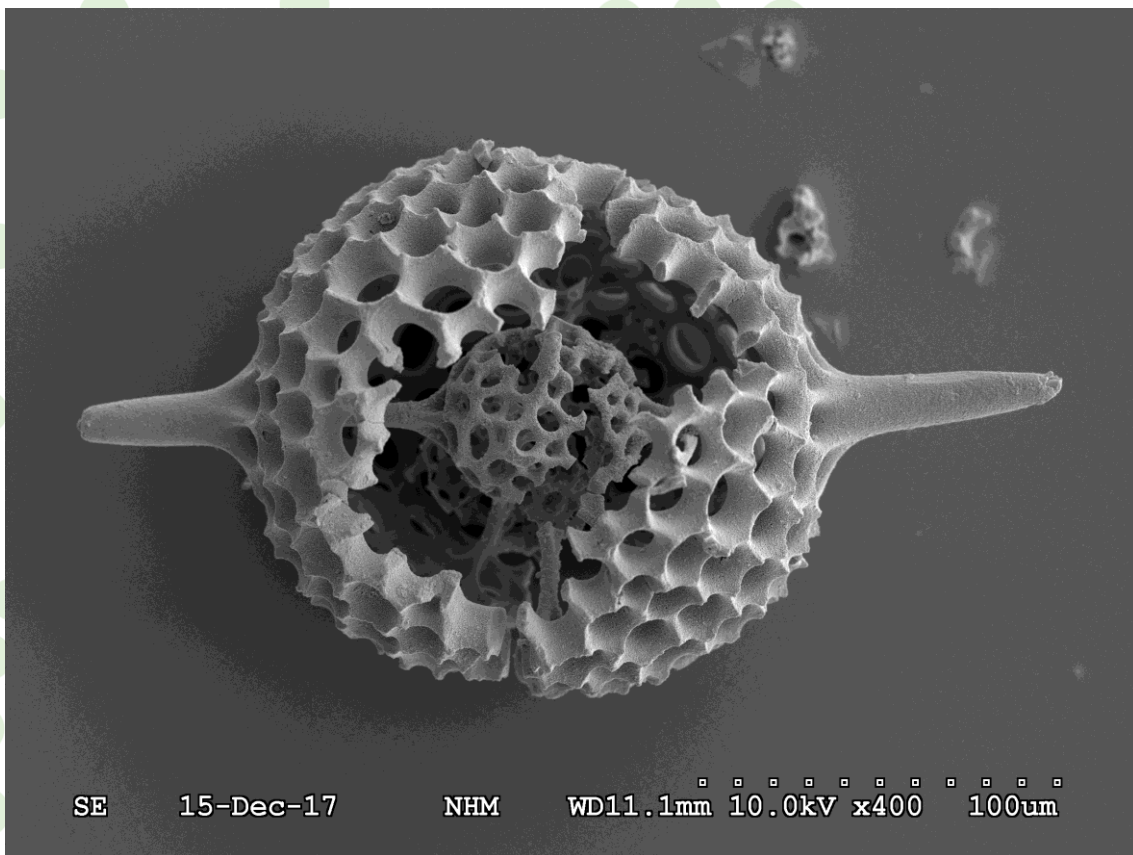


Hitachi S-3600N Scanning Electron Microscope

User instructions



Geo Lab

Department of technical and scientific conservation |
Seksjon for konservering og forskningsteknikk

U562 – Analyzelaboratorie SEM, XRD, CT

- **Scanning electron microscopy**
Hitachi S-3600N scanning electron microscope (SEM)
- **X-ray diffraction**
Siemens D5005 powder X-ray diffractometer (PXRD)
Rigaku Dual Beam Synergy-S single-crystal X-ray diffractometer (SXRD)
- **Computed tomography**
Nikon XT H225 ST micro computed tomograph (micro-CT)

Contact | Kontakt oss

Lab manager | Laboratorieleder

Nélia Castro tlf: 228 51641* e-mail: nelia.castro@nhm.uio.no

Associate Professor | Førsteamanuensis (SEM, PXRD and SXRD)

Henrik Friis tlf: 228 51622* e-mail: henrik.friis@nhm.uio.no

Professor (micro-CT)

Øyvind Hammer tlf: 228 51658* e-mail: oyvind.hammer@nhm.uio.no

** If you call from the laboratory telephone, digit the last 5 digits of the number only.
Exp: Nélia Castro - 51641*

Contents

1.	Introduction	3
2.	Hygiene and safety in the laboratory	4
2.1.	General safety rules	4
2.2.	Emergency contacts	4
2.3.	Other rules	5
2.4.	Safety summary for Hitachi S-3600N	5
3.	Configuration	8
3.1.	Appearance of Hitachi S-3600N	8
3.2.	Control knobs and switches on column unit	9
3.3.	Control knobs and switches on display unit	10
3.4.	BSE detector	11
3.5.	EDS detector	12
3.6.	SEM software	12
3.7.	EDS software	16
4.	Booking the instrument	18
5.	Logbook	18
6.	Sample preparation	19
6.1.	Types of samples	19
6.1.1.	Coated samples	19
6.2.	Specimen holders	19
6.2.1.	Specimen height adjustment	21
6.2.2.	Set specimen size and height	21
7.	Operating instructions	23
7.1.	Startup	23
7.2.	Specimen exchange	24
7.3.	Image observation (SE and BSE)	27
7.4.	Chemical analysis (EDS)	33
7.4.1.	Point Analysis (automatic standardless)	33
7.4.2.	Multi point analysis	34
7.4.3.	Line fast scan	35
7.4.4.	Mapping (fast)	36
7.4.5.	HyperMap	37
7.4.6.	Creating a report	38
7.5.	Saving data	40
7.6.	Shutdown	41

Abbreviations

Abbreviation	Description
BSE	Backscattered electrons
EDS	Energy dispersive X-ray spectrometry
SE	Secondary electrons
SEM	Scanning electron microscope
WD	Working distance

1. Introduction

The Geo Lab at the Natural History Museum, University of Oslo, is equipped with a Hitachi S-3600N Scanning Electron Microscope (SEM), installed in 2004, which is suitable for high-resolution imaging of geological materials (minerals, microfossils) and semi-quantitative X-ray microanalysis of both conductive and non-conductive specimens.

The Hitachi S-3600N SEM can be operated in two modes:

- High-vacuum (HV) mode (< 1 Pa). Used for high-resolution imaging and semi-quantitative X-ray microanalysis of conductive samples or coated samples.
- Variable pressure (VP) mode (10–200 Pa). This allows observation of non-conductive samples in their natural state or in a wet condition, without the need for coating.

