed spectrum acquisition, up to 1000 spectra per second.

To achieve the highest stability and speed, the camera's parallel and serial clock drivers and analog to digital converters are located in the camera head. Power and digital data are sent to and from the controller box via 7m cables allowing the controller to be sited away from the S/TEM. The data interface to the host computer is via industry standard FireWire 800.

### 1.1.2 Electronics

Supporting electronics hardware for the Enfinium consists of the GIB, GIB power supply, controller for the camera, high voltage electronics for the electrostatic shutter (if equipped), DualEELS (if equipped), and high speed 2kV spectrum offset (if equipped) .

#### Gatan Instrumentation BIN

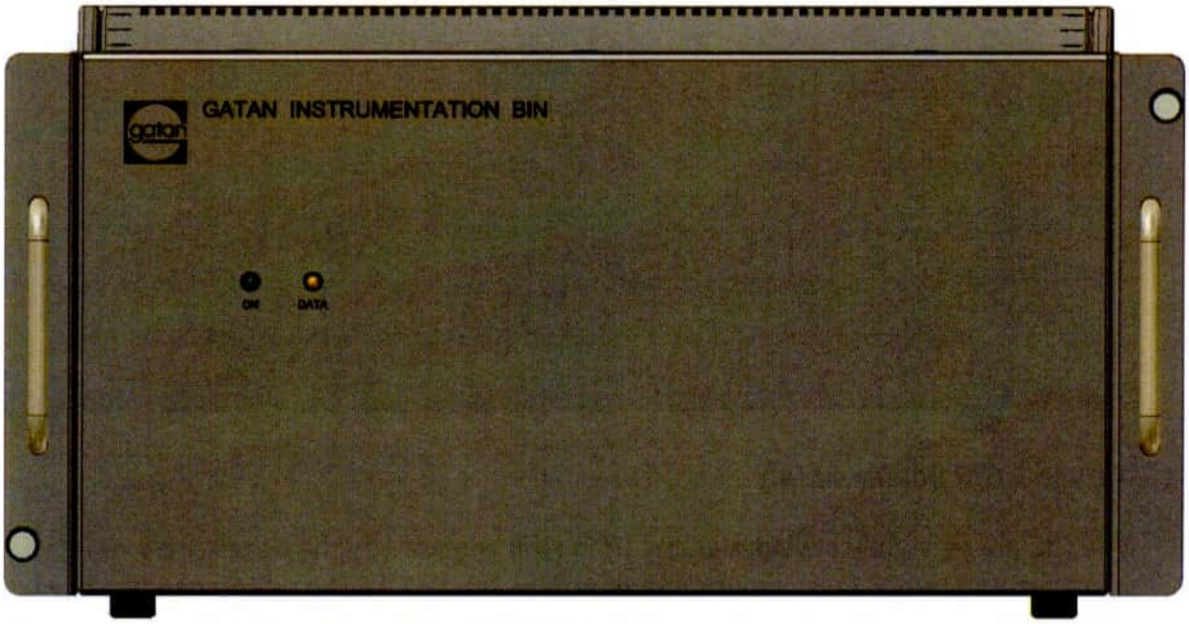



***Figure 2 Gatan Instrumentation Bin (GIB)***

The GIB houses the main spectrometer control electronics, which drives the Enfinium prism current, all its dodecapole lens currents, the entrance aperture and 1 kV drift tube supply. It is interfaced to the host computer via a USB link.

The dodecapoles use current drivers with 20-bit resolution and better than 1 bit noise and 1 ppm/°C stability. The thermal management (airflow and conduction) of the electronics was carefully simulated and verified experimentally to ensure the cooling necessary for optimal stability and reliability. The cables from the GIB to the Enfinium are 7 m long, allowing the electronics to be located in a separate equipment room if desired.
The green LED is lit when the power is ON. The amber data LED flashes when data is sent between the computer and the GIB.

## *GIB Power Supply*

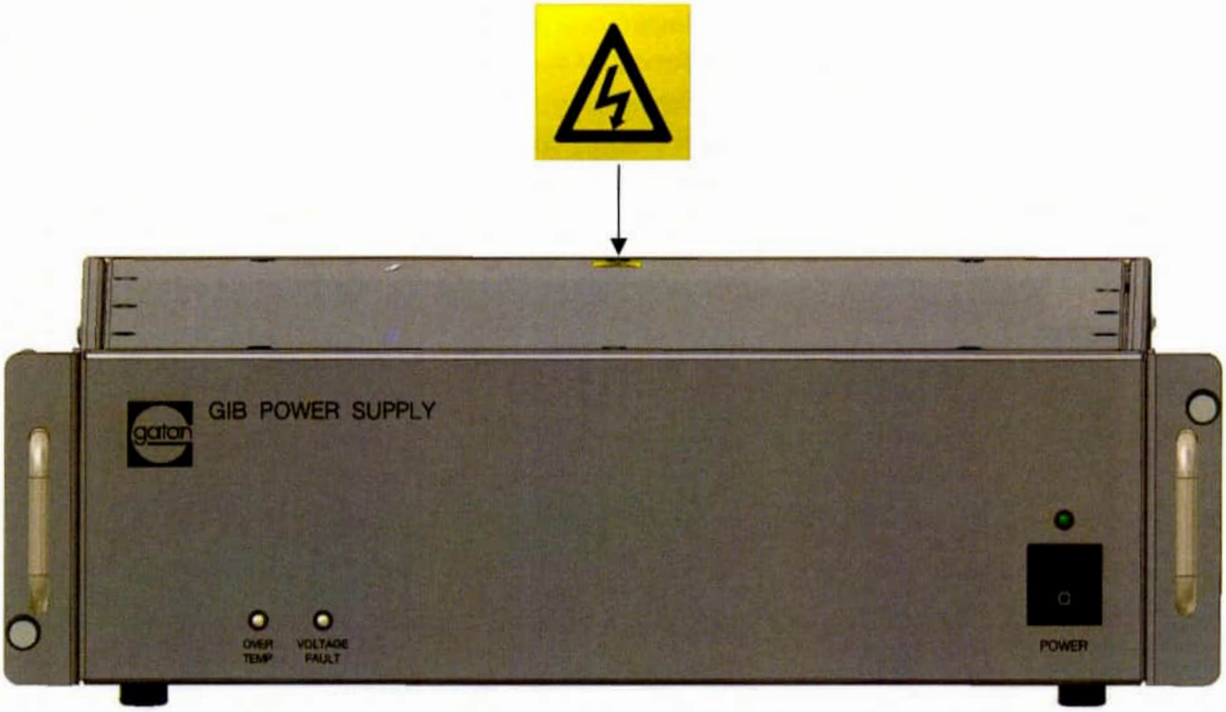



***Figure 3 GIB power supply***

The GIB power supply provides all the DC power required by the Gatan Instrumentation Bin and electrostatic shutter (optional for Enfinium SE). The OVER TEMP LED is lit when the power supply is overheating and has automatically shut down power. The VOLTAGE FAULT LED is lit when one of the voltages is out of specification. It may also indicate that some of the power rails have been shut down to protect the system.

## *High Voltage Electronics*

The high voltage electronics for the electrostatic shutter, fast drift tube, and DualEELS deflector are mounted on the spectrometer itself to minimize connection capacitance and maximize speed. They are setup and controlled over a USB interface.

"POWER" indicates external DC power is applied to the unit.

"HV ON" indicates the internal high-voltage is active.

"SHUTTER SIGNAL" indicates the state of the external shutter input (light is active high). In normal operation, the SHUTTER SIGNAL light indicates high-voltage is applied to the internal shutter or deflector plates of the Enfinium™.

"OVER TEMP" indicates a fault condition.


***Figure 4 Fast electrostatic shutter and DualEELS™ deflector module***


HIGH-SPEED SPECTRUM OFFSET MODULE
gatan
WARNING
Hazardous Voltage
Potential shock hazard.
No user seviceable parts inside.
Contact manufacturer for service/repair.

*Fi*

## *Camera Controller*


***Figure 6 Enfinium Camera Controller***

The camera controller provides all power and control signals for the UltraScan1000XP FT detector of the Enfinium. It also includes circuitry to drive the Peltier cooling system of the camera. Cooling reduces thermal counts or "dark current." This is especially important during longer exposures.

***OT*** **–** Controller Power Supply Over Temperature- lights when controller overheats. (Normally Off)

***VOLTS*** **–** Camera Head Power – When lit camera head power is on. (Normally on unless a fault condition exists)

***CNTLR*** **–** Controller Electronics Initialized – When lit indicates controller electronics have initialized properly. (Normally on unless a fault condition exists)

***CAMERA*** **–** Controller communication with the camera head – When lit indicates camera controller has initialized the camera head properly. (Normally on unless a fault condition exists)

***DATA*** **–** Firewire Activity – When flashing indicates activity over the PC's firewire interface.

***Shutter*** **–** Shutter Active – When lit indicates shutter output is active.

***TEC*** – Thermo-Electric Cooler Status – When lit green indicates that cooling system has reached temperature preset. Amber indicates near cooling preset. Red indicates cooling system has not reached temperature preset. When unlit, TEC power is off.

# 1.2 Software

The Enfinium is supported by Gatan Microscopy Suite (GMS) software versions 2.1.1 onwards (GMS 1.9.4 for some OEM systems). The software supporting the Enfinium for both operation and data acquisition has been extensively revised from previous generations. Automation of data acquisition plays a key part of the new software interface. Many of the key improvements have been enabled by the optional fast electrostatic shutter, since this provides highly linear exposure control with ample range to attenuate the full intensity range of the EELS spectrum. The key Enfinium software advancements are described in the data acquisition section 1.2.4.

The components of the Enfinium software system play various roles in its overall operation. During routine use, all the main control panels of the system are displayed within and accessed from the DigitalMicrograph (DM) program, the centerpiece of the GMS software system.

Normally, there is no need for users to be aware of all the individual modules and files. The GMS software license that comes with each Enfinium ensures that all are properly installed on the Enfinium host computer by the GMS Installer program. However, it may be helpful to understand how the various components relate to each other in the operation and use of the Enfinium. The following is a brief overview of the Enfinium software.

## *1.2.1 Software Functions*

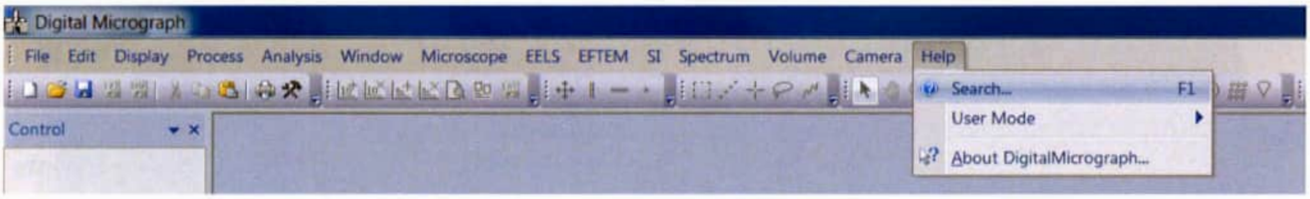
There are five different types of functions that the Enfinium software performs:

- Low-level instrument interface and control
- Generic data handling and display
- High-level instrument control and operation
- Data acquisition for different techniques
- Data analysis and processing for different techniques

The *Enfinium Owner's Manual* (this book) provides instructions for using both low-level and high-level instrument control software to set up, calibrate, and adjust the Enfinium optics (see Chapter 6, "Getting Started").

Operations involved in capturing, displaying, processing, and analyzing data from the Enfinium are described in detail in the corresponding user guides, tutorials, and on-line help files installed with each GMS installation. **The user guides and tutorials can be found in PDF form in the Gatan Documentation folder, accessible via a short-cut placed on the desktop.**


**Online help can be viewed by selecting Search from Help I Search sub menu in DigitalMicrograph or via a short-cut placed on the desktop.**





On systems installed by TEM vendors, the configuration of the software may vary. Please contact the TEM vendor for specific details.

### 1.2.2 Low-Level Instrument Interface and Control

At the lowest level in the Enfinium software set are the modules that handle the hardware-level communication with the various devices in the spectrometer system. These include the following components:

- **Drivers**
  These handle communication with the Gatan hardware.

- **DigitalMicrograph plug-ins**
  These special dynamic link libraries (.dll files) and compiled script packages (.gtk files) make the spectrometer and detector hardware accessible within the DM environment.

- **FilterControl plug-in**
  This plug-in plays a special role within the Enfinium software system. It represents the most direct software link to the electronics of the Gatan Instrumentation Bin (GIB). It also exposes a user interface, e.g.,***Enfinium SE Control, Enfinium ER Control, or Enfinium ER High-Vac Control***, that includes a number of high-level calibration procedures for the spectrometer electron optics. Normally, only Gatan service engineers directly access the FilterControl plug-in via Enfinium Control user interface because it gives a direct view of the current state of the spectrometer. This plug-in can be used for diagnostic purposes and troubleshooting. To see the window the DM User

mode needs to be set to "Power user". Find this under the "Help" menu, see the previous figure.

- **Generic Data Handling and Display**
  Apart from instrumentation control, a complete system for analytical TEM based on EELS must also include a data handling module that can manage a variety of data types, such as energy-loss spectra, spectrum images, or STEM spectrum images. This "data system" module must provide facilities for viewing and manipulating such data sets. Within the Gatan Microscopy Suite (GMS), this data system role is handled by DigitalMicrograph. For a thorough discussion of the features and user interface of DigitalMicrograph, please refer to the *DigitalMicrograph User's Guide*, included in the Documentation folder created with your GMS installation. Please also see the on-line help available via the Search... command in the DigitalMicrograph Help menu.

### 1.2.3 High-Level Instrument Control and Operation

- **DigitalMicrograph**
  DigitalMicrograph's user interface panels provide uniform access to the overall TEM instrument system. For the Enfinium, several such DM control palettes consolidate and simplify control of the spectrometer electron optics and its detector.

- **AutoPEELS**
  The high-level operation software also includes an automation module, AutoPEELS. AutoPEELS is unique to Gatan's GIF Quantum and Enfinium product line. At the click of a button AutoPEELS measures and tunes the electron optical performance of the spectrometer system. This powerful automated tuning system allows the Enfinium to be operated with ease by users with any EELS experience level.

- **Instrument-specific Operations**
  In some types of TEM-integrated Enfinium installations, control panels for the Enfinium are duplicated by the host TEM software. In this case, the user may operate the Enfinium via either user interface. Please refer also to the operating instructions of the host TEM instrument for the details of Enfinium operation via the TEM instrument interface.

### 1.2.4 Technique-Specific Data Acquisition

A key aspect of EELS data sets is that the extraction of important specimen information often involves several correlated data items. In other words, a complete EELS data set often involves several images or spectra taken under well-defined and recorded experimental conditions. Simple readouts of individual images or spectra are often not sufficient. In order to streamline the collection of EELS data sets, the Enfinium software includes data acquisition panels with buttons that trigger automated data collection sequences. These sequences result in high-quality spectra and extended energy ranges with fully automated exposure control

### 1.2.5 *Technique-Specific Data Processing and Analysis*

EELS data sets require further processing in order to reveal the information about the TEM specimen from which they were taken. Data processing and analysis modules in the Enfinium software allow you to extract this specimen information.

The principle component of the Enfinium data analysis suite is Gatan's EELS Analysis package, which provides a variety of quantitative analysis routines for extracting specimen thickness, elemental composition, and dielectric response function from spectra and spectrum images. Another important component is the (optional) map extraction module, which can extract specimen thickness maps, elemental edge count maps, and jump ratio maps from STEM spectrum images.

# 2. Precautions and Warnings

Review the following precautions to avoid injury and prevent damage to this product, or any products to which it is connected. To avoid potential hazards, use the product only as specified. Read all safety information provided in the component product user manuals and understand the precautions associated with safety symbols, written warnings, and cautions before accessing parts or locations within the unit. Save this document for future reference. Follow all warnings and instructions marked on the equipment. Ensure that the voltage and frequency of your power source matches the voltage and frequency inscribed on the equipment's electrical rating label. Never push objects of any kind through the openings in the equipment. Dangerous voltages may be present. Conductive foreign objects could produce a short circuit that could cause fire, electrical shock, or damage your equipment.

## 2.1 Approval and Safety Information

This chapter presents agency approval and certification information for the Enfinium. It also provides a summary of the safety recommendations throughout this manual. Gatan, Inc., recommends following all safety precautions to prevent harm to yourself or the equipment. Please follow all warnings marked on the equipment as well.

**CE Certification**
The product(s) described in this manual meet(s) the intent of:

**2004/108/EC EMC Directive**
**2006/95/EC Low Voltage Directive**
**2002/95/EC Restriction of Hazardous Substances (ROHS) Directive**
**2002/96/EC Waste Electrical and Electronic Equipment (WEEE) Directive**

**This product has been designed and tested in accordance with the 96/29/Euratom Directive for X-ray safety.**

**Compliance with RoHS and WEEE Directives**
As a company that has always been firmly committed to environmentally responsible practices, Gatan, Inc., is in complete accord with the content and spirit of Directive 2002/96/EC of the European Parliament and of the Council of 27 January 2003 on "Waste Electrical and Electronic Equipment" (commonly referred to as the WEEE Directive). The WEEE Directive applies to all products placed on the controlled market after 13 August 2005.
At present, any Gatan, Inc. products sold within the European Union may be returned for proper disposal. We have placed great emphasis on creating a truly easy-to-use return process for our many customers around the world. To return a product for disposal, customers simply need to contact our customer service department to obtain an RMA number authorizing the return.

We are pleased that our current disposal process is a relatively streamlined one. Of course, we are still busy evaluating other options that will further reduce the transport overhead of recycled products.

The Declaration of Conformance is available from Gatan, Inc., or from your authorized distributor. A copy is attached at the end of this manual.

Gatan, Inc., 5794 W. Las Positas Blvd., Pleasanton, CA 94588 USA
Tel. +1 (925) 224-7360
Toll Free: +1 888-887-3377
Fax. +1 (925) 463-0204
Contact: service @gatan.com
Customer Service - http://www.gatan.com/company/service/

# 2.2 Handling and Setup Precautions

The Enfinium should only be removed or installed on a TEM by Gatan-trained personnel using the supplied jack or lifting system.

## 2.2.1 Connection to the Power

Prior to plugging the unit in, read all Safety Precautions contained within this manual and ensure the power switch on the back of the unit has been turned off ("I" refers to on, and "O" refers to off.) Apply all applicable connections detailed below.
The system is supplied with a protected ground power strip, which must be used with all equipment (power supply, camera controller, computer and monitor). The ground on this power strip must attach directly to the TEM's main grounding point.
Use only the power cord provided with this product and proper electrical power as specified on the equipment's rating label.
Ensure the outlet is equipped with a proper ground pin. Should there be any questions as to whether this is the case, contact a licensed electrician.
Turn the "On/Off" switches to the "ON" position. Power is applied to units and the front panels will now have green power indicator lights on. To disconnect the unit, simply turn the "On/Off" switches to the "Off" position. If the unit is going to be moved or stored, simply remove the line cord from wall outlet.
All Enfinium electronics require an unrestricted air supply for proper ventilation. Care should be taken not to obstruct vents on this equipment.

## 2.2.2 Instruction for Protective Earth

The main protective earth connection for this system is made through the ground pin of the mains AC cord that plugs into the GIB power supply unit. Never operate this without proper connection to protective earth. Mains AC and ground connection to the facility should always be inspected by a licensed Electrician.

## 2.3 Operating Precautions

You must be aware of safety when you install and use this system. This guide provides various procedures that require careful attention to precautions. The following symbols appear in this document, as referenced from ANSI Z535.6.

⚠ This is the safety alert symbol. It is used to alert you to potential personal injury hazards. Obey all safety messages that follow this symbol to avoid possible injury or death.

The meaning of the different signal words are as follows:

DANGER indicates a hazardous situation which, if not avoided, will result in death or serious injury.

WARNING indicates a hazardous situation which, if not avoided, could result in in death or serious injury.

CAUTION, used with the safety alert symbol, indicates a hazardous situation which, if not avoided, could result in minor or moderate injury.

NOTICE is used to address practices not related to personal injury.

⚠ DANGER

Disconnect power before servicing or replacing fuses and only use value specified on the product's rating label.

Do not operate without covers: To avoid electric shock or fire hazard, do not operate this product with any removed enclosure covers or panels.

Never use the equipment in a manner not specified as the protection provided by the equipment may be impaired.

To avoid the risk of electric shock: Do not operate in wet, damp, or condensing conditions. When supplying power to the system, always make connections to a grounded main. Always use a power cable with a grounded plug (third grounding pin).

No user serviceable parts. Do not remove cover. Only qualified, trained service personnel may repair and calibrate unit.

Never operate this system without proper connection to protective earth. Never disconnect this systems connection to protective earth.

⚠ WARNING

To avoid electrical hazards (heat, shock and/or fire hazard), do not make connections to terminals outside the range specified for that terminal. See the product user manual for correct connections.

To avoid injury, fire hazard, or explosion, do not operate this product in an explosive atmosphere.

Always position the equipment so as not to limit access to the mains disconnect at the rear of the electronic enclosures.

Some of the following precautions and warnings also appear elsewhere in this manual, at the time they are most likely to be encountered.

⚠ NOTICE

Using city water is potentially dangerous to the Enfinium, as some tap water contains dissolved salts that attack the filter's metal parts.

Do not leave a voltage on the drift tube when venting the TEM camera chamber.

Avoid using beam current/exposure time combinations that saturate the CCD detector.

Shutter settings should always be set to normally closed.

Never attach any magnetic objects to any part of the spectrometer. Some materials used in the spectrometer are magnetic to ensure good shielding from external stray fields. If magnetized with a strong magnetic field, these materials retain some magnetization even after the magnetic field has been removed. This residual magnetic field may cause permanent shifts in the image and spectra.

## 2.4 Warning Labels

The following warning labels are applied at various locations about the product. Their meanings are as follows:

Insert Ground Here

High voltage interior of GIB power supply – no user serviceable parts inside
High voltage interior of shutter – no user serviceable parts inside
High voltage interior of fast drift tube – no user serviceable parts inside
High voltage interior of detector – no user serviceable parts inside

Refer to Manual when you see this label

separate collection required for electrical and electronic equipment waste

# 3. Theory of Operations

## 3.1 The Electron Optics

In normal operation, the entrance aperture is used to define the collection angle and to limit spectral aberrations. The electrons enter the magnetic prism and are bent over approximately 90°. The exact bending angle of an electron depends on its energy; the greater the energy loss it has experienced, the larger the angle the electron will be bent through. Besides bending the electron beam and creating energy dispersion, the prism also has a focusing action and at some distance behind the prism, an energy-dispersed, focused image is formed of the TEM's projector lens crossover. This is the energy-loss spectrum.

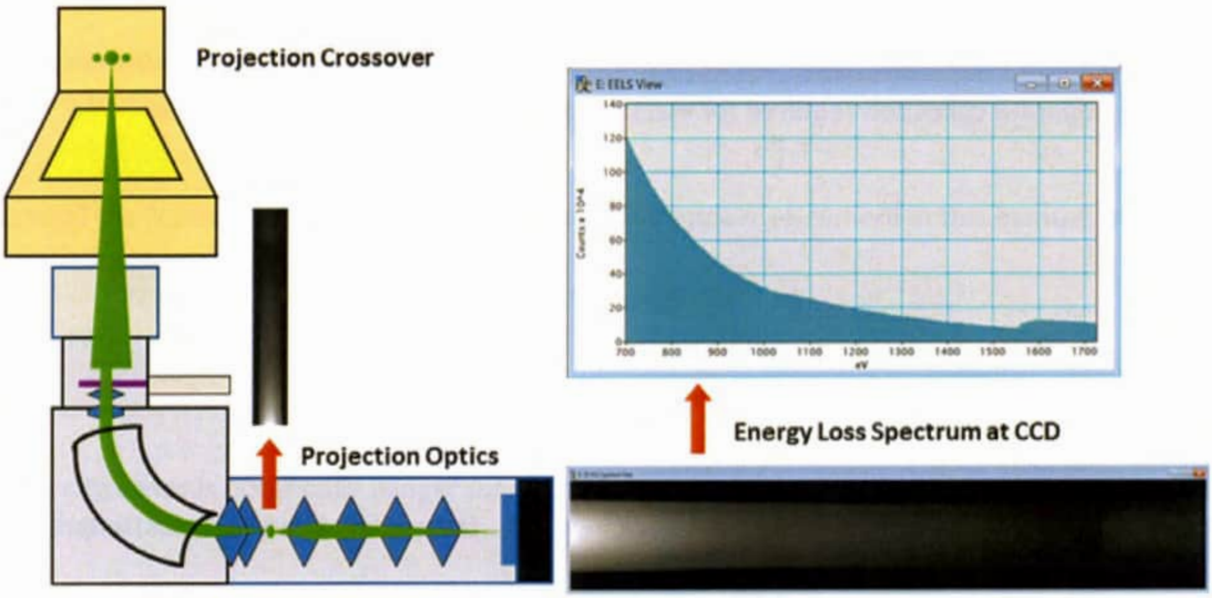



*Figure 7 Ray path diagram*

***The diagram shows the conjugate planes for the spectrum mode at the projector lens crossover; the beam-trap aperture and the CCD detector. The projector optics after the beam-trap aperture focuses the spectrum at the beam-trap aperture plane onto the detector.***

To optimize the low kV performance, the bending radius of the magnetic prism has been reduced to 75 mm, reducing the dispersion by 25%. The magnification at the beam-trap has been reduced, resulting in an energy dispersion at the beam trap of ~2.5 μm/eV at 200 kV, or

~7.6 μm/eV at 60 kV. The lower dispersion allows the Enfinium to have a 2 keV field of view at 200 kV and a 682 eV field of view at 60 kV (which can be doubled with DualEELS).

The prism is of the gradient type and uses inclined pole faces to focus simultaneously in the x and y directions. The entrance and exit faces are straight and the Enfinium relies fully on the optics external to the prism to correct the various important spectrum aberrations.

The dodecapole locations and first order trajectories are chosen such that sufficient range is available on each dodecapole to accommodate the sum of the multipole moments necessary to control the first order properties (focus, magnification, energy dispersion), as well as correct the various aberrations. See reference [14] for further details.

Since each dodecapole co-locates previously separate multipole elements on a single magnetic yoke, the alignment is much improved. The magnetic yoke is designed such that it stays well below magnetic saturation at the maximum possible excitation (i.e. current through the coil).

One dodecapole in front and two dodecapoles located after the magnetic prism project the magnified, focused spectrum onto the beam-trap aperture plane, correct all important 2$^{nd}$ and 3$^{rd}$ order aberrations, and, for the Enfinium ER, minimize the important 4th and 5th order spectrum aberrations. The much improved aberration correction has allowed the entrance aperture to be opened to 5.0 mm diameter (for Enfinium ER) while minimizing isochromaticity. The two dodecapoles between the magnetic prism and the beam-trap aperture magnify the energy dispersion by 3.3 times while maintaining the focus of the spectrum at the beam-trap aperture plane. This increases the dispersion to 2.5 μm/eV at 200 keV primary energy.

The remaining four dodecapoles are located after the beam-trap aperture and project an aberration corrected energy-loss spectrum on the CCD camera.

A range of spectral magnifications or dispersions have are pre-programmed at installation allowing the user to choose between dispersions ranging from 0.05 eV to 0.5 eV (1.0 eV for the Enfinium ER) per CCD channel at 200 kV.

## 3.2 Spectroscopy Operation

In spectroscopy, an image of the projector lens crossover is projected onto the camera. When properly adjusted, the observed CCD image consists of sharp vertical lines representing electrons of particular energy. Displacement of lines in the horizontal direction indicates a difference in energy between electrons.

The projector crossover is present under all normal TEM or STEM operating conditions. (If the projector lens were not excited strongly enough to form a small crossover, the image on the final viewing screen would only be a few millimeters in diameter). The crossover moves up and down in the column slightly depending on the exact operating mode, but this movement can be easily compensated by small adjustments of the pre-prism quadrupole FOCUS X. Thus the spectrometer can be tuned for any operating condition of the microscope.

The acquired spectra correspond to whatever feature was in the center of the microscope viewing screen, because this part is selected by the entrance aperture of the spectrometer when the screen is lifted up. It can be a precipitate image in TEM, a Bragg beam from a diffraction pattern, etc. Three basic operation modes are possible: TEM Imaging, TEM Diffraction, and STEM. See Appendix D, "Size of Projector Crossover," for additional details.

Due to mechanical variability of the TEM and spectrometer, the center of the TEM screen (or viewing camera) is not at the exact position of the spectrometer entrance aperture. This is normal and will not affect the operation of the system. For STEM-EELS acquisition, the alignment of the ADF STEM detector and the entrance aperture is important. The Gatan supplied detectors should be concentric with the entrance aperture and are adjusted at installation. For 3rd party or OEM supplied detectors, please contact the appropriate organization for assistance.

### 3.2.1 TEM Imaging mode

With the TEM in the imaging mode, an image is formed on the viewing screen, while the projector crossover contains a small diffraction pattern. Because the spectrometer projects an energy-dispersed diffraction pattern onto the detector, this mode is also called, somewhat counter intuitively, "diffraction-coupled" mode. The camera length L of the pattern is given by

$$L = \frac{h}{M}$$

and the diameter $d_p$ of the projector crossover therefore by

$$dp = \frac{2\,ß\,h}{M}$$

where

- $h$ is the distance from the projector crossover to the viewing screen
- $M$ is the image magnification
- $\beta$ is the half acceptance angle as defined by the objective aperture

In practical terms, with h = 50 cm and at M = 10,000x, L = 50 µm. A diffraction pattern encompassing angles to 50 mrad is therefore only 5 µm in diameter at the projector back-focal plane and it becomes even smaller at larger microscope magnifications. This means that good energy resolution can be attained in the TEM imaging mode while accepting practically all the electrons scattered from a particular specimen area. If a smaller range of scattering angles is required, this can be selected using the objective aperture. See Appendix D, "Size of Projector Crossover," for additional information.

In the TEM imaging mode, the intensity of the spectrum can be easily changed by changing the illumination focusing and/or the size of the condenser aperture. The selected specimen area is directly visible on the viewing screen and can be changed by translating the specimen, shifting the image electronically, changing the microscope magnification and changing the spectrometer entrance aperture. The collection efficiency can be very high in this mode. These characteristics make the TEM imaging mode ideal for the initial examination of any specimen.

Quantitative analysis, however, is made nearly impossible in the TEM imaging mode by the fact that electrons of different energies are spread over areas of different size in the image. If the illumination is focused to a small area, or the specimen is not homogeneous, the intensity ratio between low-energy edges and high energy edges can be changed by as much as a factor of 10 in the imaging mode simply by changing the focus of the objective lens. As a consequence, attempting to quantify the specimen composition without taking account of this effect is likely to be highly spurious. It is therefore almost always better to collect spectra for quantitative analysis in the diffraction mode (see Section 3.2.2). An exception occurs when the illumination is highly defocused (over tens of microns), the specimen is homogeneous over tens of microns and the size of the illuminated area on the viewing screen is much larger than the spectrometer entrance aperture. In this case, the electrons that miss the spectrometer entrance aperture due to chromatic aberration will be replaced by a similar number of electrons that enter the aperture in error also due to chromatic aberration. Operating under these conditions is sometimes useful on contamination-prone specimens, or when one needs to spread the illumination over a large area to minimize radiation damage, while maintaining some spatial resolution or a high collection angle.

**NOTE:** Operating in TEM imaging mode for spectroscopy should not be used except for initial examination, as it may give misleading analysis results. Please read the following explanation.

The TEM imaging mode at first sight appears to allow the selection of a small specimen area for microanalysis by the spectrometer while a larger area of the sample is being illuminated. This might seem a useful way to get a spectrum from a small particle or interface when it is impossible to get a small enough probe. A user with a LaB6 instrument would be tempted to believe they could do analysis similar to an owner of an FEG instrument. However, they would be incorrect. The image seen on the TEM screen is from the region of the EELS spectrum that has the most electrons, on a thin sample, the zero loss. Unfortunately electrons that have lost energy are focused by the TEM objective at a different location due to its chromatic aberration ($C_c$). This means the image at the energy loss being observed in the spectrum will come from a different area of the specimen. Thus this method will always give a false result, as shown below. Electrons that have been scattered by large angles and have also suffered an energy loss will be displaced laterally in the image by a distance d given by

$$d = q \cdot \Delta \mathrm{f}$$

where

- q is the scattering angle
- $\Delta$f is the defocus error of the objective lens of the microscope

The defocus error depends on the energy difference between electrons for which the objective lens was focused, and the energy-loss electrons of interest. It is equal to:

$$\Delta f = C_C \cdot \frac{\Delta E}{E_O}$$

where

- $C_c$ is the chromatic aberration coefficient of the objective lens
- $E_0$ is the primary energy

The focusing is normally done looking at the image formed by the elastically-scattered electrons, in which case becomes equal to the energy loss. For a 100 kV primary energy electron which lost 1000 eV while being scattered over 5 mrad, the displacement (referred back to the sample) amounts to 100 nm ($C_c$ = 2 mm). Thus, to obtain information from areas smaller than 100 nm, a small probe should be used, as in the STEM mode.

### 3.2.2 TEM Diffraction mode

With the TEM in the diffraction mode, a diffraction pattern is formed on the viewing screen, while the projector crossover contains an image of the illuminated area of the specimen. Because the Enfinium projects the image onto the detector, this mode is sometimes called, again somewhat counter intuitively, "image-coupled" mode. The magnification of the image related to camera length is

$$M = \frac{h}{L}$$

And the diameter *Dp* in eV at the beam trap of the projector crossover is therefore determined by

$$D_P = \frac{h \cdot d_S}{L} \cdot K_S$$

where

- $ds$ is the diameter of the illuminated specimen area
- $h$ is the distance from the projector crossover to the spectrometer entrance aperture
- $L$ is the camera length at the spectrometer entrance aperture
- $Ks$ is the spectrometer energy dispersion and magnification factor or 0.3 eV/µm at 200 kV see dispersion table in Appendix E for a list of values for various energies.

For example, a 3 µm diameter-illuminated specimen area will give rise to a 3 µm projector crossover size for L=70 cm and h=70 cm. This permits better than 1 eV energy resolution at 200 kV. The TEM diffraction mode is therefore useful for acquiring spectra from large areas,

although the size of the illuminated area must be carefully monitored when small camera lengths are used, if the energy resolution is not to worsen considerably (see Appendix D, "Size of Projector Crossover").
In the TEM diffraction mode, the angular acceptance range of the spectrometer is limited by the spectrometer entrance aperture and can be varied by changing the camera length or by selecting a different aperture size. The diffraction pattern visible on the viewing screen makes it possible to monitor the specimen phase, thickness and diffraction condition. Assuming that no selected area diffraction aperture is used, the spectrum is collected from the whole illuminated area of the specimen and the spatial resolution is therefore determined purely by the probe-forming performance of the microscope. In this case, unlike in the imaging mode, chromatic effects do not appreciably change the collection efficiency of the spectrometer in the diffraction mode, making quantitative chemical analysis of small sample areas much more reliable.

The diffraction mode is ideal for acquiring the high quality spectra necessary when looking for low-concentration elements, or when performing EXELFS analysis. It is also very useful when the diffraction condition and/or collection angles must be closely monitored, as for instance in channeling experiments.

### *3.2.3 STEM mode*

In a correctly set up STEM mode, a diffraction pattern is displayed on the viewing screen and the projector crossover contains an image of the probe. Electron-optically, this mode is therefore similar to the TEM diffraction mode.

In the STEM mode, it is easy to image a large area of the sample even though the illumination is focused into a small probe (via collection of a STEM image), and to position the probe accurately on the feature of interest (using the scan coils.) This makes the STEM mode ideal for acquiring EELS data from precise areas of the sample (precipitates, interfaces, cell membranes, etc.).

When the STEM probe is scanning over large areas of the specimen, the motion of the probe will be transferred to the spectrum giving a motion of the energy loss peaks. This effect is not apparent at high magnifications but becomes increasingly important as the field-of-view is increased. The use of descan coils is recommended to remove the motion of the probe in the energy loss spectrum.

## 3.3 Selecting an Energy Loss

It is possible to shift between different energy losses by moving the spectrum at the beam-trap plane. There are two ways in which this can be done in Enfinium:

- Adjusting the prism current
- Adjusting the voltage on the Drift Tube

Each method is discussed below.

### *3.3.1 Adjusting the Prism Current (Energy Shift)*

The prism current is used when aligning the ZLP position to the zero energy channel of the spectrometer. This is accomplished by clicking the Center ZLP function on AutoPEELS. The prism current method of moving the spectrum has the advantage of allowing almost unlimited offset of the spectrum (up to 10 keV).However, a disadvantage of using the energy shift is the small hysteresis in energy offset that can be observed when the current is varied over a large range. Therefore it's not recommended using this method for precision edge onset measurements.

### *3.3.2 Adjusting the Voltage on the Drift Tube (Drift Tube Offset)*

The second method applies a voltage to the electrically isolated drift tube that passes through the magnetic prism. In response to such an applied voltage, the electrons will accelerate by a corresponding number of eV while they traverse the drift tube. The energy-loss spectrum at the beam trap plane will thus shift downward by a number of microns corresponding to that (temporary) change in kinetic energy. At higher drift tube voltages the energy resolution slightly degrades because of a lensing effect.

The advantages of this method are its fast response (< 0.1 s), its accuracy, and the absence of hysteresis.

For spectroscopy applications where a high accuracy edge onset measurement is required, it is recommended to apply energy offsets up to 1000 eV (up to 2000 eV with optional 2 kV voltage offset module) with the drift tube voltage. Energy offsets above 1000 eV can be achieved by applying increments of -1000 eV to the prism current (energy shift) control. For spectroscopy applications with the aim of achieving the ultimate energy resolution it is recommended to use the prism current (Energy Shift) rather than the drift tube offset due to the slight lensing effect of the drift tube.

# 4. GIF Quantum and Enfinium Facilities Requirements

## 4.1 TEM Requirements

### 4.1.1 TEM Compatibility

The GIF Quantum and Enfinium should only be used with TEMs listed on Gatan's compatibility chart or specifically approved in writing by Gatan Engineering.

## 4.2 Enfinium Operating Requirements

### 4.2.1 Environment, Water, and Air

***Table 1 Requirements for Enfinium Operation***

| | |
|---|---|
| Ambient temperature | 16-24 °C |
| Water cooling | 0.2-0.5 l/min at 15-21 °C<br>40 N/cm$^2$ (60 PSI) maximum inlet pressure<br>(no restriction on the outlet flow) |
| Pressurized air | Dry air, 40-60 N/cm$^2$ (45-90 PSI) |
| Dew point | < 10 °C |
| Humidity range | 20-80% |
| Ventilation | Do not block or cover ventilation holes |
| Pollution degree | 2 (see IEC 61010) |
| Installation category | 2 (see IEC 61010) |
| Altitude | 2000 m max |
| Indoor use only | |

*Table 2 Requirements for Enfinium Non-Operation*

| | |
|---|---|
| **Storage temperature** | **0-65 °C** |
| **Dew point** | < 15 °C |
| **Humidity range** | 10-90% |

To guarantee the optimum energy and spatial resolution for each Enfinium model, the stray magnetic field at any point along the beam path between the TEM projector lens crossover and the entrance aperture may not exceed the limits listed in the table below.

*Table 3 Stray Magnetic Field Limits*

| SPECTROMETER MODEL | SPECTROMETER SPECIFICATION (meV) | GATAN FIELD SPEC (mG P-P) LINE FREQUENCY | 1-1000 HZ |
|---|---|---|---|
| **976** | 400 | 1 | 0.3 |
| **977** | 100 | 0.4 | 0.15 |

### 4.2.2 Electrical Power

Mains supply voltage fluctuations are not to exceed 10 percent of the nominal supply voltage.