Instrument features:

- High-resolution secondary (SE) and backscattered (BSE) electron detectors.
- Bruker XFlash® 5030 energy dispersive X-ray detector (EDX), running on Quantax 400 (Esprit 1.9), for semi-quantitative elemental analysis and hyperspectral mapping, with <127 eV FWHM at MnK α energy resolution.
- Large chamber accommodating samples up to 150 mm in diameter and ~50 mm in height.
- A 5-axis motorized stage with eucentric tilt (0 – 52°, depending on sample size) and 360° rotation.

Sample preparation equipment:

Electrically insulating samples may be coated with a thin layer of conducting material, e.g. carbon, gold or some other metal alloy. The choice of material depends on the data to be acquired; carbon is most desirable for elemental analysis, while metal coatings are most effective for high-resolution imaging.

- Agar Auto Carbon Coater B7367A, designed to evaporate conducting carbon layers.
- Jeol Fine Coat Ion Sputter JFC-1100, with a PtPd metal alloy target.

Operation of this instrument is restricted to trained personnel familiar with the analyze system as well as the product documentation, general safety precautions, and laboratory rules. New users need to undergo a training period before they are allowed to work with this equipment independently. Contact the laboratory manager or deputy (e.g. your supervisor, your group leader, or your contact person at NHM) if you want to be trained to operate this instrument independently.

2. Hygiene and safety in the laboratory

2.1. General safety rules

All equipment in this laboratory uses **high-voltage**.

Laboratory users are **NOT allowed to**:

- Walk in the back of the equipment
- Touch equipment or computer cables
- Remove protection covers of the equipment
- Touch the interior of the equipment

Not attending these rules can lead to:

- Electrocution
- Burns

2.2. Emergency contacts

Fire: 110

Ambulance: 113

Police: 112

In case of unexpected incidents during the use of the equipment (for example if an alarm goes off), contact immediately one of the persons listed below.

Lab manager, Nélia Castro

tlf: 228 51641 e-mail: nelia.castro@nhm.uio.no

Associate Professor, Henrik Friis

tlf: 228 51622 e-mail: henrik.friis@nhm.uio.no

2.3. Other rules

- The **computers** in this lab are **not for personal use** (e.g. e-mail or internet search); their use is limited to data collection and data processing.
- Users are **only allowed to use the computer(s) of the instrument** they have booked.
- Users are **not allowed to connect any device (including USB devices) to the computers** in this lab.
- The use of **laptops** in the laboratory **shall be avoided**. Users are welcome to use the common area outside the laboratory to work in between analysis (computers available for UiO users).
- If the users need to connect a personal device to the electricity (e.g. laptop, mobile, etc.), **the electricity plugs available in the column in the middle of the room** must be used.
- **Food or drinks** are **not allowed** in the lab.

2.4. Safety summary for Hitachi S-3600N

Personal injuries can result from the inappropriate use of the instrument. The following warnings apply to the use of the Hitachi S-3600N SEM:

WARNING	Consequences	Safety measures
Beware of high voltage!	Electric shock can cause fatal or serious injury.	DO NOT remove the protective covers from the instrument. Do not disconnect the high-voltage cable.
Beware of high temperature!	Can cause burns.	DO NOT remove the protective covers from the instrument.
Beware of radiation!	Exposure to radiation.	SEM is serviced and safety checked regularly. The sample chamber is shield to stop the escape of any radiation and interlocked, meaning that cannot be opened when in use. The operator will not be exposed to X-rays or any radiation when the machine is in working under normal working mode. DO NOT deviate from normal working mode. NEVER try to override the interlock.

The following cautions should be taken when using the Hitachi S-3600N SEM:

Action	Consequences	Safety measures
Prolonged use	Eye strain, tiredness, and musculoskeletal strain or injury.	Take regular breaks (about 10-15 minutes break per hour during operation). Adjust height of the seat, so that the back and neck and upright.
Opening and closing the specimen chamber to exchange sample	Personal injuries caused by jamming your hand.	Handle the specimen chamber with care.

Damage to the instrument can result if the following prevention measures are not contemplated when using the Hitachi S-3600N SEM:

Instrument part	Danger and consequences	Prevention measures
BSE detector	The BSE detector is vulnerable to shock. If the surface of its semiconductor element is flawed due to any shock, degradation in performance could result.	Always leave the BSE detector in the OUT position when not using the instrument. Always use the camera unit to control the movements of the BSE detector and/or specimen inside the chamber. Before inserting or withdrawing the BSE detector, be sure to move the specimen to the specimen exchange position (see Sections 3.4 and 7.2 for details). When loading a new sample make sure to check the specimen height using the height gauge (Section 6.2.1). Before proceeding to observation, specify a specimen size value and a specimen height offset value properly on the Stage Control dialog box (Section 6.2.2). NOTE: Be aware that the minimum WD for BSE detector is 7 mm. There is no sound alarm if you exceed the minimum WD.
CL detector	The CL detector and the BSE detector cannot be used at the same time. The CL detector will shock with the BSE detector if inserted before withdrawing the BSE detector.	Always withdraw the BSE detector before inserting the CL detector (and vice-versa). Use the same prevention measures mentioned to the BSE detector when it comes to monitor the movement of the CL detector and specimen inside the chamber, loading new

The CL detector is sensible to light (e.g. from the camera unit inside the chamber or room light when the chamber is vented).

samples, and proceeding to observation.

Make sure the CL detector is OFF before turning on the camera unit and before opening the chamber.

3. Configuration

3.1. Appearance of Hitachi S-3600N

Figure 1 shows the appearance of the Hitachi S-3600N. The instrument has two main units: the column unit and the display unit.