*Table 4 Electrical and Power Specifications*

| EQUIPMENT | VOLTAGE | CURRENT | FREQUENCY | POWER (MAX) | POWER (TYP) |
|---|---|---|---|---|---|
| **Camera Controller** | 100/120/220-230/240 VAC | 8/6.3/4/4 A | 50/60 Hz | 375W | 80 W |
| **GIB Power Supply** | 100/120/220-230/240 VAC | 5/5/3/3A | 50/60 Hz | 900W | 460 W |
| **Computer*** | 100-240 VAC | | 50/60 Hz | 750W | |
| **Monitor*** | 100-240 VAC | | 50/60 Hz | 200W | |
| **DigiScan II** | 100/120/230/240 VAC | | 50/60 Hz | 30 W | 20 W |
| **Electrostatic Shutter** | 24 VDC | 1000 mA | | | |
| **Fast Drift Tube** | 24 VDC | 1250 mA | | | |
| | **Enfinium Hi Vac SE/ER model Only** | | | | |
| **Vacuum Pump for the camera** | 100/115/220-230 VAC | | 50/60 Hz | | 120 W |
| **Ion Pump Controller** | 110/220 VAC | | 50/60 Hz | | 50 W |
| **Heater for Ion Pump†** | 90-130 VAC | | 50/60 Hz | | 140 W |
| **Heater Tape for the Spectrometer†** | 90-130 VAC | | 50/60 Hz | | 522 W |

**NOTE:** Ground for spectrometer hardware power must be the same as for TEM power.

* refer to the documentation for the exact model supplied
† Only used during installation or service

*Table 5 Fuse Specifications*

| EQUIPMENT | VOLTAGE | FUSE |
|---|---|---|
| **Detector Controller** | 100 V | T8A 250 V |
| | 120 V | T6.3A 250 V |
| | 220-230 V | T4A 250V |
| | 240 V | T4A 250V |
| **GIB Power Supply** | 100 V | T10A 250 V |
| | 120 V | T8A 250 V |
| | 220-230 V | T4A 250 V |
| | 240 V | T4A 250 V |
| **DigiScan II (optional)** | 100-240V | 1.0A,SLO BLO |

The GIB power supply fuse holder has two locations for the fuses. One side holds the US shape fuse and the other side holds the European shape fuse. Typically the European size fuse is provided.

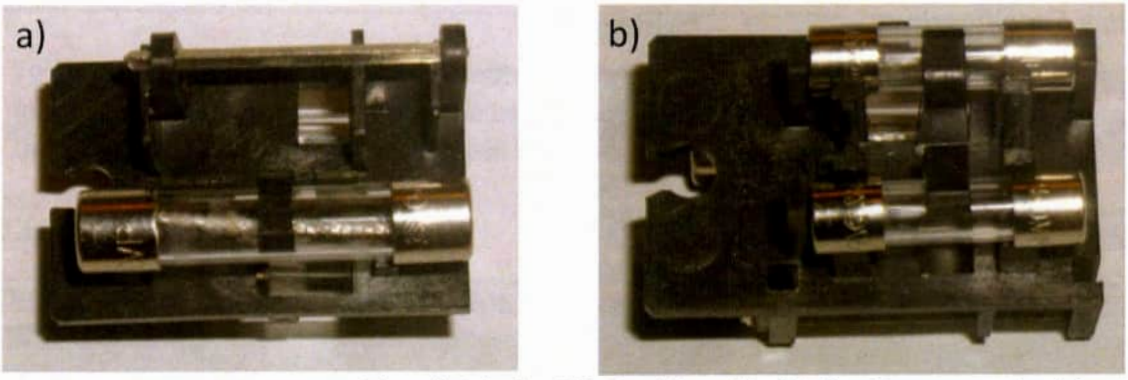



Figure 8 GIB power supply Fuse Holder a) side for US sized fuse b) side for European sized fuse.

### 4.2.3 Weight

The weight of the Enfinium shown in Figure 1 varies depending on the TEM, voltage, and detector configurations. A 300 kV model below an UltraScan camera housing weighs 240 lb. It does not include the weight of the UltraScan camera or any other optional detector above the GIF/Enfinium.

### 4.2.4 Environmental Loading

Table 4-6 represents the typical heat output to the air and water during normal operation, there is some variation with Enfinium model, TEM voltage and exact alignment.

***Table 6 Heat Load***

| EQUIPMENT | HEAT SINK | HEAT LOAD |
|---|---|---|
| **GIB and GIB power supply** | Air | 515 W |
| **Enfinium mechanical on TEM** | Water | 35 W |
| **Enfinium mechanical on TEM** | Air | 2.5 W |
| **Camera** | Water | 80 W |
| | Air | 130 W Max, 50 W Typical |

### 4.2.5 Connection Diagrams

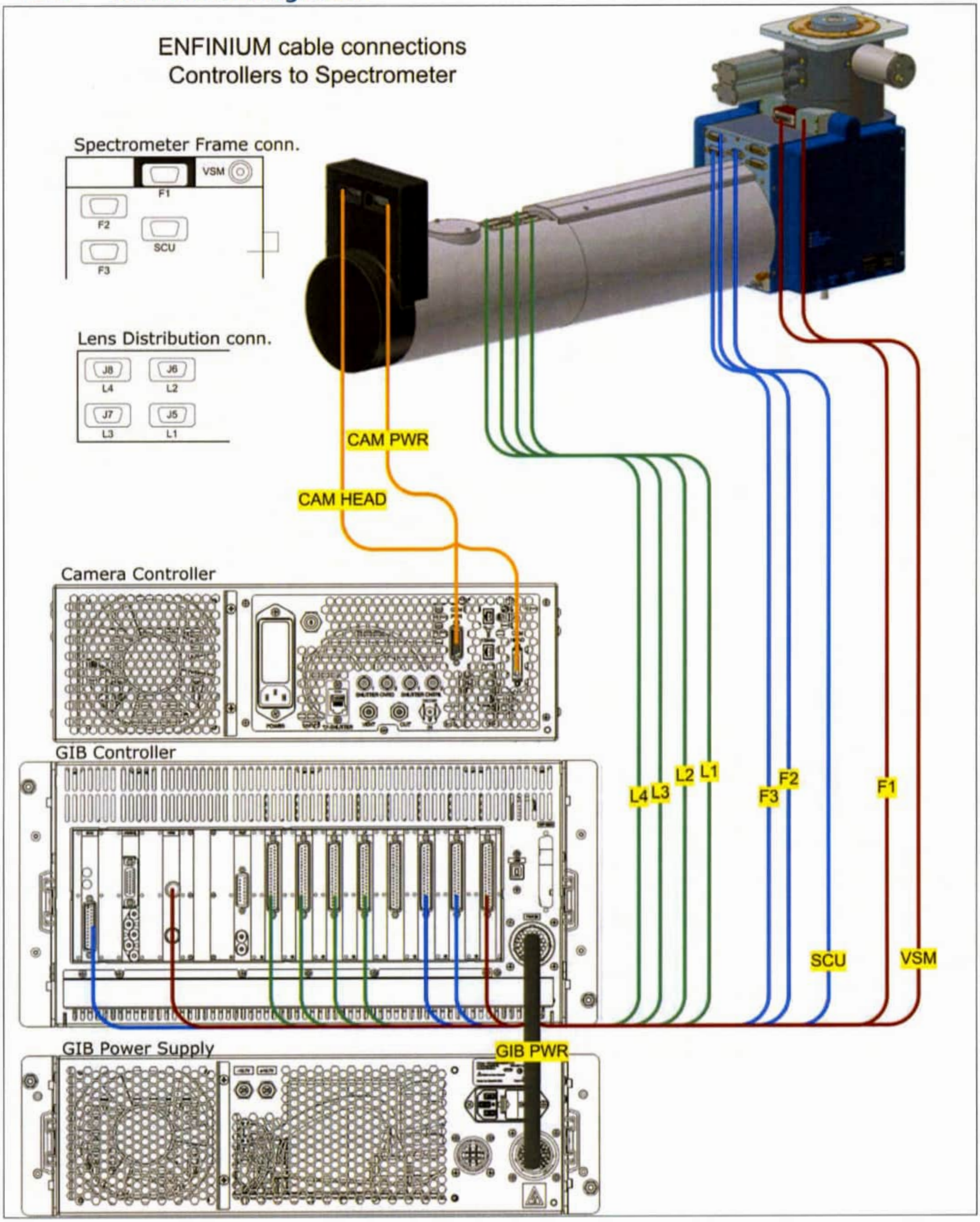



*Figure 9 Controller to Enfinium cable connections*

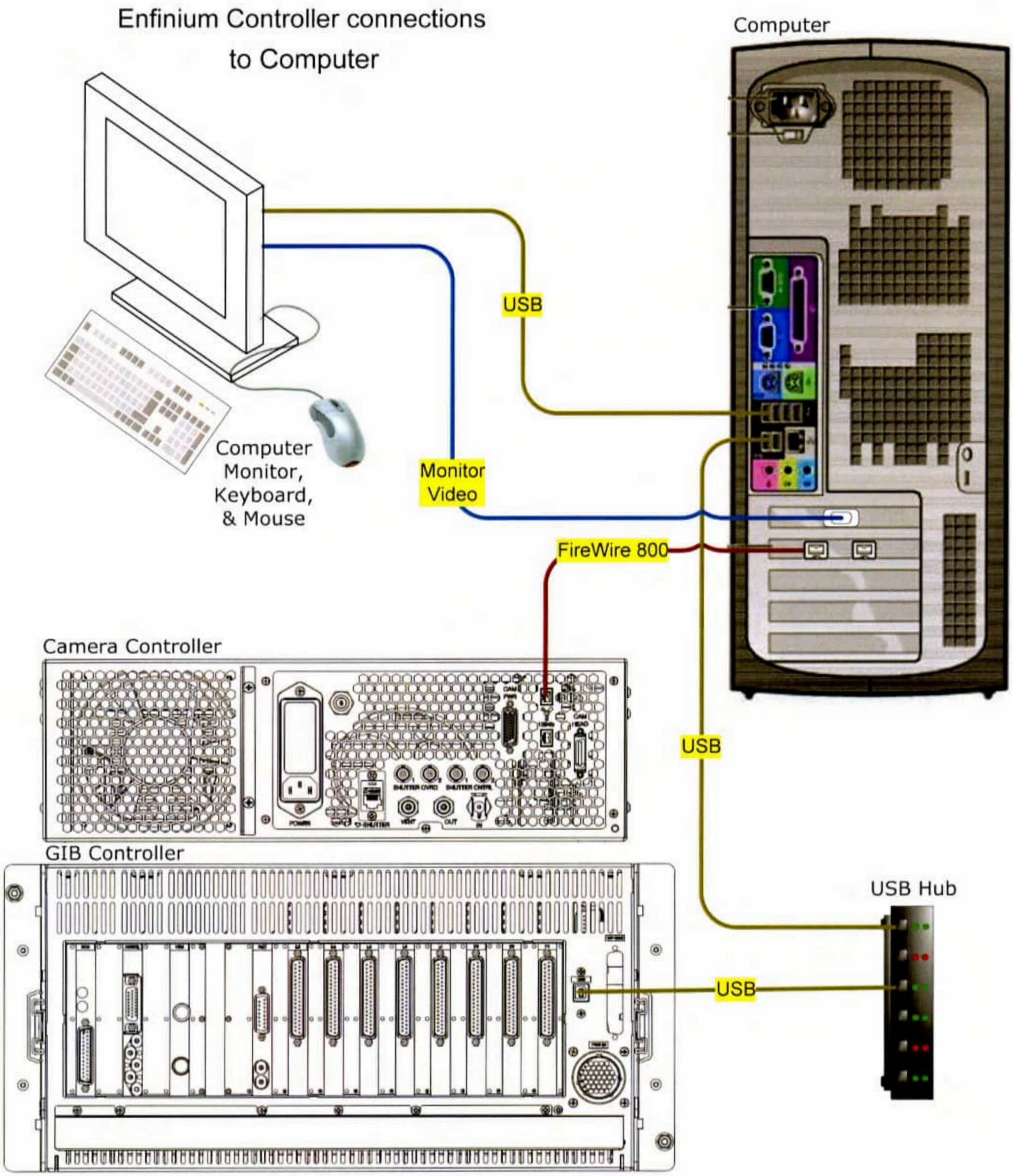



*Figure 10 Enfinium Controller to Computer connections*

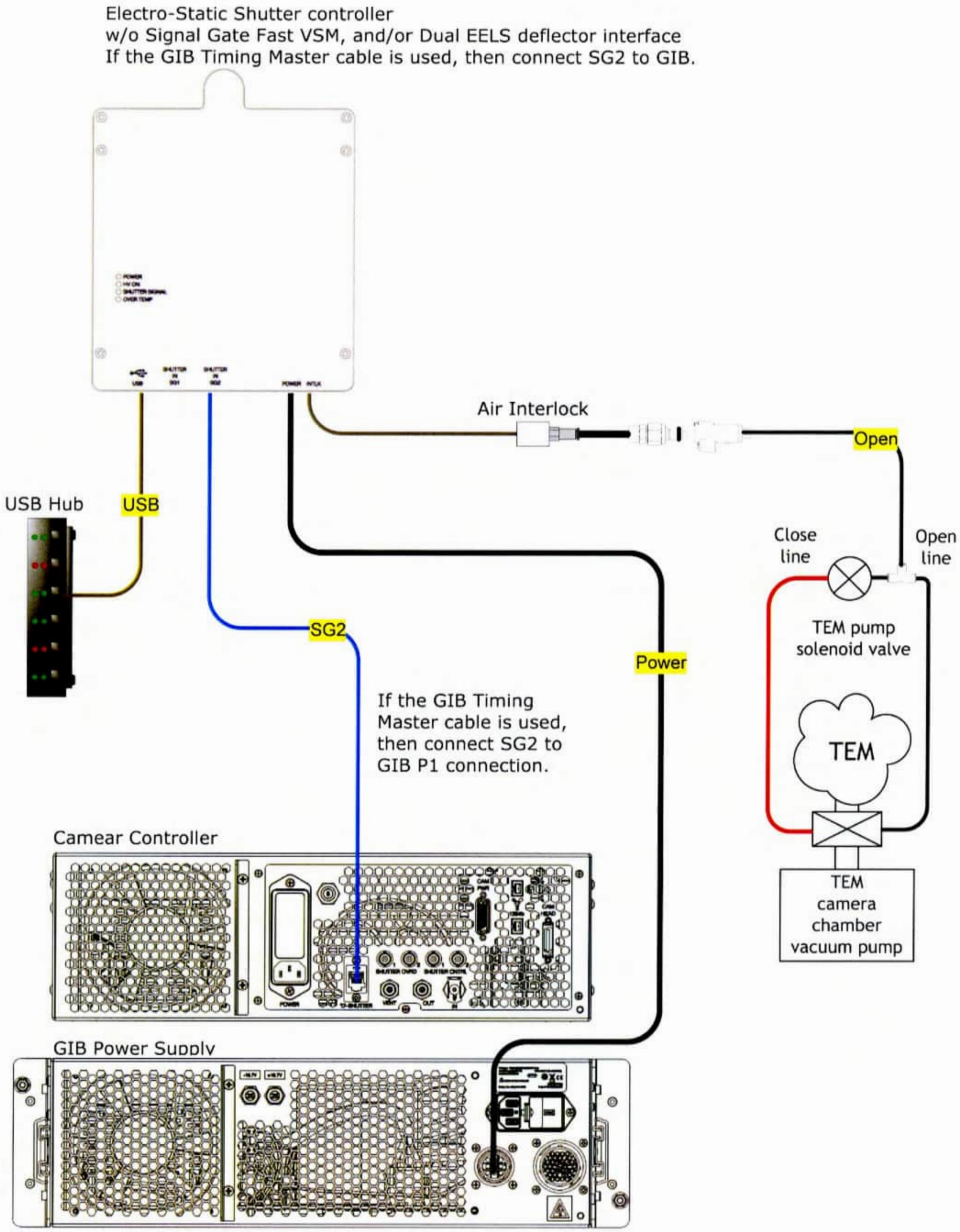



***Figure 11 Electrostatic shutter controller connections***

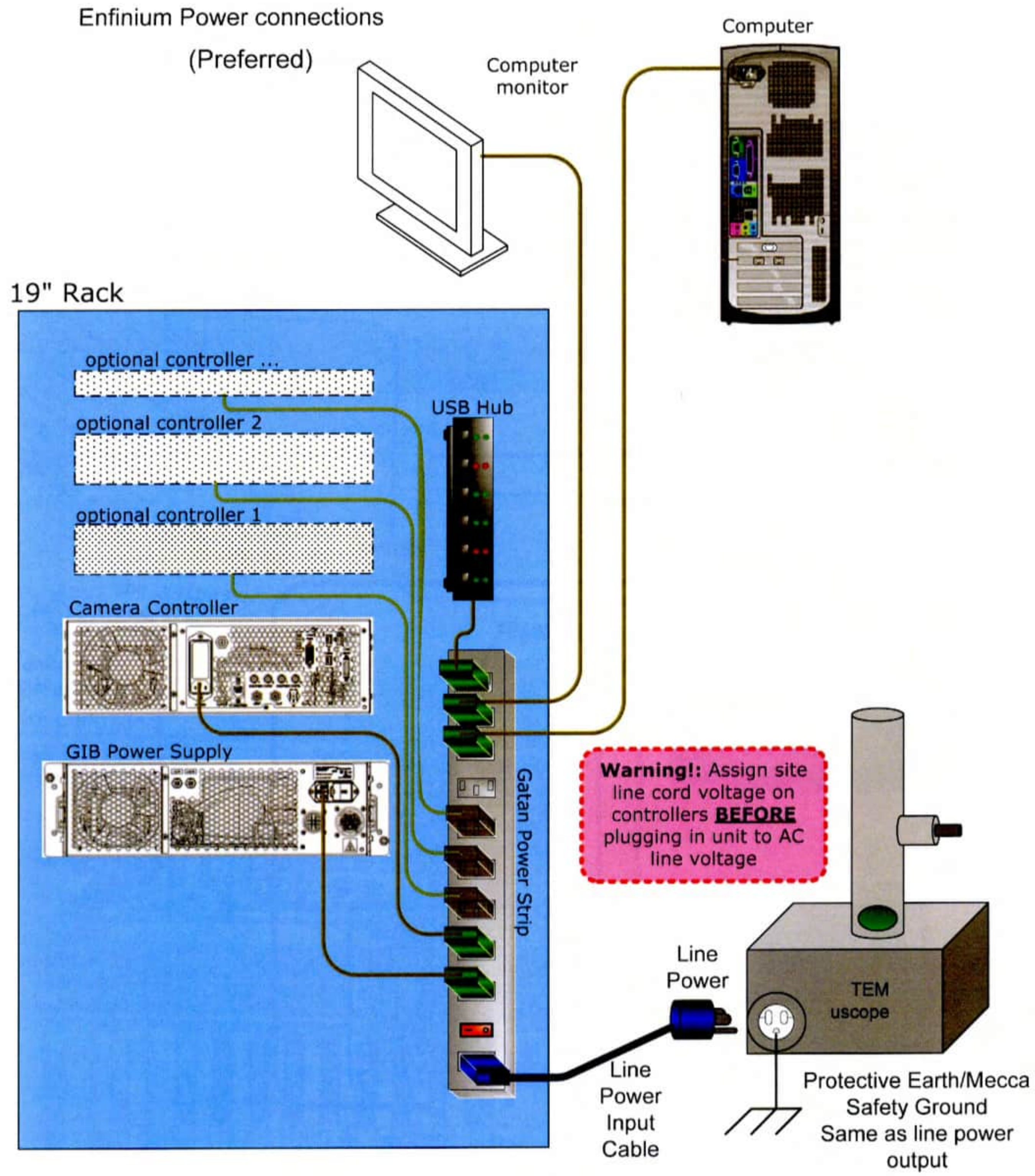



***Figure 12 Power Connections***

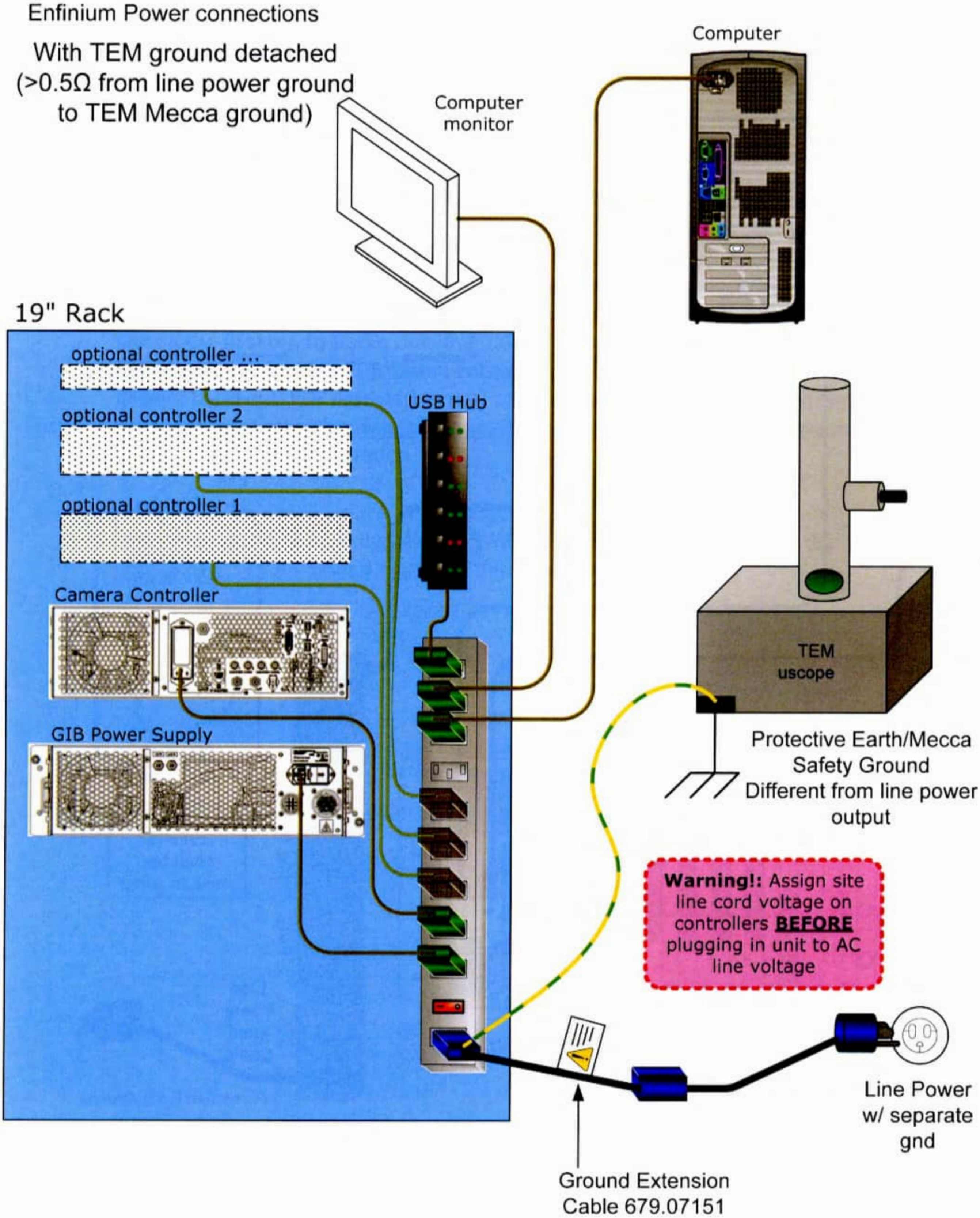



*Figure 13 Power Connections with TEM ground detached*

Fast VSM, and/or Dual EELS deflector controller connections

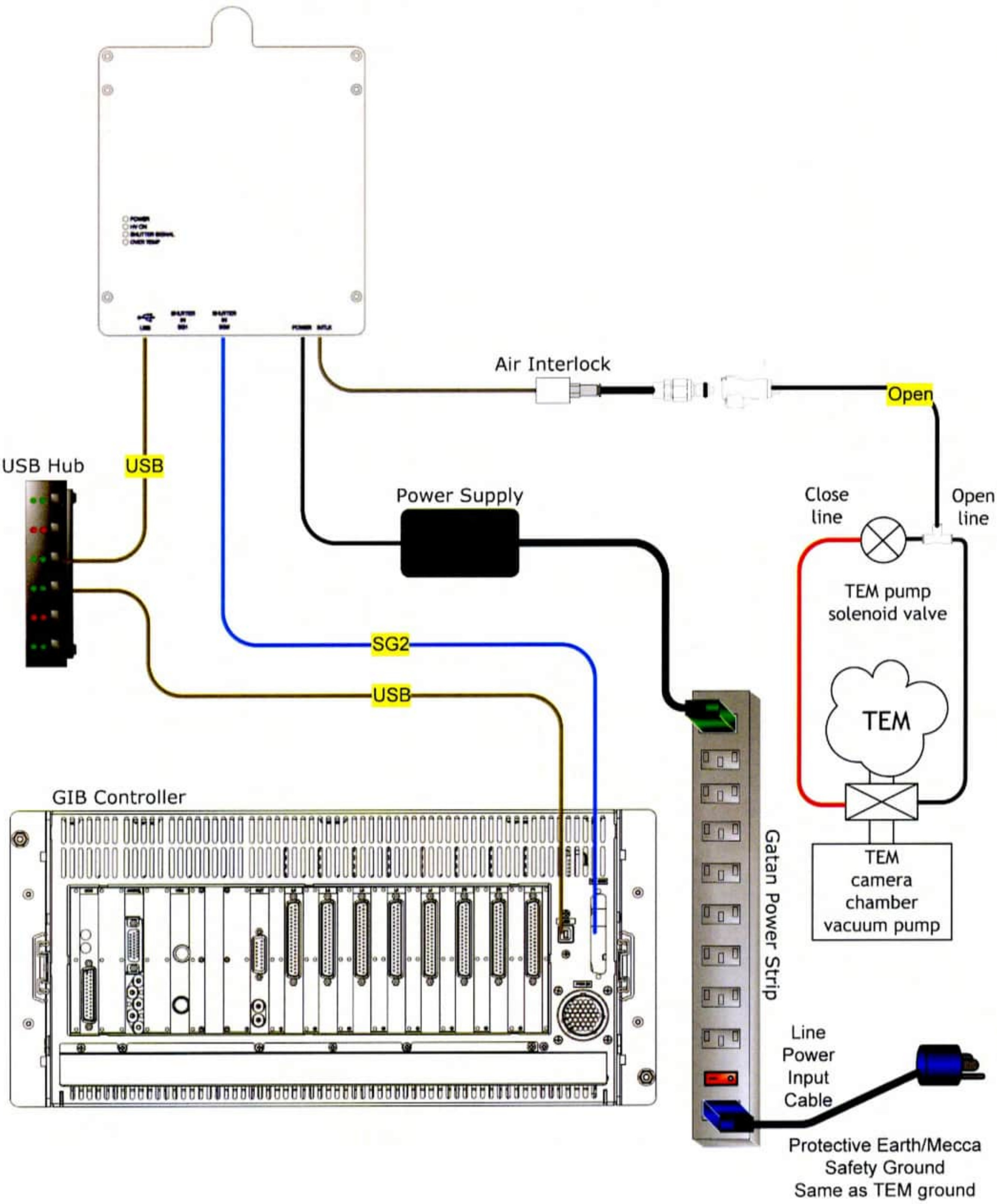



*Figure 14 Fast VSM and/or DualEELS deflector controller connections*

## 4.3 Instrument Control

To be able to operate the spectrometer efficiently, the user will need to be thoroughly familiar with the operation of the Enfinium camera, DigitalMicrograph, AutoPEELS, and the EELS analysis extensions to DigitalMicrograph (plug-ins). **Additional information is supplied in the separate manuals for these items. When the Gatan Microscopy Suite (GMS) is installed on a computer, a directory is created with a link on the desktop to Gatan Documentation. This documentation folder contains the manuals in Acrobat PDF.** Gatan encourages users to either read them on-line or print them out. PDF readers for most types of computers and operating systems are available at http://www.adobe.com. The Help menu in DigitialMicrograph also provides direct access to documentation.

Specific information on using Microsoft Windows can be found either via the built-in help system or via a myriad of third-party manuals.

# 5. Installation

## 5.1 Hardware Installation

Installation of the Enfinium can only be performed by a trained Gatan service representative. See the "Support" section in the "Preface" of this owner's manual for the Gatan service office nearest you.

## 5.2 Software Installation

Normally, two disks are required to install the Gatan Microscopy Suite (GMS) software for your Enfinium system, a GMS License Files CD and a GMS Software Installer DVD. Typically, all GMS software required for your installation will be installed and set up by the Gatan installation engineer as part of the final on-site installation and adjustment of your Enfinium system. For details of the installation process, please see the PDF document "GMS - Getting Started" included on the GMS Software Installer DVD.

When installing GMS software on a computer for the first time, start by inserting the appropriate GMS License Files CD ('on-line' or 'off-line'). This will automatically run a license installer program. The GMS License Files CDs contain the site ID and license files for all components ordered for your installation. They were made specifically for your site and should be kept in a safe place. Please do not duplicate or distribute the contents of these CDs, except for archival purposes. Please also make sure not to install license CDs with differing site IDs, neither should both the 'on-line' and 'off-line' licenses be installed on the same computer. Doing so might disable your GMS software.

After the GMS License Files CD is ejected, insert the GMS Software Installer DVD. This automatically launches the GMS installation program. Follow the prompts and enter the requested information about your system configuration. Further details of your installation are specified in the GMS license files installed previously. The installer program and auxiliary files on your GMS Software Installer DVD are generic and may freely be duplicated and used for multiple installations. Please note that each GMS Software Installer DVD is for a specific GMS software version. The minimum version required for Enfinium support is GMS 2.1.1. (GMS 1.9.4 for certain OEM installations).

# 6. Getting Started

After the Enfinium has been installed by a Gatan-authorized installation engineer, the system is ready for use. All setup and calibration for routine work should be complete.

As a significant extension of the TEM/STEM electron-optical column, an EELS spectrometer and its specialized detector system add many new imaging and analytical capabilities to the host microscope. Most of these techniques require specific system setups and special data acquisition and analysis software.

Rather than covering the entire gamut of electron energy-loss spectroscopy (EELS) techniques, this chapter provides brief instructions on how to set up and operate your TEM-EELS spectrometer system to perform a rudimentary data acquisition that form the basis for almost all other EELS experiments:

- EELS acquisition in TEM image mode

For details on more advanced techniques (e.g., STEM EELS Spectrum Imaging), please refer to the specific user guides provided with the specialized software packages used to perform them. These can be found in the Gatan Documentation directory (shortcut to this directory is placed on desktop at the time of installation).

## 6.1 System Startup

For optimum stability, it is best to leave the various electronic modules and control software of the Enfinium turned on and running, much like the host TEM instrument and its various control systems. If the spectrometer system has been powered down, a minimum warm-up time of 1-2 hours is required to allow the system to come to a steady state.

**To bring up the system from a cold start, do the following:**

1. **Turn on the GIB power supply (Figure 3)**
   - Set the front panel switch to on.
   - The switch will light up (green) when power is applied.

2. **Power up and set the camera controller electronics (Figure 6)**
   - Set the POWER switch to on (the LED above it will light).

3. **Turn on the Gatan Microscopy Suite (GMS) computer**
   - In most cases this is a separate computer supplied by Gatan .
   - In some cases, this might be the host computer of the TEM (e.g., FEI Tecnai) and it might already be running.
   - (For Windows XP only) Please note the windows login account must have "power user" or above privileges.

4. **Launch the DigitalMicrograph program on the GMS computer**

The DigitalMicrograph program icon looks like this:


- Upon launch, DigitalMicrograph will open communication with and run startup diagnostics on all the Gatan systems installed on your TEM, including the CCD camera at the end of the Enfinium system. Simultaneously, Enfinium Control (Refer to **1.2.2 Low-Level Instrument Interface and Control** for a brief description of Enfinium Control user interface) to runs the prism and lenses through a demagnetization cycle, and puts the spectrometer in a standard initial state.

- Some brief diagnostic messages are output to the Results window to confirm the functional state of these systems. For Enfinium ER model, during the startup process, you should hear one or more puffs of air as the pneumatics are cycled and set to the initial configuration.

- Open AutoPEELS palette if it is not already open. The primary interface for using your Enfinium for EELS acquisition is the AutoPEELS floating palette. It can be opened by selecting the AutoPEELS item in the Window | Floating windows sub-menu

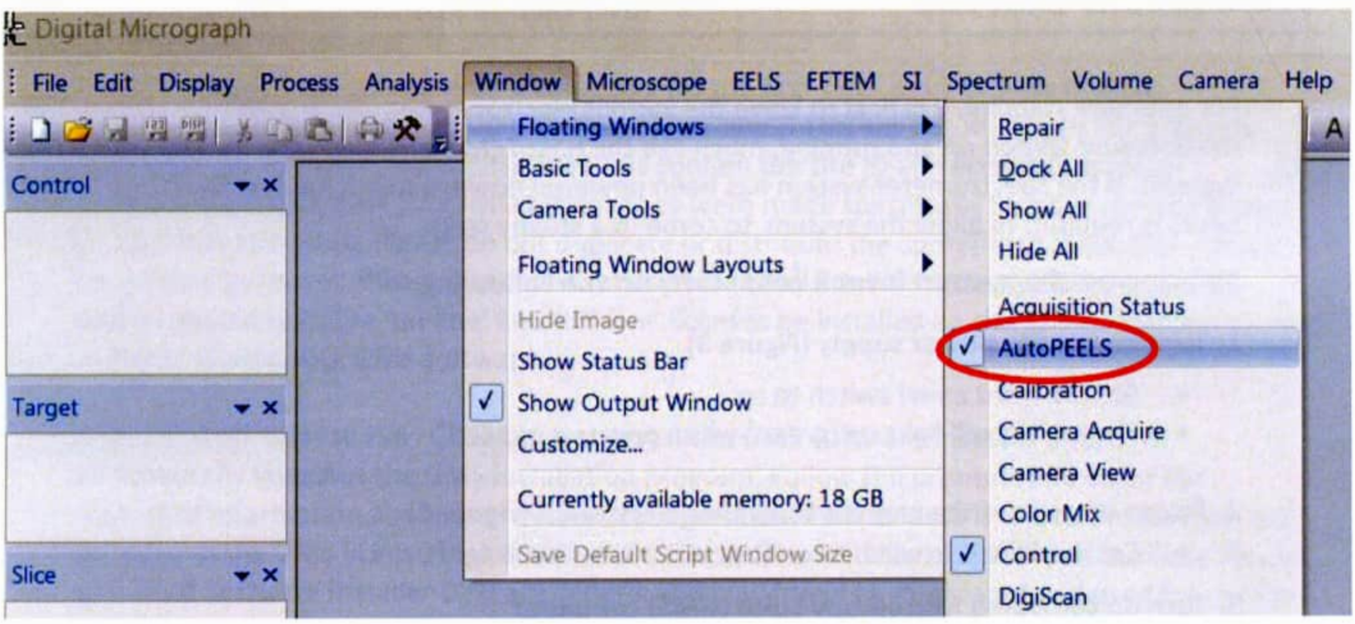



***Figure 15 Opening AutoPEELS palette from Window | Floating windows sub-menu***

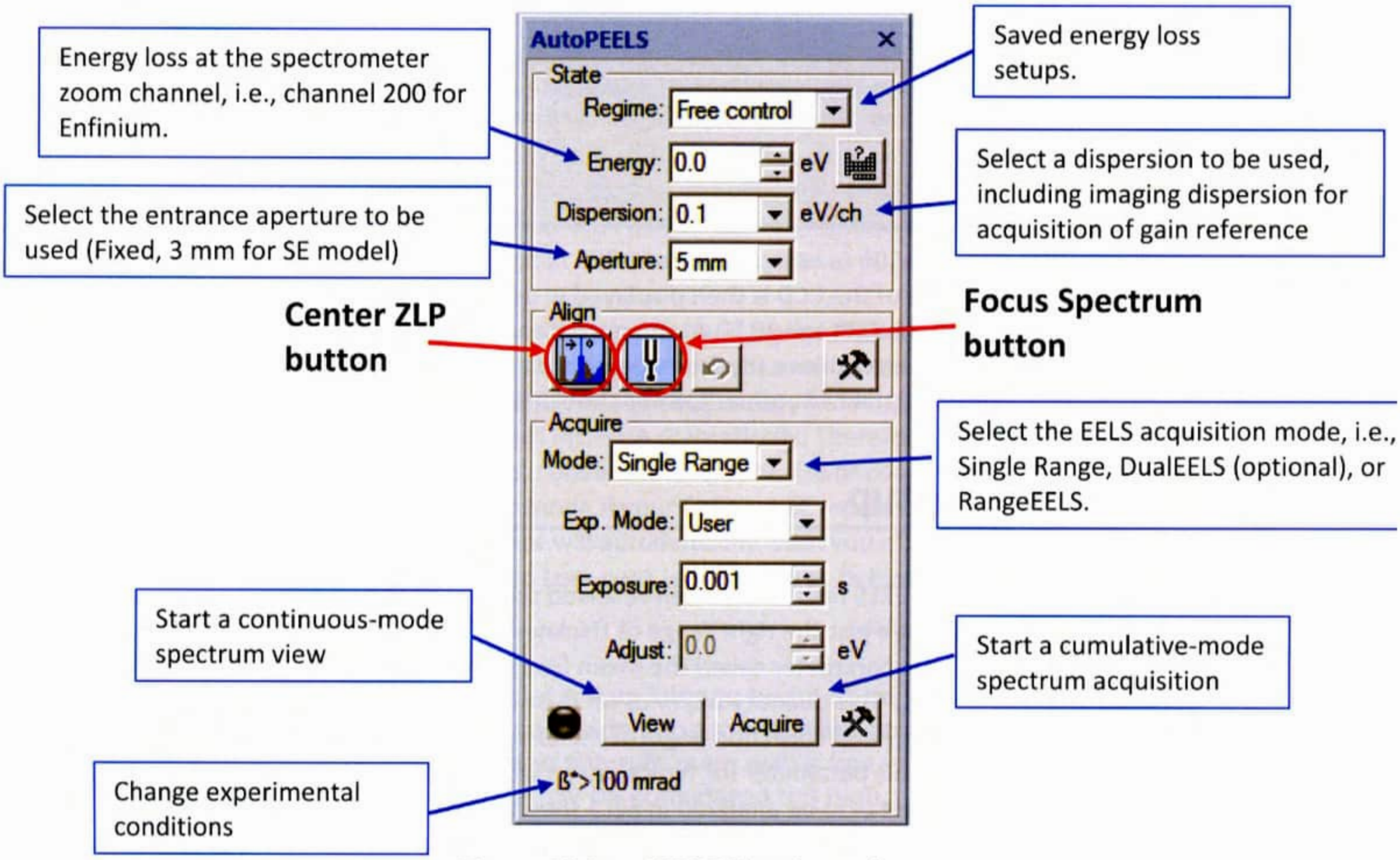
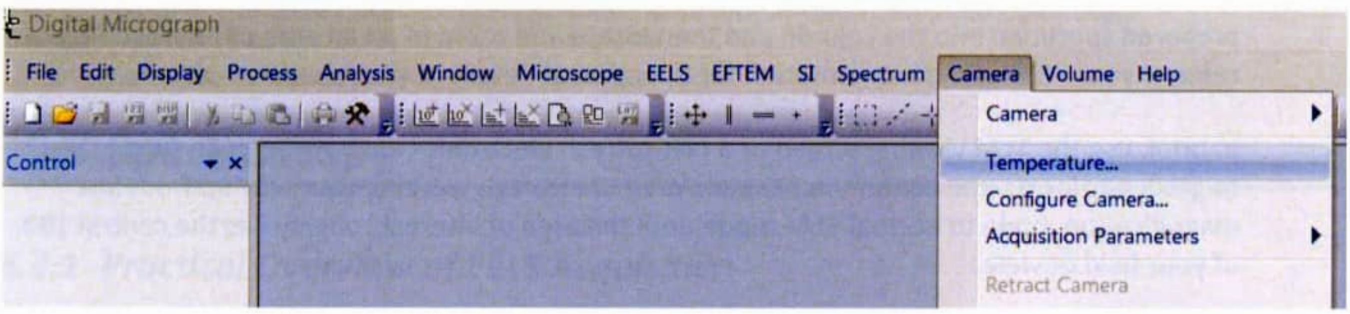
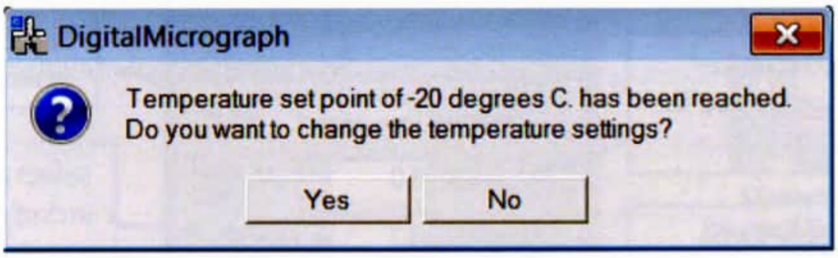



***Figure 16 AutoPEELS floating palette***

As shown above, the AutoPEELS panel indicates that the Enfinium is configured for EELS spectroscopy in 0.1 eV/ch dispersion. **Full details regarding the use and operation of the AutoPEELS palette can be found in the AutoFilter-EELS Mode chapter located in the DigitalMicrograph online Help documentation.**

**5. Set the camera temperature control from the DM camera pull-down menu as shown below**






- The temperature of the CCD is then displayed in degrees Celsius. For normal operation, this value should be set to –20 °C. Starting from room temperature, it normally takes about 30 minutes to achieve the final operating value. The software is normally set to start cooling the camera automatically at start-up of DigitalMicrograph.