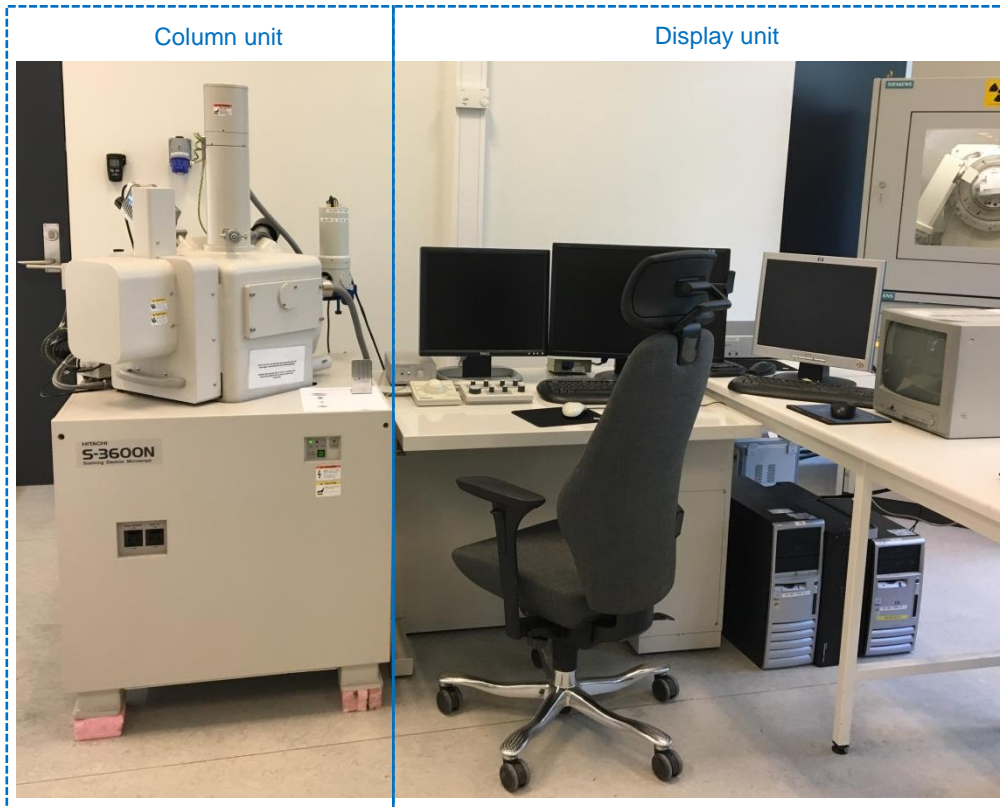


Fig. 1: Appearance of the Hitachi S- 3600N.

3.2. Control knobs and switches on column unit

Figure 2 shows the appearance of the column unit.

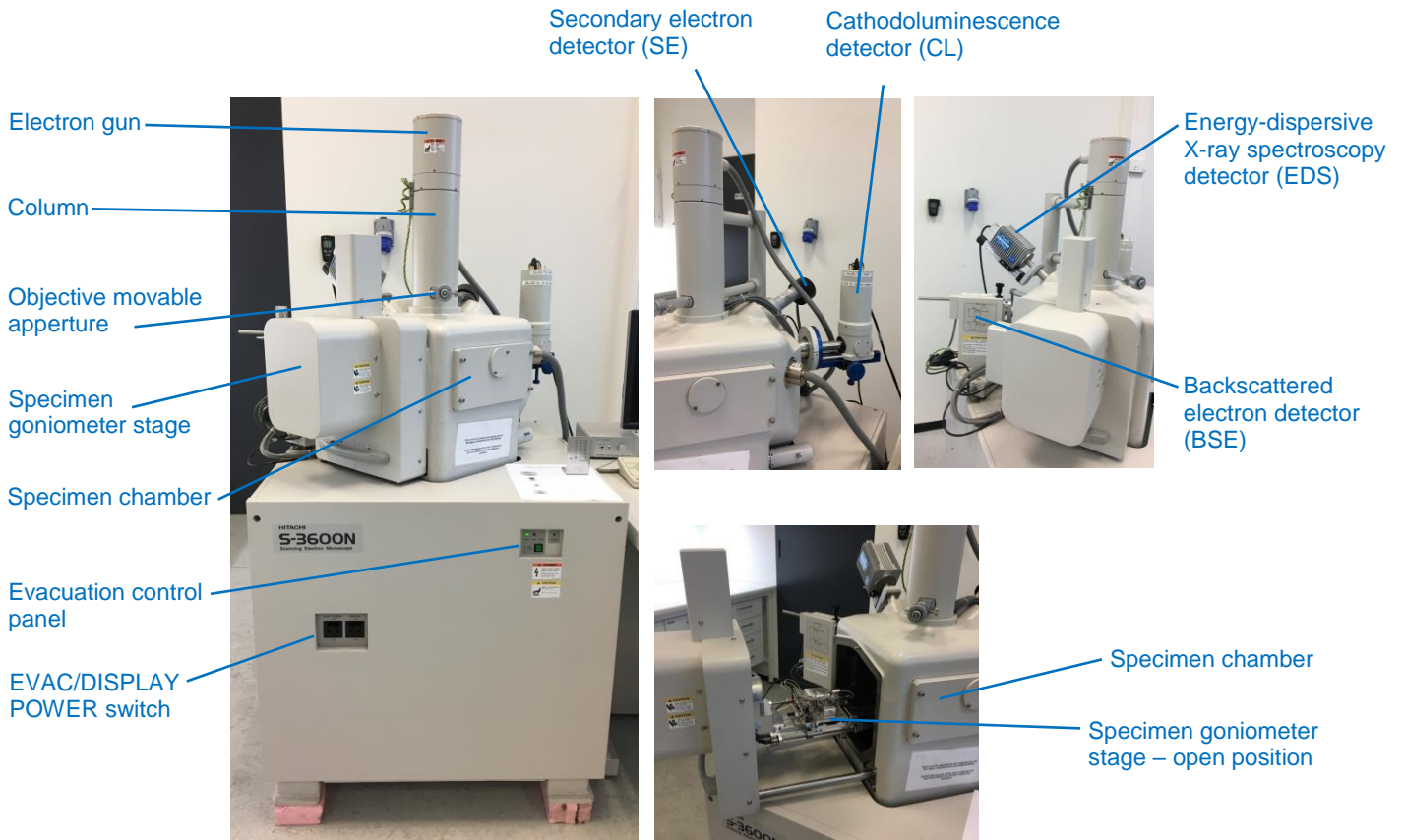


Fig. 2: Appearance of the column unit.

3.3. Control knobs and switches on display unit

Figure 3 shows the appearance of the display unit.