## 6.2 Experiment Setup

In order to achieve good EELS results with well-resolved spectral signals, it is very important to prepare the sample to have just the right range of thickness in the area of interest. The reason for this revolves around a parameter called the mean free path for inelastic scattering, often denoted by the symbol $\lambda_{in}$. This quantity depends on the elemental composition of the sample as well as the beam energy and collection angle of the experiment, but a very rough, order-of magnitude estimate for this parameter for typical materials at 200 keV beam energy is about 100 nm. Ideally, sample areas to be analyzed in EELS should have a thickness in the range of about 0.3 – 1.0 times $\lambda_{in}$. For many materials this means sample thicknesses of about 50-70 nm are fairly ideal.

Your Enfinium system is supplied with a number of ready-made TEM samples that have areas of suitable thickness and which are used by the installation engineer to perform initial calibrations and checks of your spectrometer. These samples can also be used in your first few Enfinium sessions to become familiar with its operation.

### *6.2.1 Locating a Sample Area for EELS Analysis*

Start your EELS session as you would for any other type of TEM observation. Load your suitably prepared specimen into the column and then locate and zoom in on an area of interest. Please refer to your TEM user documentation for operational details of your specific instrument.

In brief, use the TEM viewing screen or a fast-refresh electronic camera with wide field of view to gradually locate and center your sample area of interest, working your way up from low magnification mode to normal TEM mode until the area of interest roughly fills the central 10% of your field of view.

### *6.2.2 Viewing the Sample Area*

The next steps may differ slightly depending on the degree of integration between the Enfinium and your host TEM instrument. There is, however, one basic point to keep in mind. The entrance opening to the Enfinium is a 5 mm diameter aperture in the case of the Enfinium ER, or a 3 mm aperture for the Enfinium SE, that is approximately aligned (to within 2-3 mm) with the center of your TEM viewing screen or wide-view observation camera. Only that portion of the viewed image (or diffraction pattern) that can pass through this entrance aperture will contribute to EELS spectra.

Follow these steps to set your system up for spectrum-acquisition. The guide uses data acquired from a BN-C sample for illustration. Gatan supplies BN-C samples with spectrometers because 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.

Because the spectrum covers only a narrow band on the CCD and typically 90% of the electrons will be elastically scattered electrons and end up in a narrow zero-loss peak, one should realize that by switching from Imaging Mode to Spectroscopy Mode, the electron density at the camera (number of electrons / area) will increase dramatically. Therefore, it is wise to reduce the beam intensity (microscope gun bias, condenser lens, spot size or condenser aperture) before switching to the Spectroscopy Mode if your EnfiniumSE model is not equipped with a fast beam blanker. The AutoFilter software will automatically warn you of this when switching to spectroscopy mode. If the Zero Loss peak is saturated at 1x binning at the exposure being used the beam is too bright.

**CAUTION**: Do not let a very intense beam sit on the camera/detector for too long as it may cause damage to the scintillator. If the beam intensity causes the camera to saturate even with a low exposure time, (0.1 s or less) it should generally be considered too high.

While observing a spectrum, one can select the desired energy dispersion from the set of pre-programmed dispersions (the range of available dispersions can vary with primary energy). Typically, low dispersions (higher numerical values) are used to get an overview of the energy-loss spectrum while higher dispersions (lower numerical values) are used to study spectra in more detail. All dispersions are focused at the beam trap.

A tutorial is given below to help a user become familiar with the system. After working through the tutorial, a user should be able to run the spectrometer and take spectra. Details on EELS acquisition user interface, EELS analysis within Gatan's DigitalMicrograph environment can be found in the Gatan Documentation folder installed on Gatan supplied computer.

# 6.3 Spectroscopy

## 6.3.1 *Practical Overview of EELS Acquisition*

- Refer to **Appendix A Using Enfinium Control as a Power User** for the use of Enfinium Control Palette (**For diagnostic purpose only**)

1. **Clear the beam path into the spectrometer**

- Please take care to retract anything that may obstruct the beam between the TEM viewing area and the spectrometer entrance aperture, including the viewing screen, beam stops, film plates, bottom-mounted digital cameras, and on-axis STEM detectors.

## 2. Locate the beam

- Locate a hole in the sample at any magnification in the normal magnification mode (between 2K and 200K).
- Illuminate the center 2 to 10 cm of the TEM screen.
- Lift the TEM screen.
- Hit the View button in the AutoPEELS floating palette, and the ZLP should be displayed. Pressing the escape key or space bar stops the view.
- Finally click on Center ZLP button in the AutoPEELS floating palette and the beam should be aligned. Watch the screen to monitor the progress bar to see when it has finished.

If you don't already see the ZLP;

- First confirm that the Primary Energy shown in Enfinium Control palette matches the actual beam energy. Normally, this value is updated automatically via the GIF integration software with the host TEM. If your TEM has a GIF integration package, please make sure the communication between GMS and the host TEM is working. Otherwise, enter the beam energy in the Global Info dialog under the Microscope -> Configure sub-menu . This should then also update the value shown in Enfinium Control palette. The Primary Energy control coarsely sets the current passing through the prism to an appropriate value to achieve a 90 degree bending of the beam for the indicated beam energy.
- If the Center ZLP routine fails, try directly manipulating the Adjust field in Enfinium Control palette. Slowly vary Adjust (usually within the range ±1000 eV) until you see some beam intensity. Then try Align ZLP again.

## 3. Prepare a gain reference

At this point you should take a gain reference image. Ideally, the gain reference should be updated at the start of a spectroscopy session, though in practice about once a week should be sufficient. This is an image of a uniform beam and shows the variation in gain between pixels. After you have prepared the gain reference it will be used to correct variations in all subsequently acquired images. The spectrometer detector consists of a CCD, fiber optics, and a scintillator. When these are all combined the final system has slightly different response to electrons at different locations. A single primary electron gives a slightly different signal in the CCD depending upon location and hence the pixel it excites. By taking an image of flat illumination any such variation can be quantified and removed. Normally gain variations amount to less than 5%.

**Gain reference procedure for dedicated EELS spectrometers:**

- In the AutoPEELS palette, set the dispersion to Imaging, the start energy to 0eV and set the aperture size to 5 mm (3 mm, Fixed for SE model).
- Open the EELS Acquire Setup dialog, and in the Options tab check on the Show Original Image for View item. Set the vertical binning 1x in Detector Setups.
- Hit View in the EELS Acquisition floating window, with exposure set to 0.1s. The intensity in the spectrum 2D view should be centered, if not adjust the Start Energy until the beam is centered.
- With the spectrum view still running, adjust the beam on the TEM to attain a count level of about 20,000 (1/3rd the saturation limit of the CCD), and leave the system viewing for 5 minutes. The reason the beam is left over the whole detector for five minutes is to "anneal" the scintillators. This removes any "history" and reduces the memory effect of future bright beams.
- Stop the spectrum view.

If, at any later time, you see fixed pattern noise in the image or the detector is temperature cycled, you will need to repeat the process of obtaining a gain reference. The choice of the parameters in the "Prepare Gain Reference" dialog" has an effect on all of the images that you acquire later. All images that you subsequently acquire are divided by the gain reference image. 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 in the resulting image. 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 100 frames averaged later then make sure the gain reference has 100 frames averaged. The target intensity is set at a level well below the saturation level so that the detector is unlikely to saturate in a single frame. On the Enfinium, saturation varies from CCD to CCD but is normally between 55,000 and 65,000 counts.

## 4. Focus the system

- To focus the system click on the AutoPEELS Focus Spectrum button. Normally only first order focus, i.e., FX and FY, needs adjustment. Quick Tune is configured by default to correct first order focus. - **please refer to EELS Alignment in the AutoFilter documentation found in DigitalMicrograph Online Help documentation for a more in-depth description.**
- In the event that the auto-focus routine does not perform adequately, the spectrometer can be manually aligned. For further details please refer to the Manual Alignment section.

## 5. Selecting an area and setting the microscope

To gather a spectrum, locate an area of interest on the sample. To obtain good EELS data it is important that the area you look at is both thin and clean. If visible contamination forms while you observe the sample, high-quality spectroscopy will be impossible.

- Find the general region of interest at low magnification.
- Increase magnification once the region of interest is located.
- Reduce the spot size and condenser aperture to typical values for micro analysis (see the TEM user guide). Put the beam on the feature of interest, selecting a region which

appears highly electron transparent if possible. Avoid focusing to a very small spot where possible as this tends to contaminate some samples (see the section on contamination in the Additional Tips section.)

- Select the diffraction mode on the TEM and set the camera length to between about 5 and 20cm to give a collection angle in the region of 20mrad. Make sure the center spot (disc) is in the center or the screen.
- Check that the view parameters are similar to those shown below.
- Lift the screen. Then click the View button to start the system. You should see a low-loss spectrum, including the zero loss peak.
- Now adjust the diffraction shift setting on the TEM to optimize the amount of signal entering the detector.
- Align the ZLP on the 0eV channel and focus the spectrum using the Centre ZLP and Focus Spectrum buttons in the AutoFilter Align Group box.

## 6. Set energy range

Now that you have started to view of a spectrum it is a good idea to think about what your aim is. For this exercise we will acquire a spectrum covering a large energy range. The energy range covered by a spectrum is governed by both the Start energy and the Dispersion. The start energy setup 200 channels in from edge so an offset of 0 eV shows the whole of the zero loss peak. For example a start value of 0eV and dispersion of 0.5eV/ch would give a 2048 channel spectrometer a spectrum covering the range -100eV to 924 eV. The example below demonstrates acquiring a spectrum from 100eV to over 500eV. This is a common region to want to explore since it contains some of the clearest core loss edges. The low loss region may also contain some edges but these are difficult to obtain meaningful information from. The low loss region is also very bright so, if we take a spectrum containing the zero loss and this is not saturated, then the intensity in the spectrum at 500eV is very small and none of the clearly resolved core-loss edges will be seen. By starting at 50eV or 100eV, we avoid the brightest part of the spectrum and can optimize the exposure on the most interesting part of the spectrum. In general, low-loss and core-loss experiments should be performed separately, with the core-loss experiment first to minimize any scintillator artifacts from high dose.

The edges of interest in this BN-C range from 188eV loss to 402eV loss. To ensure that we have a decent pre-edge background for the first edge (about 50eV), and also that we capture the tails of the last edge (about 80eV), we need to acquire a spectrum over approximately 350eV. Over 2048 channels (for an Enfinium spectrometer), the required dispersion is therefore 350/2048 = 0.17eV. Hence, we need to use the 0.25eV dispersion. Any greater dispersion (smaller number) will capture a window that is too small. Set the dispersion to 0.25eV/ch and the start energy to 130eV. This will give us an energy range from 80eV to about 590eV. Choose an exposure of 0.1 second and click view. The spectrum look like the one below.

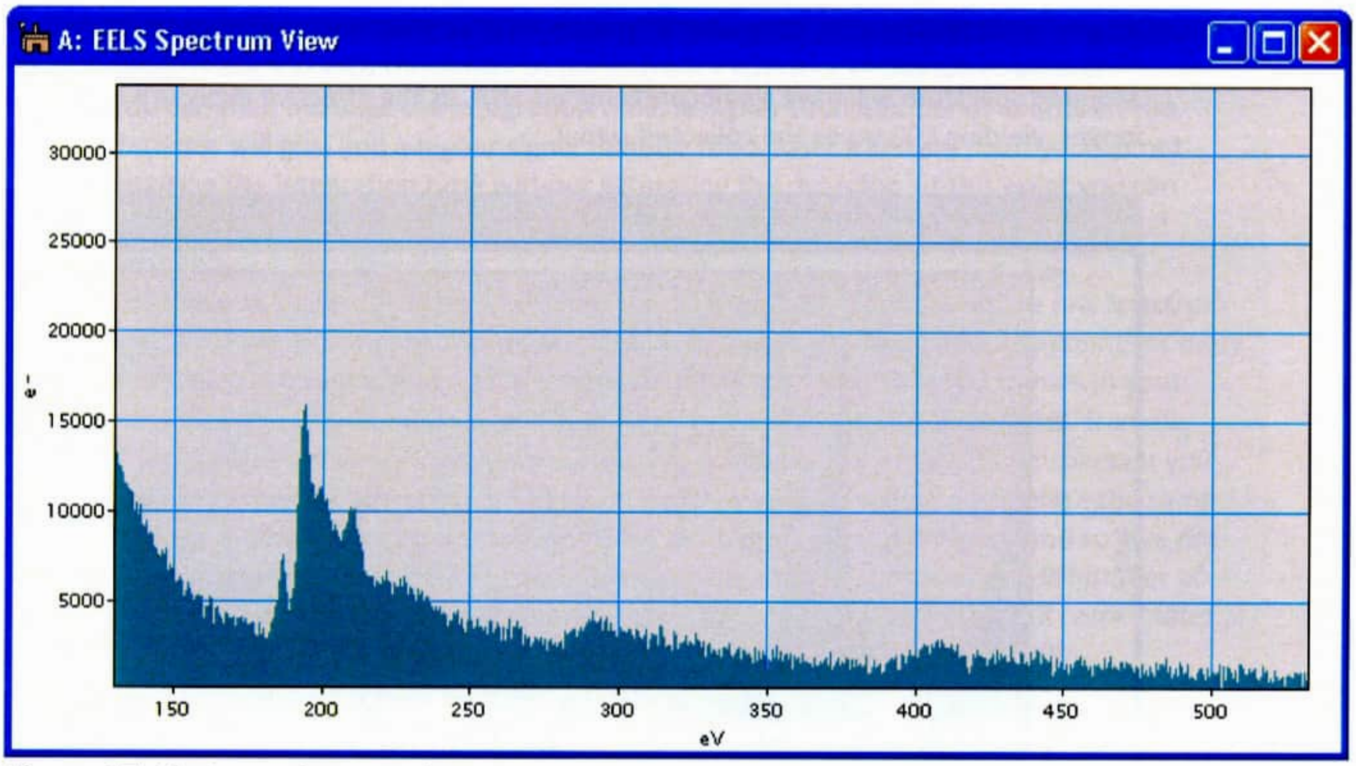



*Figure 17: First core-loss spectrum*

Adjust the diffraction shift setting on the TEM to optimize the signal strength in the spectrum as noted earlier. This should improve the spectrum as shown below.

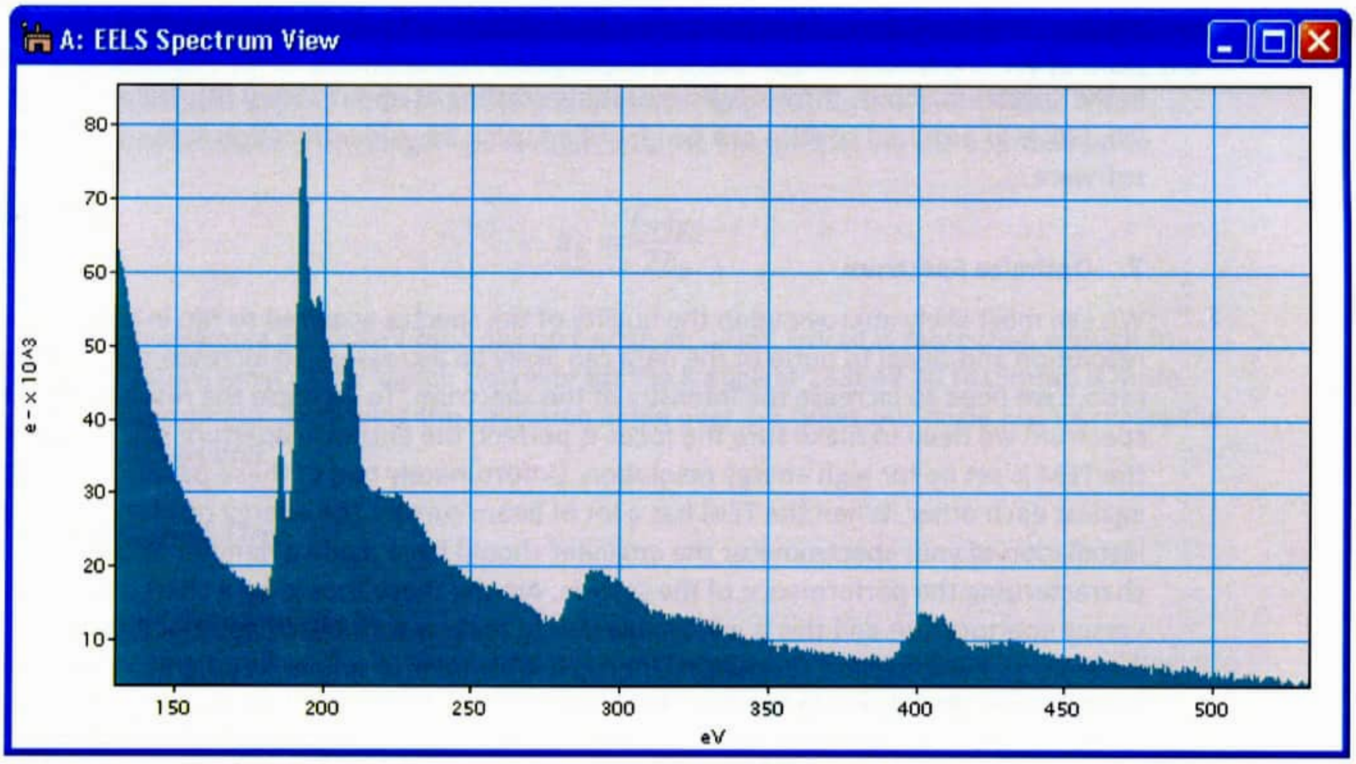



*Figure 18: Core-loss with Diffraction shift optimized*

Because of the lower intensity of core-loss edges, to acquire a good quality spectrum we need to integrate for longer. Stop the view acquisition, enter an exposure time of 1 second in the EELS Acquisition dialog and hit the Acquire button. Specify 10 frames to sum when prompted. The acquired spectrum will have significantly higher SNR, as the effective exposure is now 100 times longer, yielding 100 times the collected signal.