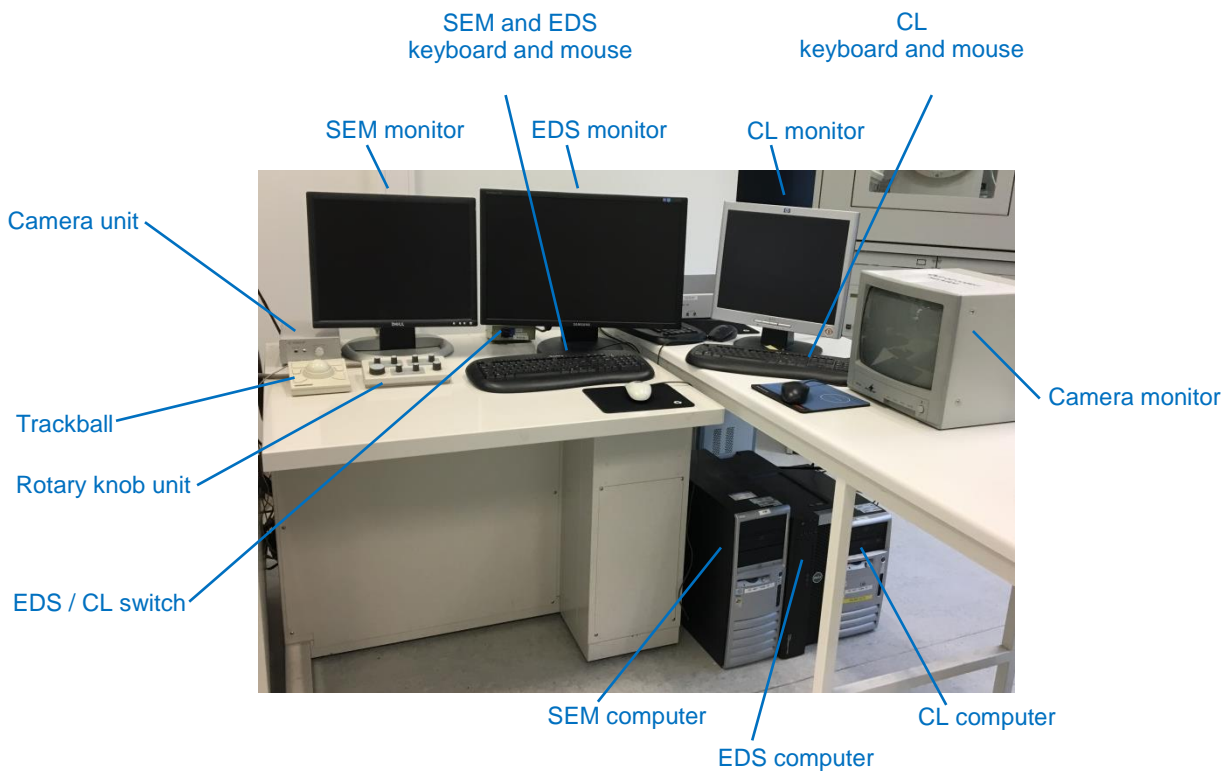


Fig. 3: Appearance of the display unit.

The **camera unit** and **monitor** are used to visualize the inside of the specimen chamber and monitor the movement of the specimen goniometer stage.

The **EDS / CL switch** is used to switch between the EDS and the CL detectors. **CAUTION: The CL detector and the BSE detector cannot be used at the same time. Check the prevention measures that apply to the CL detector in Section 2.3.**

The **trackball** is used to move the specimen stage on the X and Y axes.

The **rotary knob unit** is used to adjust the image magnification, contrast, brightness, focus and astigmatism. Figure 4 shows the appearance of the rotary knob unit.

- Magnification knob is used to change magnification. Buzzer sounds upon reaching lowest or highest magnification.
- Contrast and brightness knobs are used to adjust contrast and brightness of image. Turning contrast clockwise increases the contrast. Turning the brightness clockwise make the image brighter. If the busser sounds, it indicates the limit of the adjustable range.
- Focus knob is used to adjust the focus. The variation degree of this knob is linked with magnification, and the lower the magnification, the larger the variation in focus. First adjust

the focus roughly at low magnification, then increase the magnification and finely adjust the focus.

- Stigmator/alignment XY knobs are normally used to correct astigmatism. IMPORTANT: Do not use these knobs without the presence of the laboratory manager.
- Image shift/multi function X/K₁ Y/K₂ knobs are assigned the functions given in the rotary knob K1, K2 display area of the status bar. IMPORTANT: Do not use these knobs without the presence of the laboratory manager.

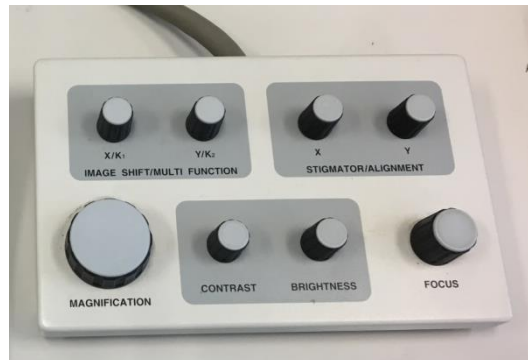


Fig. 4: Appearance of the rotary knobs unit.

3.4. BSE detector

The BSE detector is situated in the left side of the column unit. **CAUTION: The BSE detector is vulnerable to shock. Check the prevention measures that apply to the BSE detector in Section 2.3.**

Figure 5 shows the BSE detector in the position OUT (left) and COMPO/TOPO mode (right) respectively. For observation of composition and topographic images insert the BSE detector fully, COMPO/TOPO mode. For observation of 3D images insert the BSE detector to the midpoint position (not pictured in the image). In order to move the BSE detector pull up the lock knob.



Fig. 5: Detail of the column where BSE and EDS detectors are visible. BSE detector can be seen in the position OUT (left) and COMPO/TOPO (right). EDS detector it is in INN position in both images.

3.5. EDS detector

The EDS detector is situated in the left back side of the column unit. Figure 5 shows the EDS detector in the position INN. In order to move the EDS detector the screw in the back of the detector has to be turned. The EDS detector can be left at the position INN at all times since there is no incompatibility between the EDS detector and other detectors.

3.6. SEM software

The software Hitachi S-3600, installed in the SEM computer, is used to control the operation of the SEM, including the BSE and SE detectors. Described next is the operation window (Fig. 6) used to control the Hitachi S-3600N.

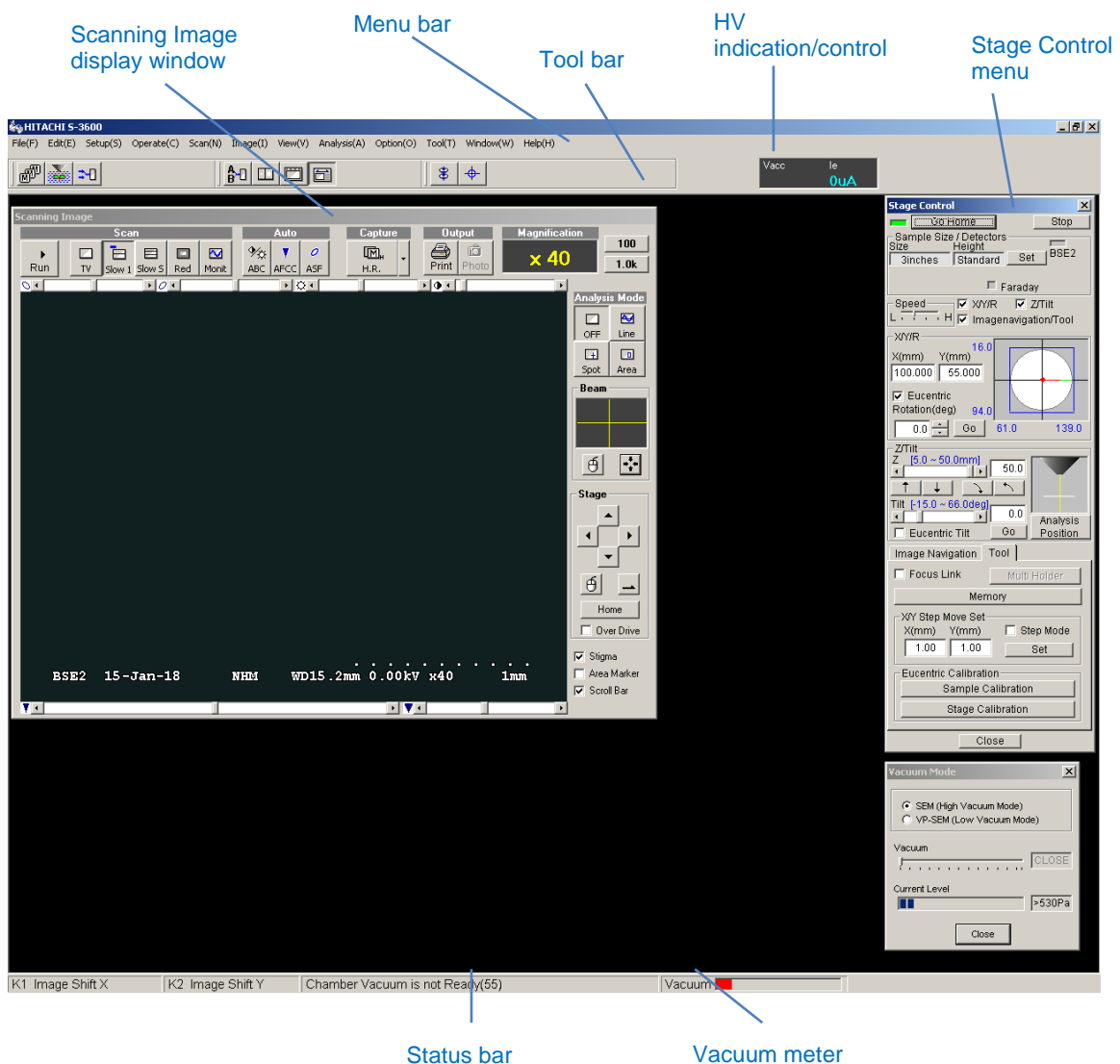


Fig. 6: Overview of the operation window of the software used to control the Hitachi S-3600N SEM.

Scanning Image display window

Displays the image under observation (Fig. 7). Also contains dedicated buttons for changing the scanning speed, adjust the image brightness and contrast, etc., and functions for adjusting the image via mouse operation.

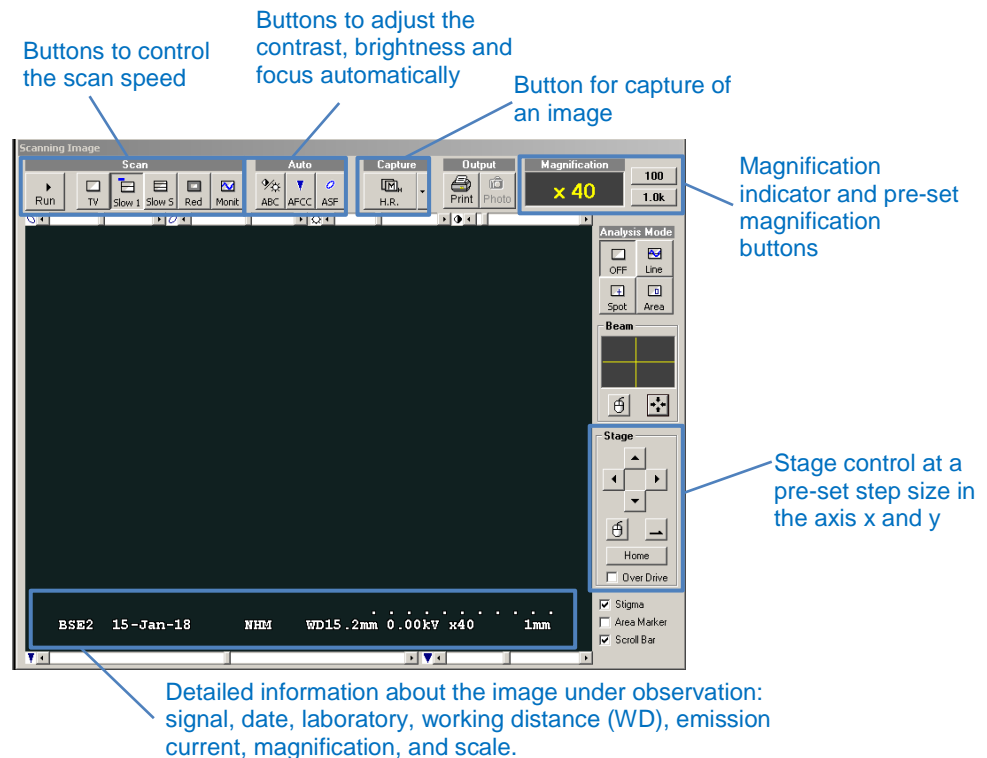


Fig. 7: Overview of the Scanning Image display window.

Menu bar

Each item in the Menu bar opens menus for a group of related operations. In Figure 8 are described the most commonly used menus and operations. Note that most of these can also be opened using buttons in the Tool bar or in the Scanning Image window.

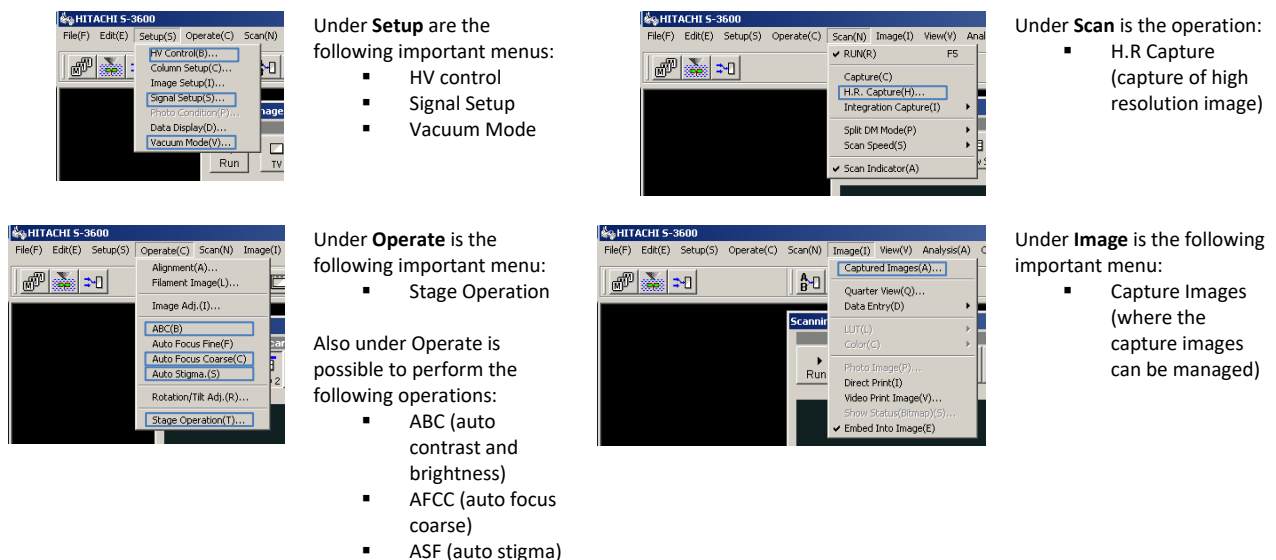


Fig. 8: Overview of the most important menus and operations found under each item in the Menu bar.