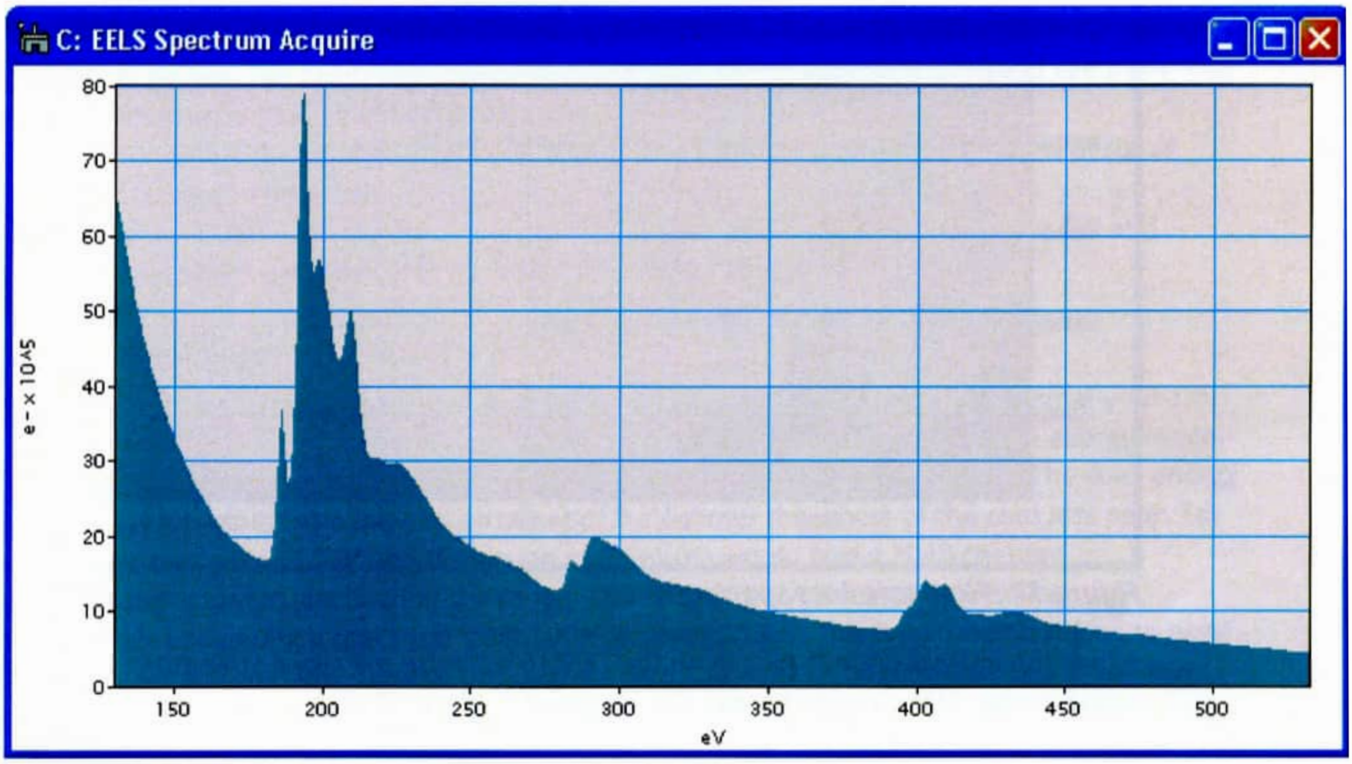



***Figure 19: Core-loss with exposure optimized***

In the spectrum above, three edges are visible starting at about 190eV (B), 285eV (C) and 400eV (N). Edges in acquired spectra can be identified using edge identification in the EELS analysis software.

## 7. Optimize Spectrum

We can most likely improve upon the quality of the spectra acquired so far. In particular the resolution and signal to noise of the data can likely be increased. To increase the signal to noise ratio,6 we need to increase the intensity of the spectrum. To increase the resolution of the spectrum we need to make sure the focus is perfect, the entrance aperture size is suitable and the TEM is set up for high energy resolution. Unfortunately two of these parameters work against each other. When the TEM has a lot of beam current the energy resolution is poor. At installation of your spectrometer the engineer should have made a number of charts characterizing the performance of the system. Among these should be a chart of resolution versus aperture size and this is will enable you to make a sensible tradeoff between the aperture size and desired resolution. There are a number of simple ways to increase the signal to noise ratio of the spectrum.

- First try moving the diffraction pattern a small amount using the projection shift or diffraction shift to maximize the intensity in the spectrum. This just makes sure the beam is aligned with the center of the entrance aperture of the spectrometer
- You can then increase the integration time. A higher total number of counts on the detector will give you a higher signal to noise ratio. After a certain point you cannot increase the integration time without saturating the detector. At this point you can instead sum multiple reads of the detector to improve the signal to noise ratio still further.
- The noise in a gain corrected spectrum comes from both the noise in the raw spectrum and the noise in the gain reference image. This means you need as many counts or more in the gain reference image as in the spectrum. If you try to sum 100 frames in your spectrum you should make sure the gain reference was also taken with 100 frames.
- If you cannot achieve a high enough signal to noise ratio at a particular dispersion you can always reduce the dispersion and try again. A lower dispersion will place the same number of electrons into a smaller number of channels of the detector and so give rise to a higher signal to noise ratio. At 0.05eV/ch the signal is spread over 10x number of pixels than at 0.5eV/ch. If your primary aim is to detect a trace amount of some material then use a lower dispersion.
- The TEM's camera length affects the final signal. This and the entrance aperture determine the collection angle of the spectrometer. Smaller camera lengths increase the collection angle, and increase the amount of signal.

  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 such that it some of the bright field disc is excluded from the spectrometer by the entrance aperture, a significant fraction of the signal can be rejected. To collect the maximum inelastic signal and minimum background a useful rule of thumb is to try to make the collection angle 2 to 3 times the characteristic angle *qc* for the energy-loss. The characteristic scattering angle is related to the energy-loss via the approximation

$$\theta_E = \frac{E_{edge}}{2E_0}$$

  For example at 200keV for a Silicon L edge at 100eV 1mrad is more than enough to capture most of the signal. However for the K edge at 1836eV 10 to15mrad is more appropriate. Increasing the collection angle over 2-3 times this angle just increases the background.

### 6.3.2 Additional Tips

**Specimen Contamination**

On many samples when the beam is condensed on the specimen a small black spot will begin to appear at the beam position. This is particularly noticeable in FEG TEMs since they have much brighter beams. The black material is carbon or silicon based contamination formed from the effect of the intense beam cracking residual hydrocarbons or silicones on the specimen surface. In comparison to the majority of elements carbon and silicon are light so it gives little contrast in the TEM. When it builds up to a thick layer it becomes electron opaque. If you get this sort of

contamination then your EELS spectra will deteriorate with time. This effect can ruin spectra very quickly. Time spent removing some of the contamination will be more effective than adjusting the TEM controls. Three common techniques to clean a sample before it goes into a TEM are:
1) Heat the sample by placing it on the surface of a light bulb to remove the hydrocarbons before putting it in the TEM.
2) Use a liquid nitrogen holder to hold the hydrocarbons / silicones in place.
3) Use a plasma cleaner to remove the hydrocarbons before putting it in the TEM. Note that traditional $O_2$/Ar plasma cleaning can rapidly destroy sensitive materials. A notable example of this is the carbon support commonly used for TEM samples. $H_2$ based plasmas are gentler and effectively remove mobile hydrocarbons without rapidly destroying solid carbon. However, you should experiment and exercise caution, lest you end up with an empty carbon grid.

### 6.3.3 Manual Alignment

To Find the beam by hand in EELS mode, start a live view with short exposure (e.g. 0.05s) in Single Range mode.

- Change the value of Adjust from -2000 to +2000. The beam should be in this range, typically it is within a few hundred volts of where you start.

**To focus lenses by hand**

- Start an EELS Single Range view in the normal way with a sharp feature in the field of view. The zero-loss peak is easiest, but carbon k-edge or transition element L-edges are good candidates as well.
- Click the Focus Spectrum button in the AutoPEELS Align group box and select Manual.
- Select a 2.5 mm entrance aperture (Fixed, 3 mm for SE model)
- Make the spectrum as sharp as possible by adjusting FX and FY.
- Select a large entrance aperture (e.g., 5 mm).
- Focus with SX and SY.
- Go to the aperture you intend to work with.
- Refocus FX and FY.

# 7. Troubleshooting

| Symptom | Action |
|---|---|
| The FilterControl **plug-in** does not recognize the hardware. | • Make sure the Power Supply and the Gatan Instrumentation Bin are turned on.<br>• Check the connection between the Gatan Instrumentation Bin and the Gatan computer (see 4.2.8 Connections Diagram, Figure 10).<br>• Unplug the USB cable and reinsert it. This causes the USB to reinitialize. Make sure the appropriate COM port is recognized in Windows Device Manager under Ports (COM&LPT) it shows up as a USB to UART device. Unplugging the USB cable should cause the GIB COM port to disappear. |
| The ZLP cannot be found. | • Verify the presence of a suitably intense electron beam in the center of the viewing screen.<br>• Make sure the viewing screen is lifted.<br>• Verify that the electron beam is not deflected when the viewing screen is raised: raise the viewing screen a little and verify that the beam can still be observed (especially important for the Philips CM series TEMs where through the "Detector Configuration" menu the beam can be made to deflect to the various detectors when the viewing screen is raised).<br>• Make sure any detectors above the spectrometer are retracted.<br>• If the system has a camera in front of the spectrometer, make sure it is retracted. If this is a Gatan camera with three colored air lines, make sure the red line has pressure and the green line does not. If the camera detector has a single air line, make sure it is pressurized.<br>• In case of the presence of a BF/DF detector: make sure the bright field detector is retracted. Select the annular dark field detector in those systems where only this detector allows the electron beam to enter the spectrometer.<br>• Make sure the spectrometer entrance aperture is not blocked by dirt or a photographic plate stuck in the film mechanism.<br>• If you have multiple cameras, make sure the Enfinium camera is selected.<br>• Make sure there is communication between the FilterControl plug-in and the Gatan Instrumentation Bin (the yellow DATA LED on the front panel of the Gatan Instrumentation Bin should be flashing).<br><br>In the Enfinium Control palette (See Appendix A), verify following:<br>• Verify the Drift Tube Voltage is turned off (unselected) or set to |

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| | 0.<br>• Verify the Primary Energy is set to the nominal operating energy of the TEM. |
| | |
| The Drift Tube Offset does not work. | For all models:<br>• Make sure the Drift Tube Offset is turned on (selected) in the Enfinium Control palette.<br><br>For models with standard Drift Tube:<br>• Check the connection between the Gatan Instrumentation Bin and the spectrometer (see 4.2.3 Connections Diagram, Figure 9). The cable is black and has a high voltage BNC connector on both sides. |
| The pneumatic aperture does not work. (For ER model only) | • Check the presence of pressurized air.<br>• Check the pneumatic connections.<br>• Make sure the electronics controlling the pneumatics are working: listen for puffs of air escaping from the Gatan Instrumentation Bin while changing the aperture size in the AutoPEELS software.<br>• If the spectrometer has not been used for an extended period of time, the O-rings may stick. Apply a high pressure (not to exceed 100 N/cm2) to the colored hoses coming from the aperture mechanism to work the O-rings loose.<br>• Check for water in the air lines. If condensation can be seen in the air lines, then first install a water separator on the air line to the TEM. Make sure this is at a location as cold as or colder than the TEM. Then contact Gatan service to clean the air system and grease the pneumatic parts. |
| The zoom point is different for the various dispersions. | • The zoom point should be set such that when one switches between energy dispersions, the energy-loss feature at pixel column 200 remains stationary.<br>• If this is not the case, then it is likely that engineering alignments have drifted a small amount. Gatan Service may be able to correct this via a remote connection. |
| The spectrometer stops working sometime after it is turned on. | • The spectrometer lenses may be overheating, in which case the protection circuitry will shut the lenses off. In this case, check the water cooling according to the connections diagram. The flow indicator should be set to 10 to 20 liters/hour. To make sure the flow indicator is not stuck at this reading, adjust the regulator valve and see if the flow rate can be adjusted.<br>• Make sure the fans in the Gatan Instrumentation Bin are |

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| | running.<br>• Make sure all the covers are on the GIB. The GIB is air cooled so make sure the vents are clear of dust and not blocked by other equipment. |
| The camera chamber vacuum is poor. | • Compare the current camera chamber vacuum reading (make sure there is no photographic film in the camera chamber) to the ones recorded just before and after the spectrometer was installed (see the Enfinium performance summary filled out at the time of the installation).<br>• Clean the camera chamber door or lid O-ring.<br>• If the camera chamber vacuum is indeed appreciably worse, perform the following tests to locate any leaks:<br>o Move the entrance aperture in/out and observe the camera chamber vacuum. A slight increase in the vacuum reading is normal when the O-rings are sliding, however, it should recover quickly once the movement has stopped. If the vacuum recovers very slowly or not at all, there may be a leak in one of the seals in the entrance aperture.<br>o If fitted, the BF/DF detector can be tested in the same way as the entrance aperture. |
| The spectra have shifted on the cameras. | • Check for changes in the local magnetic environment. Large ferro-magnetic objects may either introduce a magnetic field of their own, or distort the earth magnetic field. In either case, the electron beam going into the spectrometer may be deflected causing the spectra to be shifted. |
| The spectrum drifts or jumps. | • Something may be charging as a result of the exposure to the electron beam. Charging can typically be recognized by a gradual drift in one direction (the charge is building up), followed by a sudden jump in the opposite direction (the charge dissipates with a sudden discharge). If something is indeed charging, then increasing the beam current while keeping the illuminated area on the viewing screen the same should lead to an increased rate of drift. To determine whether the charging occurs in the camera chamber or in the spectrometer, use a selected area aperture to illuminate only a 1-2 Ømm area in the center of the viewing screen. This way all the electrons should enter the spectrometer and none can hit and charge any parts in the camera chamber. If the charging continues, it must be in the spectrometer; if it has stopped, it was in the camera chamber.<br>• The high voltage may be unstable. This can best be verified by observing the caustic at a high camera length. Select the largest condenser aperture and focus the illumination as well as possible. A small change in the high voltage will change the size of the caustic. It may be useful to "calibrate" the change in the |

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| | caustic. To do so, change the high tension of the microscope on the microscope control by 10 eV or minimum amount possible. Notice the resulting change of the caustic. Note that any change in the high voltage should change the focus of the image. A change of ΔE volts in the high voltage of nominal value E0 volts, results in a change Δf of the focal length given by:<br>$$\Delta f = C_C \frac{\Delta E}{Eo}$$<br>where Cc is the chromatic aberration coefficient of the objective lens. |
| The energy resolution is poor. | • Make sure the spectrum is properly focused.<br>• Check for a change in magnetic stray fields by adjusting AC Comp A and B in the Adjust Window in the Enfinium Control palette with exposures of greater than 50 msec.<br>• For thermionic TEMs (LaB6 or tungsten sources, non-FEGs), make sure the gun filament heating is set properly. For normal use, the filament should be just saturated; for optimal energy resolution, the filament should be undersaturated for LaB6 or tungsten.<br>• Check the emission current and compare it to the emission current used at installation, to get the demonstrated energy resolution. The lower the emission current, the fewer electrons and the better the energy resolution on the zero loss. However, the achieved energy resolution on an edge is affected by a signal to noise, the physics of the process creating the edge, low frequency interference, and other factors.<br>• Make sure the size of the projector crossover is not limiting the energy resolution. The projector crossover size can be reduced by selecting a smaller illumination area on the sample and/or increasing the camera length.<br>• Make sure the high voltage is stable: observe the caustic as described in "The image or spectrum drifts or jumps." |

**CAUTION**: Never attach any magnetic objects to any part of the spectrometer. Some materials used in the spectrometer are magnetic to ensure good shielding from external stray fields. If magnetized with a strong magnetic field, these materials may retain some magnetization even after the magnetic field has been removed. This residual magnetic field may cause permanent shifts of the image and spectra.

# 8. Care and Maintenance

This chapter contains some general tips on the care and maintenance of your Enfinium spectrometer

## 8.1 General Precautions

The following are some general precautions users should adhere to in the care and maintenance of their spectrometer.

### *8.1.1 Peltier cooler maintenance*

- The Peltier cooler for the camera is controlled through software.
- The Peltier cooler should not be on if the cooling water is not flowing. The Peltier cooler consumes about 12 W of power. If this heat is not removed by the cooling water, there is a possibility that damage to the Peltier cooler and/or the CCD may occur.
- Check the flow of cooling water periodically. If the flow rate of the cooling water deviates significantly from the value originally set, make sure the lines are not obstructed and adjust the pressure regulator to bring the flow back to the original level. Remember, if the water flow stops while the Peltier cooler is on, damage to the camera head may result.
- The Peltier cooler should not be left on when the TEM cooling water is turned off or fails. The Peltier cooler is cooled by water from the TEM chiller. If the Chiller is turned off or fails for a long period, there could be severe damage to the camera. Be aware that for some microscopes the cooling system turns off in the Stand By mode. Be sure the cooler of the camera is also turned off in that situation.
- Turn off the Peltier cooler if the microscope viewing chamber is to remain at air pressure for a long period (more than 30 minutes to an hour). When isolated by the gate valve, the CCD vacuum is good for about half an hour, after which some frosting on the CCD may occur if the CCD is kept cold. A small amount of ice will sublimate away once high vacuum is restored in the viewing chamber and the CCD gate valve opens. Removing large amounts of condensation will require the electronics to be switched off for about 30 minutes after the vacuum in the viewing chamber is restored, so that the CCD warms up while it is in a good vacuum.
- Turning off the power to the camera control unit will ensure the power to the Peltier cooler is off.

### *8.1.2 Scintillator maintenance and precautions*

- Minimize exposing the scintillator to the electron beam when the camera is not in use.
- It is particularly important to protect the camera from being unnecessarily exposed to an intense unscattered beam. As a general rule, if you avoid conditions which would

saturate any part of the sensor area in less than 0.1 sec. If you follow this rule then the scintillator lifetime will probably exceed the lifetime of the rest of the system.

- Minimize exposing the scintillator/CCD to high beam intensity when acquiring diffraction patterns or spectra. There is no defined cutoff where the scintillator will be damaged. Rather, damage can result from prolonged exposure of the scintillator to a very high beam intensity (above 30,000 counts in binned by 1 mode and 60,000 counts in binned by 2 and higher modes). If you have no saturation with a short exposure, then there is no danger. You can then increase the exposure time and still not harm the scintillator as it is the beam intensity on the scintillator that causes the problem and not the accumulated signal in the CCD.
- It is recommended to use beam stop to block the central spot when acquiring SAED diffraction patterns. If you think you have damaged the scintillator, take an unprocessed image of uniform illumination. If the spot turns out dark, you have damaged the scintillator! If it is bright, spread out the beam and set the illumination so you have about 10,000 counts/sec (in binned by 2 mode). Expose the scintillator for 3 minutes and take another picture. The bright area should be gone ( or significantly reduced) and the scintillator should operate normally.
- Warm the scintillator periodically. Some microscopes have a significant vapor pressure of pump oil in the viewing chamber. Since the camera is maintained at temperatures below -20ºC during use, it can act as a cold trap and accumulate an oil film. While thin this film does not affect spectrometer performance, but if it becomes thicker or coalesces into droplets, stronger and stronger gain correction will become necessary.
- Other problems caused by the oil film are charging and the sticking of dirt which could otherwise be blown off easily. If left for too long, the hydrocarbons chains in the oil will be cracked by the electron beam making them nonvolatile and un-dissolvable. Then it is necessary to send the camera back to have the scintillator serviced by Gatan. Fortunately, these problems can be avoided by adhering to the practice described below.
- If you have a clean vacuum in the camera chamber area, warm the scintillator once or twice a month to 35 degrees Celsius by using the menu item "Camera | Temperature". If you have back-streaming from your pump, you will need to do it once a week. The camera should only be warmed for 15 hours or overnight, but not longer. If the camera is used sufficiently without periodic warming, the electron beam can harden the trapped oil making the scintillator difficult to clean. If oil accumulates even with regular warming of the scintillator, it is recommended that the vacuum system be checked. Note that the oil contamination will not be as visible at higher voltages (e.g., >120 kV than at lower voltages (e.g., 40 kV), so that a problem can exist for some time before it is noticed, by which time the beam might already have hardened the film. This fact can be used to diagnose the presence of oil. It is recommended the user takes an image at a low kV periodically to confirm the scintillator is still clean.