Tool bar and HV indication/control

The buttons in the Tool bar are configured to enable the fast execution of operations and the fast opening of dialogs for the items that are used most frequently (Fig. 9). At the right end of the tool bar is a HV ON/OFF button and an indication/control area showing the present accelerating voltage and emission current values.

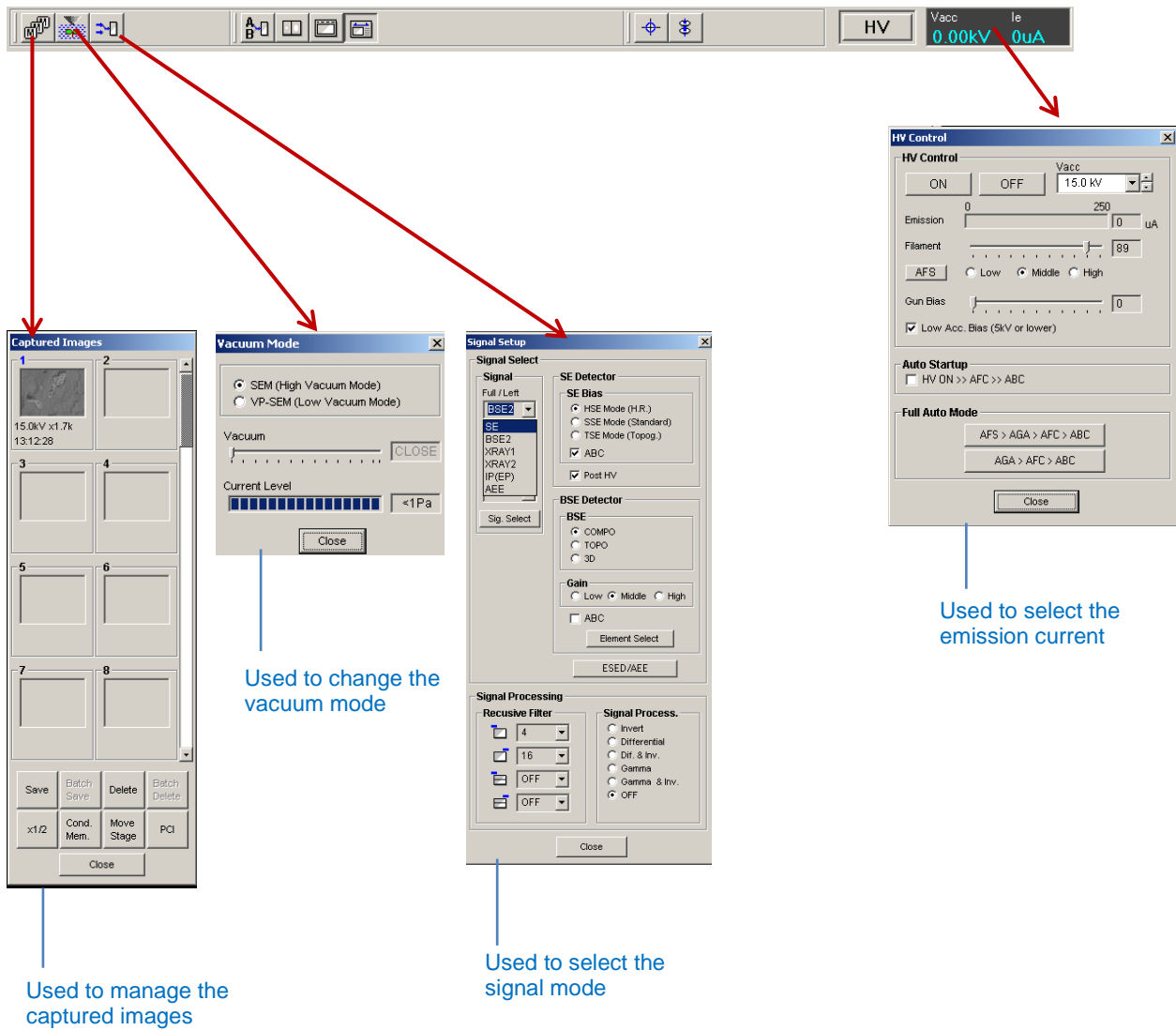


Fig. 9: Overview of the Tool bar and its most used buttons.

Stage Control menu

Controls the specimen stage inside the chamber (Fig. 10). Contains dedicated buttons to set the specimen size and height, move the stage to the exchange (home) position and to the analysis position, among other functions.

The Stage Control menu can be opened in Operate in the Menu bar.

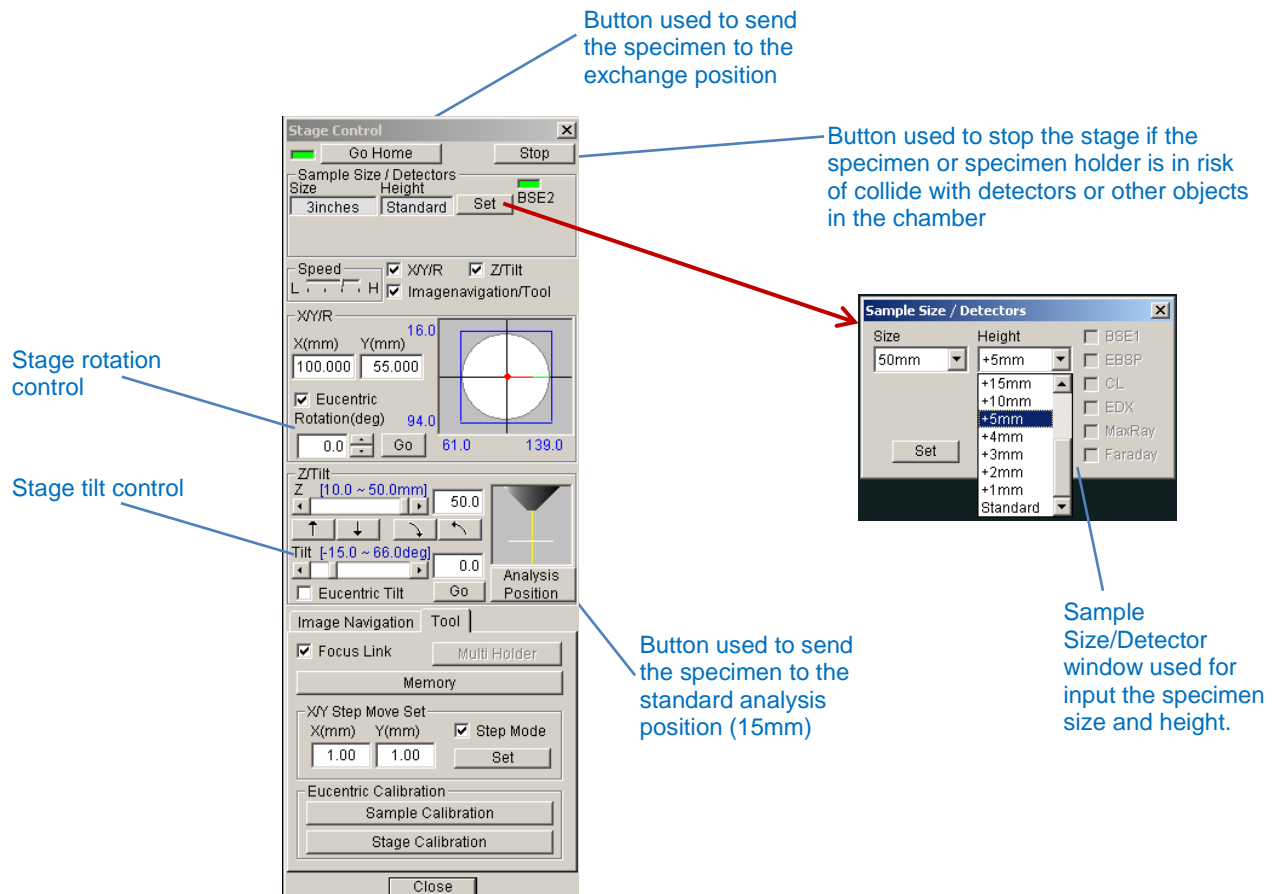


Fig. 10: Overview of the Stage Control menu. This menu can be opened in Operate in the Menu bar.

Vacuum meter

Shows the vacuum status (Fig. 11).

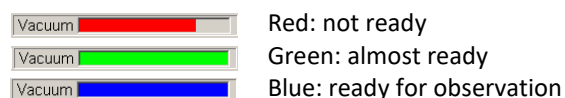


Fig. 11: Vacuum meter and its different status.

3.7. EDS software

The software Esprit 1.9 Quantax 400, installed in the EDS computer, is used to control the operation of the EDS detector. The software icon and startup screen are shown in Figure 12.



Fig. 12: Icon and startup screen of the software Esprit 1.9 Quantax 400 used to EDS operation.

On the top section of the main program window (Fig. 13) one has access to different workspaces and Sections.

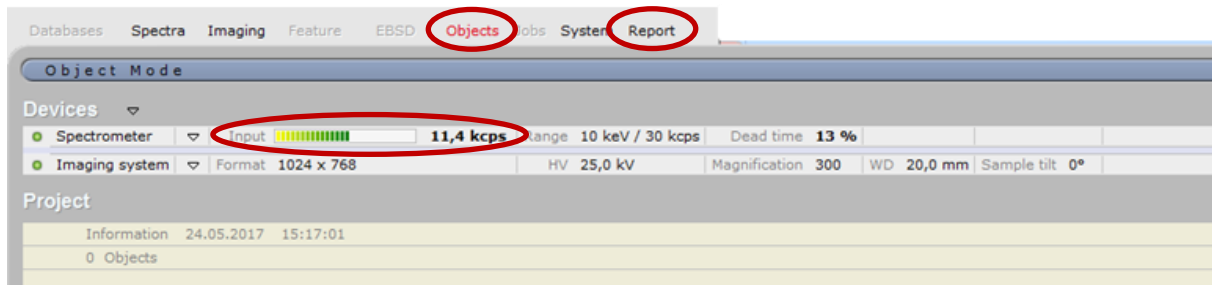


Fig. 13: Top section of the main program window, software Esprit 1.9 Quantax 400.

The workspaces most commonly used in our lab are Objects and Report:

Objects consists of a group of workspaces that combine electron beam scanning with X-ray spectrometry:

- Point allows analysis of one point with electron beam control.
- MultiPoint allows analysis of one or more points with electron beam control. It is also used to analyze arbitrarily shaped objects.
- Line scan performs qualitative or quantitative line scans.
- Mapping allows you to generate qualitative or quantitative element maps.
- HyperMap is a position tagged spectrometry tool (spectral imaging).

Report starts the Report editor. It can be used in parallel to all workspaces that provide measurement data acquisition and processing.

Instructions on how to use the Objects and Report workspaces can be found in Section 7.4. Additionally, the main program window includes the **Device** and the **Project** screen sections.

Under the section Device one can check important information such as the signal input (kcps). If the signal input is too low (signal bar is less than 1/3) try to choose another point or area on your sample for analysis or increase the accelerating voltage (HV) in the SEM software (see Section 7.3 for details).

4. Booking the instrument

You are required to book time for your analysis using the **instrument calendar on Outlook or Webmail**. The name of this instrument calendar is: **Økern: Geologisk museum, SEM**. Instructions are available at the Geo Lab webpage:

<https://www.nhm.uio.no/english/research/infrastructure/geo-lab/sem-edx/>

If you don't have access to the instrument calendar (e.g. if you are an external user), please contact the laboratory manager or deputy and ask if they can book time for your analysis.

5. Logbook

You are required to log the time you use the instrument using the excel file named **Logbook** available at the **EDS computer** (check the Desktop). At the beginning of your session please fill in the relevant information and the start time. At the end of your session, please remember to fill in the finish time and save the document before you leave.

6. Sample preparation

6.1. Types of samples

This Hitachi S3600N SEM is used primarily for **geological materials**, such as rocks, minerals and fossils. Hand-specimens, polished sections and thin-sections are commonly investigated in this instrument. The specimen chamber in this instrument is relatively large, allowing specimens with fairly large dimensions (up to ca. 150 x 150 x ~50 mm) to be used.

Non-conductive specimens must be **coated** for imaging in high vacuum conditions (see Section 6.1.1), but can be imaged **without coating** using low/environmental vacuum conditions.

CAUTION: Samples that may release oil vapors or magnetic dust must not be placed in the chamber without prior permission. When analyzing hydrated samples, WD shall be kept above 10mm.

Biological materials can be accepted for analysis under specific circumstances.