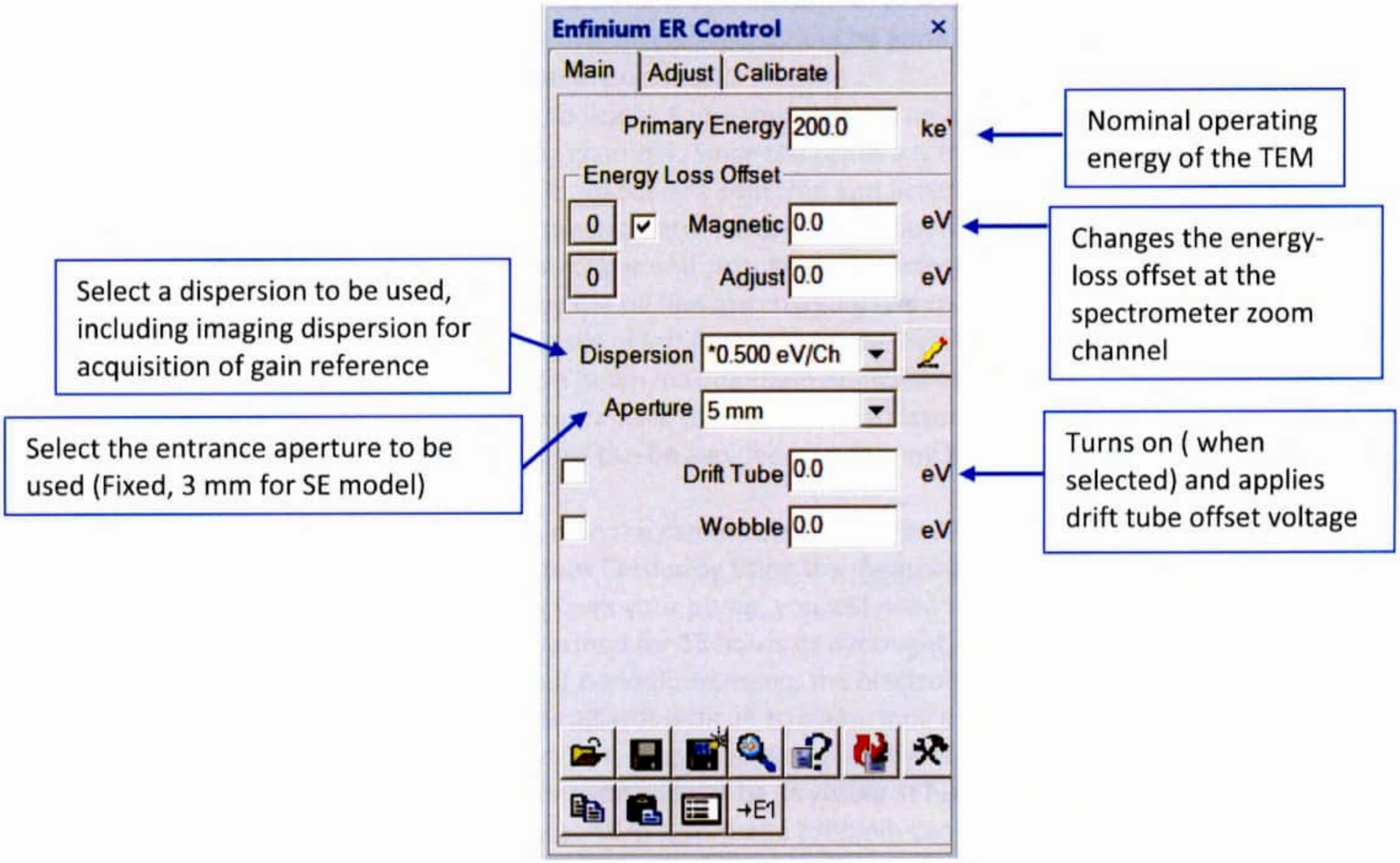
  If the surface of the scintillator becomes dirty or scratched, ship the camera to Gatan for service. Do not attempt to clean the scintillator yourself as damage can result.

# Appendices

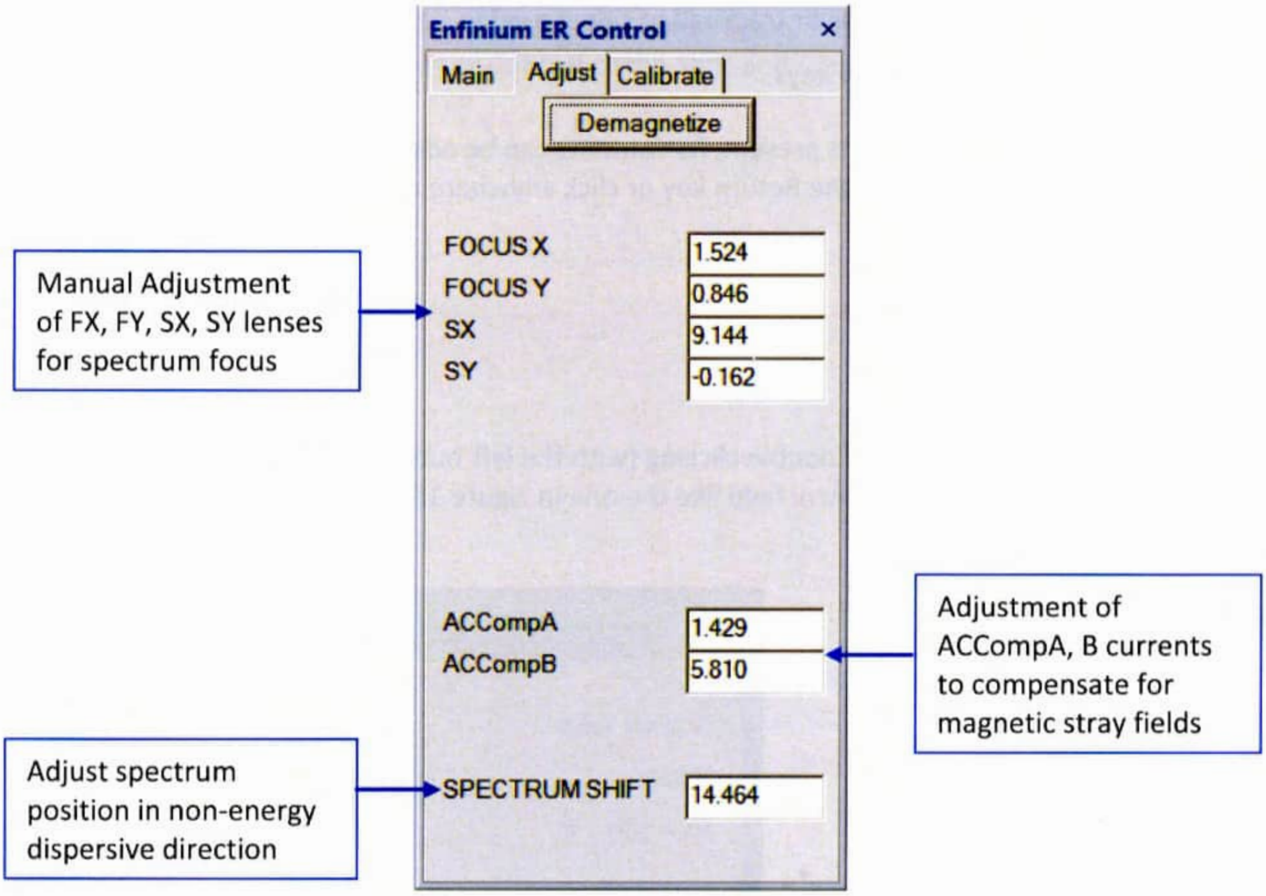
# A. Enfinium Control palette access as a Power User

The Enfinium Control palette is mainly intended for use by installation and service engineers, but it also serves some routine diagnostic purposes. The purpose of this section is to give you a brief introduction to the operation of Enfinium Control so that you can use it for some basic troubleshooting, if needed.

Enfinium Control Palette can be accessed as a "Power User" under Help | User Mode sub-menu. As a Power User, you gain access to low-level instrument control for elements such as Drift Tube voltage or FOCUS X, FOCUS Y lenses.




***Main Pane***




***Adjust Pane***

***Figure 20 Enfinium Control Palette***

- Please note that the detailed lens settings shown in the Adjust window will vary significantly from one installation to another, so please do not be concerned if your system shows different settings.

- Alternative way of focusing spectrum manually is to access FX, FY, SX, SY lenses via Enfinium Control palette |Adjust pane as shown in Figure 18. Lens strength can be adjusted by (1) entering a new value, (2) using the mouse, or (3) using the arrow keys.

- When magnetic stray fields change, you may need to adjust AC Comp A and B in the Adjust pane with exposures of greater than 50 msec

## *Entering Values*

There are three ways in which an instrument element, such as a lens or the high voltage of the TEM, can be adjusted using the Enfinium Control software:

- Entering a value in a text box

- Using the mouse or trackball to select a value
- Using the arrow keys

If a numeric entry field is present, its contents can be edited as standard text. Once the new value is entered, press the Return key or click anywhere outside the numeric field to activate the change.

## *Using the Mouse*

This method consists of double clicking (with the left button on the mouse) on the label of the element such that a control field like the one in Figure 18 appears.

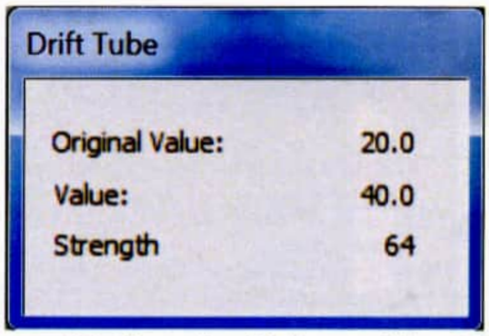



***Figure 21 Changing Values with a Mouse***

Now moving the mouse left and right will adjust the value of the element (drift tube voltage, in this case) with a step size equal to Strength / 100. For instance, a Strength setting of 2 will result in a step size of 0.02. The Strength setting is adjustable between 1 and 1024 (in factors of 2) by pressing the - or + key on the keyboard or using the mouse wheel. Once the element has been adjusted, the control field can be made to disappear by clicking the mouse's left button. Press the ESC key to exit without saving the adjustment.

## *Using the Keyboard*

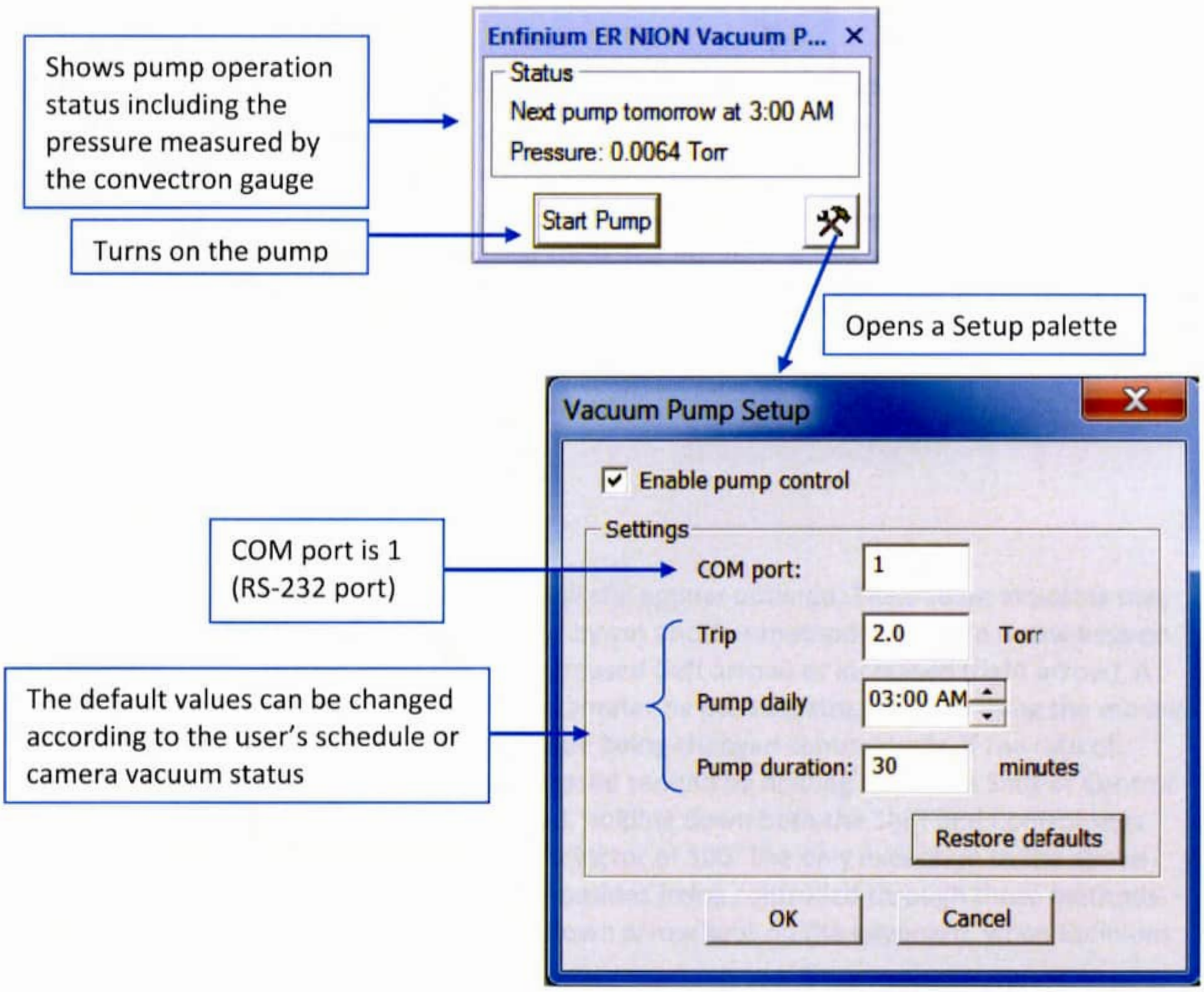
After the control field disappears, the label will still appear outlined. The outline indicates that the element is still active and can be adjusted by yet another method. Using the arrow keys on the keyboard, the element's value can be decreased (left arrow) or increased (right arrow). A single click of one of the keys changes the parameter by the step size, as when using the mouse. Holding the arrow key down results in the value being changed continuously. If the rate of change is too small, the step size can be increased tenfold by holding down the Shift or Control key. If an even larger rate of change is desired, holding down both the Shift and Control keys simultaneously will increase the step size by a factor of 100. The only exception to the above convention is the energy shift control, which besides being controlled through these methods can also be controlled by pressing the up or down arrow keys on the keyboard, when Enfinium

Control is selected and the results window is not. Once again, the step size can be increased by a factor of 10 or 100 when the Shift or Control or the Shift and Control keys are held down.

# B. Enfinium ER HiVac Vacuum Pump palette (for HITACHI and NION dedicated STEMs)

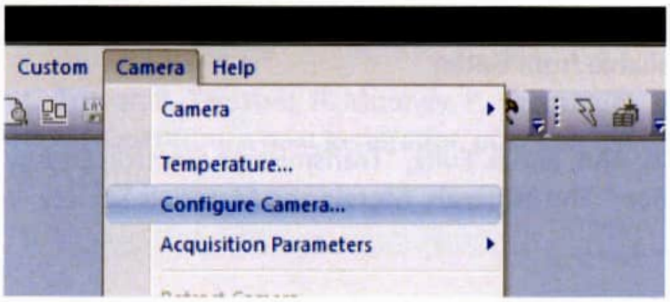
Spectrometers installed on HITACHI and NION microscopes have a separate camera vacuum maintained by a scroll pump. Remote pump operation is enabled via RS-232 interface and through the Enfinium HiVac Vacuum Pump Control palette. The use of this palette is mainly intended for use by installation and service engineers, but the default pump operation values can be changed, if needed.

Enfinium HiVac Vacuum Pump Control Palette can be accessed as a "Power User" set by going to the Help | User Mode sub-menu. Under default condition, the scroll pump is programmed to turn on (1) daily at 3:00 AM for 30 minutes, and (2) when camera vacuum measured by the convection gauge exceeds the trip pressure of 2.0 Torr. A user may wish to force a cycle of the pump before starting a long experiment so the pump does not turn on during the collection of data.

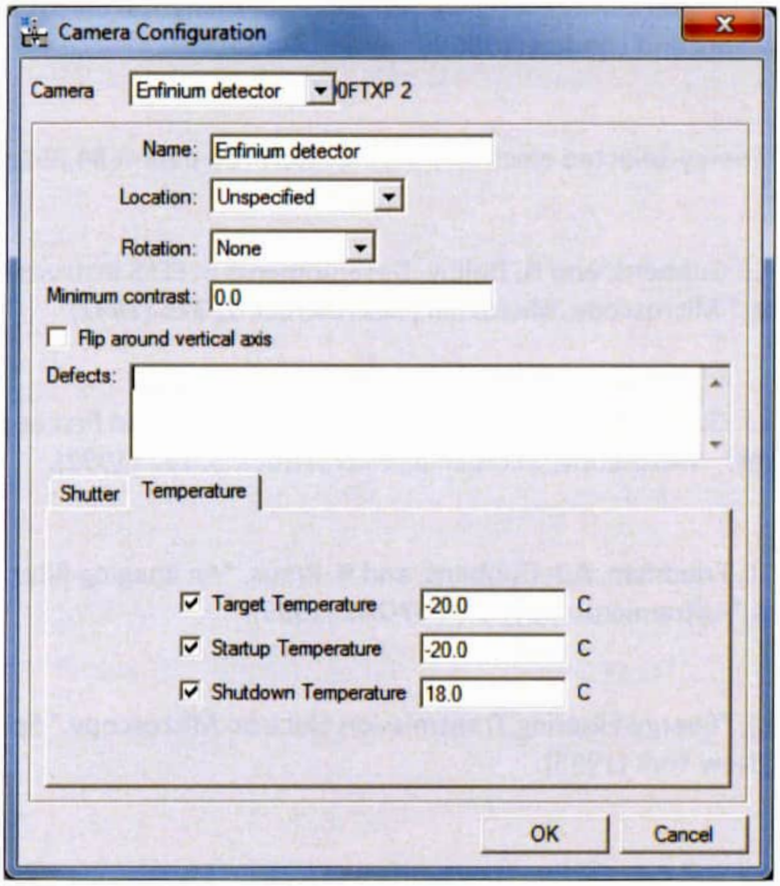


# C. Temperature control of the detector

The detector temperature can be controlled from two places, either directly by going to the camera menu and picking "temperature" or via the camera configuration palette. This is accessed via the camera menu as well but brings up a palate.




The camera configuration palate has tabs at the bottom, for the shutter and temperature. The preferred temperature at 3 different situations is set here. "Target", or the current set point. "Startup", what DM will do when it is started, and "Shutdown", what the camera controller is set to do just before DM closes.



# D. References

The following reference materials contain detailed information about the GIF Quantum and its applications, and should prove very helpful to GIF users in fully understanding both theoretical and practical operation and concepts.


1. C.C. Ahn and O.L. Krivanek, "EELS Atlas," published by Arizona State University and Gatan (1983) and available from Gatan.

2. M.M. Disko, C.C. Ahn, and B. Fultz, "Transmission Electron Energy Loss Spectrometry in Materials Science," The Minerals, Metals and Materials Society, Warrendale (1992).

3. R.F. Egerton, "Electron Energy-Loss Spectroscopy in the Electron Microscope," Plenum Press, 2nd edition, New York (1996).

4. A.J. Gubbens and O.L. Krivanek, "Applications of a post-column imaging filter in biology and materials science," Ultramicroscopy 51, 146 (1993).

5. D.C. Joy, Romig, and Goldstein (eds.), "Principles of Analytical Electron Microscopy." Plenum Press., New York and London (1986).

6. Krivanek, "Energy-selected electron imaging filter," US patent #4,851,670, 1989.

7. Krivanek, A.J. Gubbens, and N. Dellby, Developments in EELS instrumentation for spectroscopy and imaging," Microscopy, Microanal., Microstruct. 2, 315 (1991).

8. Krivanek, A.J. Gubbens, N. Dellby, and C.E Meyer, "Design and first applications of a post-column imaging filter," Microscopy, Microanal., Microstruct. 3, 187 (1992).

9. Krivanek, S.L. Friedman, A.J. Gubbens, and B. Kraus, "An imaging filter for biological applications," Ultramicroscopy, 59, 267-282 (1995).

10. Reimer (Ed.), "Energy-Filtering Transmission Electron Microscopy," Springer-Verlag, Berlin Heidelberg New York (1995).

11. D.B. Williams and C.B. Carter, "Transmission Electron Microscopy," Plenum Press, New York (1996)

12. A.J. Gubbens, H.A. Brink, M.K. Kundmann, S.L. Friedman, and O.L. Krivanek "A Post-Column Imaging Filter with a $2048^2$-Pixel Slow-Scan CCD Camera," Micron 29, 81 (1998).

13. H.A. Brink, M.M.G. Barfels, R.P. Burgner, and B.N. Edwards, "A sub-50 meV spectrometer and energy filter for use in combination with 200 kV monochromated (S)TEMs," Ultramicroscopy, 96, 367 (2003).

14. A. Gubbens, M. Barfels, C. Trevor, R. Twesten, P. Mooney, P. Thomas, N. Menon, B. Kraus, C. Mao and B. McGinn, "The GIF Quantum, a next generation post-column imaging energy filter" Ultramicroscopy, **110**, 962 (2010)

# E. Glossary

| | |
|---|---|
| ***BF/DF*** | Bright field/dark field |
| ***CCD*** | Charge coupled device |
| ***DM*** | DigitalMicrograph, the core software application of the Gatan Microscopy Suite for control and analysis of TEM data |
| ***Drift Tube Offset*** | A voltage added to part of the GIF to move the spectrum by that exact amount |
| ***EELS*** | Electron Energy Loss Spectroscopy |
| ***EFTEM*** | Energy Filtered Transmission Electron Microscope |
| ***Elastic scattering*** | Scattering without a change in energy (for example, diffraction) |
| ***EXELFS*** | Extended electron energy loss fine structure |
| ***FilterControl*** | Software application specifically designed to control the Gatan Imaging Filter |
| ***GIF*** | Gatan Imaging Filter |
| ***GIF Quantum*** | Latest generation Gatan Imaging Filter |
| ***GMS*** | Gatan Microscopy Suite, complete set of interconnected applications and plug-ins for control and analysis of TEM data |
| ***HT*** | High Tension |
| ***Inelastic scattering*** | Scattering with a change in energy |
| ***Jump ratio*** | Ratio of background just before an edge to the highest point on the edge |
| ***STEM*** | Scanning Transmission Electron Microscope |
| ***TEM*** | Transmission Electron Microscope |
| ***ZLP or Zero Loss Peak*** | Feature in spectrum from the electrons that have either gone through the sample without interacting or have scattered elastically |

# F. Size of Projector Crossover

It is important to be mindful of the microscope operating conditions, especially when working at very low image magnifications or very short camera lengths, because they can affect the spectrometer's performance. The spectrometer focuses the projector crossover into the energy dispersion plane used for energy selection, which is described in detail in Section 2.3.1 and Section 2.3.2.

The projector crossover size increases at low image magnifications and large objective aperture selection OR at short camera lengths and large illumination spot sizes.

The following tables serve as a general guide to how microscope operating conditions can degrade the energy resolution of the spectrometer by what is called spectrum-image mixing.

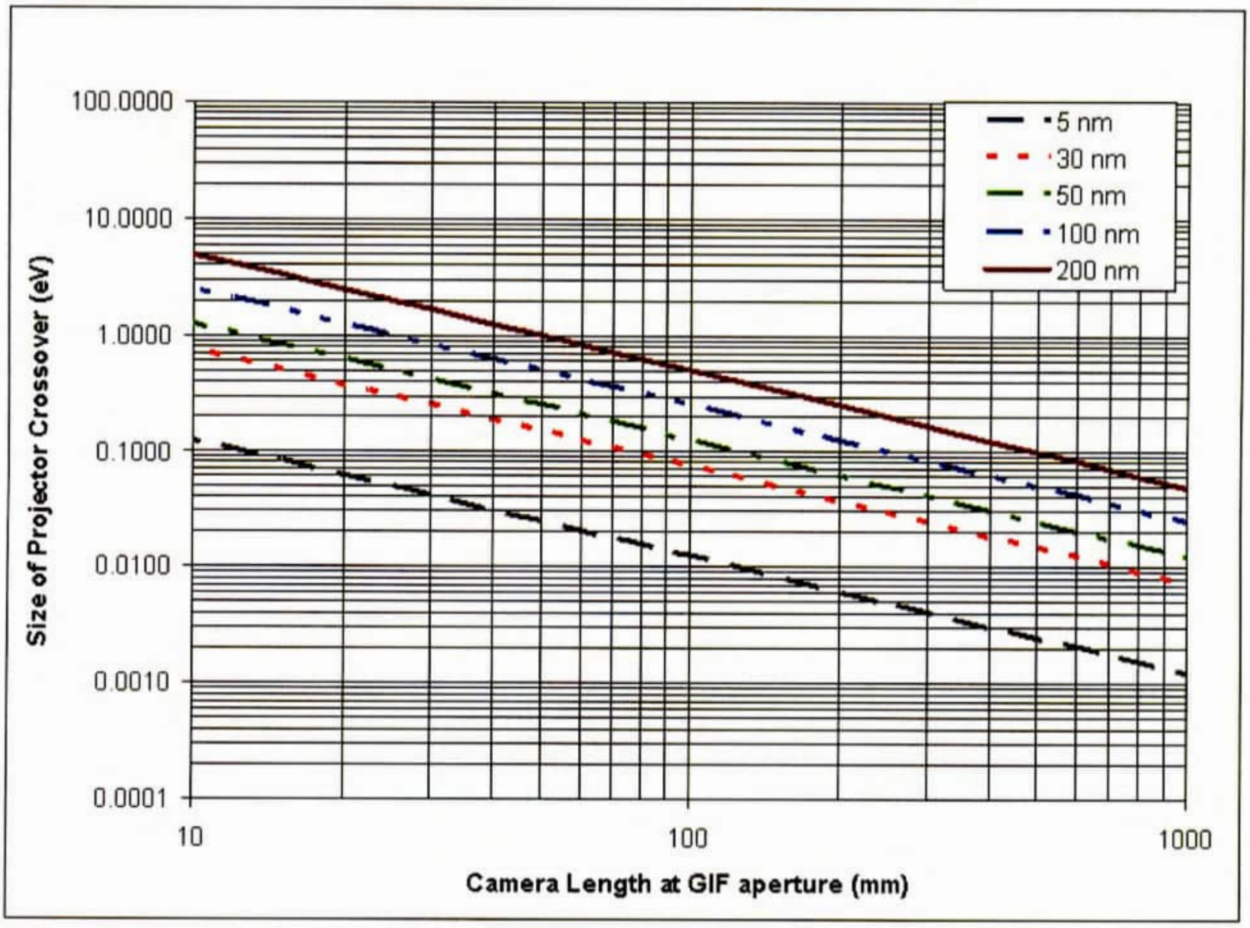



***Figure 22 Projector Crossover Size vs. Camera Length at 200 kV***

***This graph only serves as a guideline since it does not account for differences in TEM models.***

Figure 19 represents the size of the projector crossover in the spectrum plane when operating the microscope in diffraction mode. In this mode the projector crossover contains an image which is coupled into the energy dispersion plane of the filter.

For longer camera lengths or smaller collection angles, typically used for EELS, spectrum image mixing is insignificant even for relatively large spot sizes as seen in Figure 19. The calculated diameter of the projector crossover size in the filter spectrum plane is given by:

$$D_p = \frac{d_s \cdot h}{L} \cdot K_s$$

where

- $d_S$ is the diameter of the illuminated specimen area
- h is the distance from the projector crossover to the TEM viewing screen
- L is the TEM camera length at the viewing screen
- $K_S$ is the spectrometer energy dispersion and magnification factor

However, when very large collection angles are used such as for energy-filtered CBED imaging, it is necessary to account for the effect of post-specimen objective lens spherical aberrations. The diameter of the projector crossover is the sum of the spherical aberration disc and the illumination spot size. See Figure 20. The smallest aberration disc at a minimum defocus (Lichte Focus) is achieved by carefully tuning Focus X. At this condition the projector crossover size in the filter spectrum plane is given by

$$D_p = \frac{d_s \cdot h}{L} \cdot K_s + \frac{D_{Cs} \cdot h}{L} \cdot K_s$$

where

- $D_{Cs}$ is the minimum spherical aberration disc containing 100% of the electron intensity

$$\boldsymbol{D_{Cs}} = \frac{\boldsymbol{Cs \cdot \beta^3}}{\boldsymbol{2}}$$

- Cs is the objective lens spherical aberration coefficient
- $\beta$ is the maximum collection angle at the filter entrance aperture

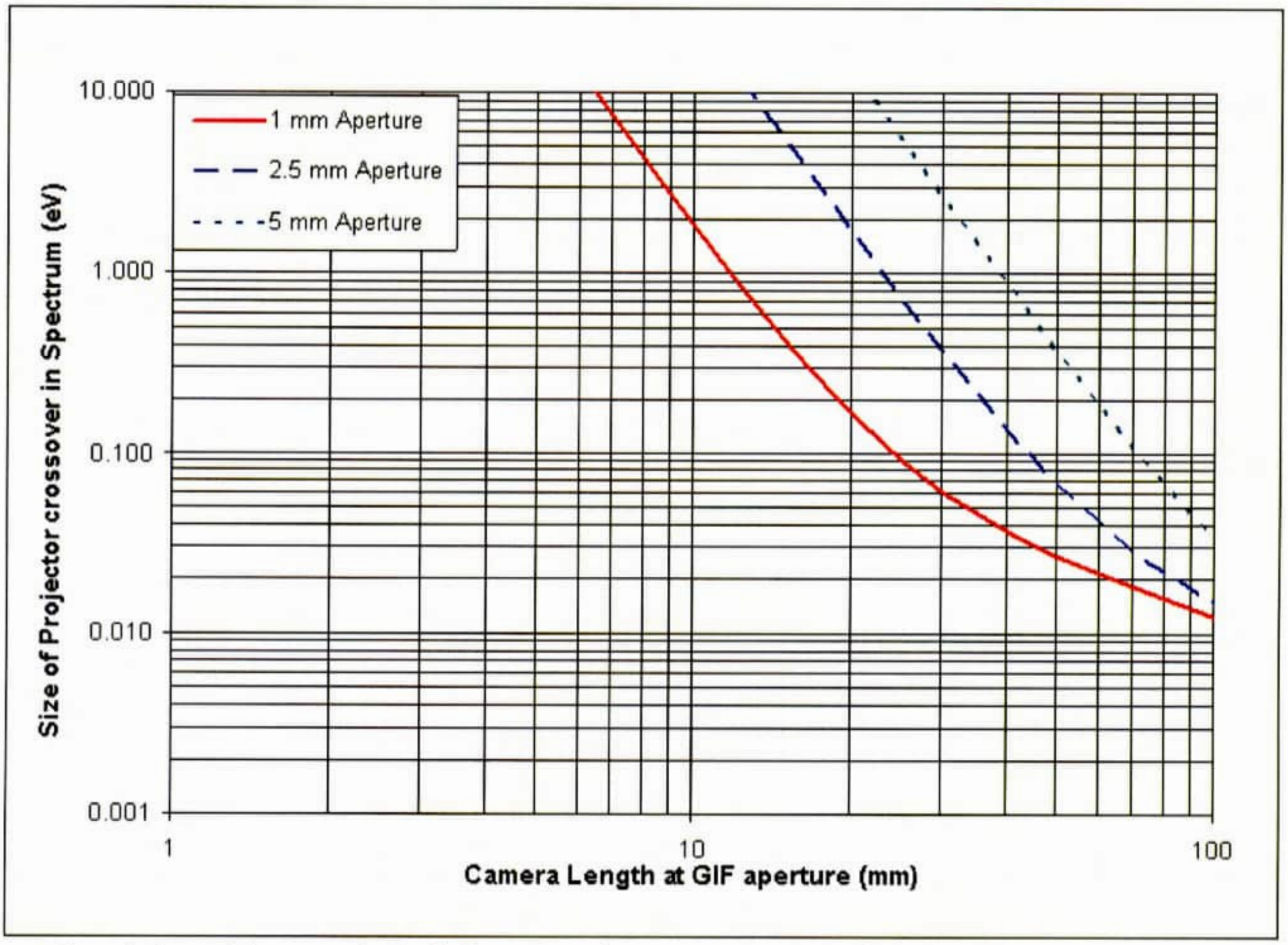
Note that the actual degradation in energy resolution is best measured on a case-by-case basis. These figures serve only as a guide.

For monochromated TEMs achieving sub-0.2 eV energy resolution, the post-specimen objective lens spherical aberrations could compromise the spectroscopy energy resolution if larger collection angles are required such as for STEM-EELS.

One can similarly estimate the spherical aberration effect on the FWHM of the zero-loss-peak in spectroscopy. The width of the spherical aberration containing 50% of the current density has been analytically derived by (Barth and Kruit, 1996 Optik (3) 101.) and is given by

$$D_{S50} = (1/2)^{\frac{5}{2}} \cdot Cs \cdot \beta^3$$

where




• $D_s$ 50 is the minimum spherical aberration disc containing 50% of the total current

***Figure 23 Project Crossover Size including the objective lens spherical aberration effect for a Cs = 1.1 mm, spot size of 5 nm at 200 kV***

***This graph only serves as a guideline since it does not account for differences in TEM models.***

# G. Dispersion

| Primary Energy (kV) | Spectrometer Constant Ks |
|---|---|
| 60 | 0.10 |
| 80 | 0.13 |
| 100 | 0.16 |
| 120 | 0.19 |
| 200 | 0.30 |
| 300 | 0.43 |

***Figure 24 Dispersion Table***

# Index

# Product Warranty

**Warranty**

Gatan, Inc. ("Gatan") warrants that each product and component thereof that is manufactured by Gatan ("Product") shall be free from defects in materials and workmanship for one (1) year following the date of shipment ("Warranty Period"). Gatan warrants that the Product meets Gatan's published specifications at the time of shipment from its factory. These warranties are provided only to the original purchaser of the Product ("Purchaser"), and are not transferable in any way.

In the event that any Product does not meet the warranties above, Gatan will, at its option, either repair or replace the defective Product or component with a conforming Product or component. The repair or replacement of a Product or component under warranty does not extend the Warranty Period. With the exception of consumable and maintenance components, replacement components shall be warranted to be free from defects in materials and workmanship for the duration of the Warranty Period or ninety (90) days, whichever is longer.

For specimen preparation equipment and specimen holders, Gatan will correct any defective Product either by repairing the defective Product or component at Gatan's facility, or by replacing the defective Product or component. The party shipping the Product or component shall be responsible for shipping costs. Unless otherwise specified, for defective Products that attach to the column (cameras, DigiScan, GIF, and PEELS) and that are subject to repair under the warranties, all repairs will be performed by Gatan at the customer site.

Instruments, parts, and accessories not manufactured by Gatan will be warranted by Gatan only to the extent that any such instruments, parts, and accessories are under warranty from the manufacturer. Gatan makes no warranty whatsoever concerning instruments, parts, or any related products not manufactured by Gatan.

**Purchaser Responsibilities**

The Purchaser bears the following responsibilities with regard to maintaining the warranties. Failure to perform the obligations below shall automatically void the warranties. The Purchaser shall:

1. Product is installed according to Gatan specifications and procedures by qualified personnel.
2. Perform the routine maintenance and cleaning at the required intervals as specified in Gatan's operating manuals.
3. Use replacement parts provided by Gatan.
4. Use Gatan or Gatan-approved consumables.
5. Provide Gatan authorized service representatives access to the Products during normal Gatan working hours to perform service.
6. Provide adequate and safe working space around the Products for servicing by Gatan authorized service representatives.
7. Provide access to, and use of, all information and facilities determined necessary by Gatan to service the Products.
8. Use only the Gatan factory service resources for off-site repair work.

**Repair and Replacement**

Any Product or component returned to Gatan for examination or repair must have Gatan's prior approval. The Purchaser must request a Return Material Authorization ("RMA") number through the Gatan service department or directly from Gatan's Warrendale facility at 724-776-5260. If the Product or component is not under warranty, Gatan will provide an estimate of the repair cost and the Purchaser must provide a purchase order ("PO") before any repair work will be performed. Any expedited repair or replacement shall also require a PO. The returned Product or component should be shipped at the expense of the Purchaser with the RMA number clearly marked on the exterior of the shipping container and on the enclosed shipping documents. If the returned Product or component is found to be defective and is covered under warranty, the Product or component shall be returned by Gatan at Gatan's cost. If the returned Product or component is found to be not defective or is not covered under warranty, the

Purchaser shall be responsible for shipping costs.

**Warranty Limitations**

The warranties do not cover:

1. Components, parts, and accessories that are expendable or consumable in the normal operation of the Product.
2. Any loss, damage, and/or malfunction that results from shipping, storage, accident (fire, flood, or similar catastrophes normally covered by insurance), abuse, alteration, misuse, neglect, or breakage or abuse of parts.
3. Operation other than in accordance with correct operational procedures and environmental and electrical specifications.
4. Performance to specifications or safety of use (including X-ray emissions) if the Product is physically installed on, used in conjunction with, or used as part of a third party's equipment, or is not installed by a Gatan service engineer.
5. Performance to specifications or safety of use (including X-ray emissions) as a result of the use of Gatan's equipment with that of a third party.
6. Modification of, or tampering with, the Product.
7. Improper or inadequate care, maintenance, adjustment, service, or calibration not performed by Gatan qualified personnel.
8. Contamination or leaks not caused by Gatan.
9. Any loss, damage, and/or malfunction that results from any software, hardware, interfaces, or consumables not specified or approved by Gatan.

**Warranty Exclusions**

In the course of normal use and maintenance, certain parts have finite lifetimes. For this reason, the consumables and maintenance parts as specified in Gatan's operating manuals have a ninety (90) day warranty.

**Post Warranty Support and Product Obsolescence**

After the expiration of the Warranty Period, Gatan will provide service support for Products at service labor rates and parts pricing in effect at the time of the repair. Gatan will continue to provide billable service support for the Products for a period of three (3) years after discontinuance or design obsolescence by Gatan. After this three (3) year period, any service support offered will be at the sole discretion of Gatan.

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THE WARRANTIES HEREIN ARE IN LIEU OF, AND EXCLUDE ALL OTHER, EXPRESSED OR IMPLIED WARRANTIES, INCLUDING, BUT NOT LIMITED TO, WARRANTIES OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE. UNDER NO CIRCUMSTANCES WILL GATAN BE LIABLE FOR ANY INDIRECT, SPECIAL, INCIDENTAL, OR CONSEQUENTIAL DAMAGES (INCLUDING LOST PROFITS), OR LOSS OF ANY KIND, WHETHER BASED ON WARRANTY, INDEMNIFICATION, CONTRACT, TORT, OR ANY OTHER LEGAL THEORY. THE LIMITS OF GATAN'S LIABILITY IN ANY DISPUTE SHALL BE THE PRICE RECEIVED FROM THE PURCHASER FOR THE SPECIFIC EQUIPMENT AT ISSUE. GATAN'S TOTAL LIABILITY FOR ANY AND ALL LOSSES AND DAMAGES ARISING OUT OF ANY AND ALL CAUSES WHATSOEVER INCLUDING, WITHOUT LIMITATION, DEFECTS IN THE PRODUCTS OR SERVICES, BREACH OF ANY OBLIGATION, OR ANY INDEMNIFICATION OBLIGATION HEREUNDER SHALL IN NO EVENT EXCEED THE PURCHASE PRICE OF THE AFFECTED PRODUCT.

**Governing Law**

THE LAWS OF THE STATE OF PENNSYLVANIA APPLY TO ALL ASPECTS OF GATAN'S WARRANTIES.

# Declaration of Conformity

## YKClowder

PO Box 320411
San Francisco, CA 94132 USA
P: (415) 859.9830 F: (415) 586.1573
www.YKClowder.com

## Attestation of Conformity

June 06, 2012 **Attestation No.: ATS12-100**

**Applicant:** Gatan Inc.
5794 W. Las Positas
Pleasanton, CA 94588 USA

**Products:** Quantum and Enfinium GIF Systems
Model Nos. 96X.XXX & 97X.XXX

We do hereby declare that the test reports submitted by the applicant for the subject products have been reviewed, assessed, and found to comply with the following Standards:

EN 55011:2007, EN 61326-1:2006, and IEC 61010-1, 2nd Edition 2002-2.

Conformances to those Standards are required for Directives 2006/95/EC and 2004/108/EC.

Issued on this date in San Francisco, CA

Moe Said

Moe Said
General Manager