6.1.1. Coated samples

The laboratory is equipped with two coating equipment:

- Agar Auto carbon coater
- Jeol Fine Coat Ion Sputter JFC-1100 for platinum-palladium (PtPs) metal coating

Instructions for use of the above mentioned equipment can be found in the sample preparation laboratory (room U 560) next to the respective equipment. Additionally, user instructions are also available at the Geo Lab webpage:

<https://www.nhm.uio.no/english/research/infrastructure/geo-lab/other-equipment/>

Only authorized personnel can use the coaters. Contact the laboratory manager or deputy the first time you want to use these.

6.2. Specimen holders

There are a number of different specimen holders available for the Hitachi S-3600N SEM. The appearance and size of the specimen holders most commonly used in the Geo Lab are listed in Table 1. Figure 14 shows schematically how the different parts of the sample holder are assembled.

Choose a proper specimen holder to the size and height of the specimen to be observed.

Carbon tape is the most common method used to attach the specimen to the specimen holder. Contact the laboratory manager or deputy if you plan to use a different method to attach your specimen to the sample holder.

Table 1: Appearance and size of the specimen holders most commonly used in the Geo Lab.

Specimen holder	Size
	6 inches
	50 mm
	15 mm
	3 inches

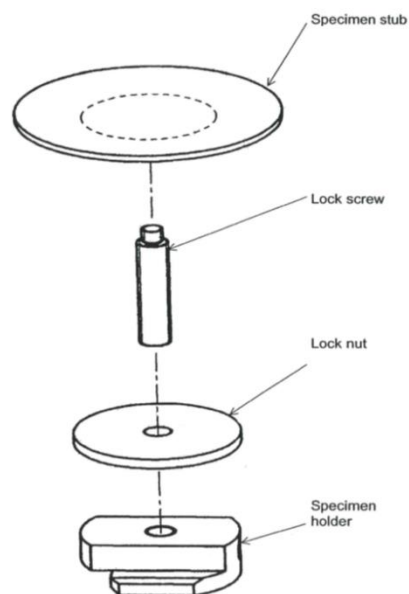


Fig. 14: Specimen holder parts and assembly.

6.2.1. Specimen height adjustment

The **specimen height** must be **determined using the height gauge** as seen in Figure 15. The specimen holder and height gauge must be on a level plane to a correct measurement.

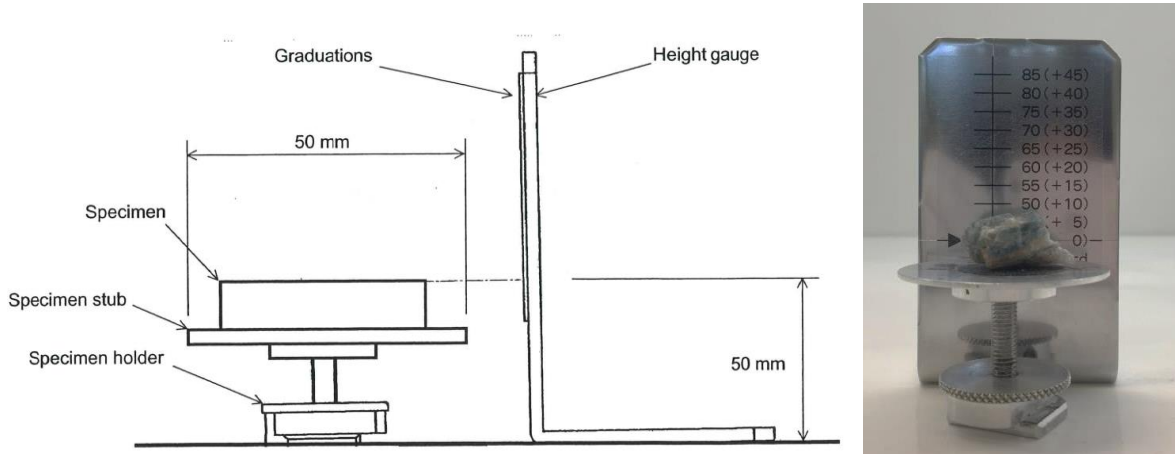


Fig. 15: Specimen height determination with the height gauge.

Height 40 mm is considered **standard**. Specimens shall be mounted at standard height when possible. Loosen the lock nut of the specimen holder to adjust the specimen height if necessary.

If the specimen **height** is inevitably **higher than 40 mm (standard)** there will be an **offset value** corresponding to the excess beyond the height level of 40 mm. This value can be **read in the height gauge (the value in bracket, e.g.: 0, +5, +10, ...)**.

For example if the specimen height is 50 mm, the offset value will be +10 (Fig. 15).

6.2.2. Set specimen size and height

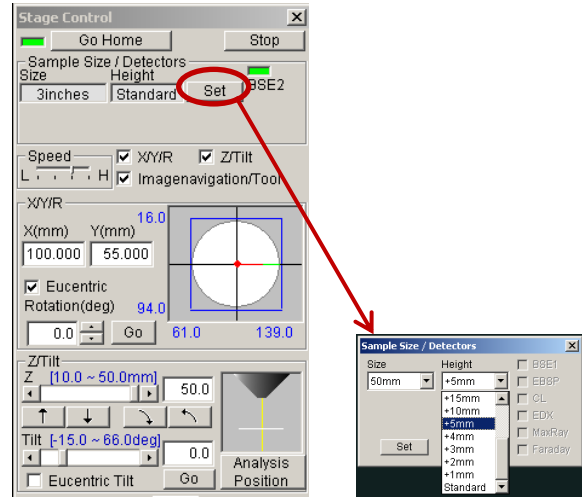
Specimen size and height must be input in the software to help moving the specimen inside the chamber in a safe way. **If this setting is improper, the specimen may collide with the objective lens or BSE detector and cause damage to it.**

Usually the specimen is smaller than the specimen holder and therefore, is the size of the specimen holder that is entered as specimen size. If the specimen is larger than the sample holder, enter the size of the specimen.

Step

- 1 Set the specimen size and height by clicking in the button Set in the Stage Control window. The Stage Control menu can be found in Operate (Section 3.5).

Example/hints



Details on when to Set the specimen size and height can be found in Section 7.2.

7. Operating instructions

The procedures for instrument startup, specimen exchange, image observation (SE and BSE), chemical analysis (EDS), and shutdown are explained in this section.

7.1. Startup

Step

1 When arriving at the laboratory make sure that the **lamp HIGH (for high vacuum)** in the evacuation control panel is **ON** and the **BSE detector** is in **OUT** position. All **computers** shall also be **ON** with the respective screens off or in sleep mode.

2 Fill in the **logbook**

3 Now you are ready to insert your first sample in the instrument.

Example/hints



Contact the laboratory manager or deputy immediately if at your arrival to the laboratory the SEM conditions differ from the description above.

Excel file in the EDS computer.

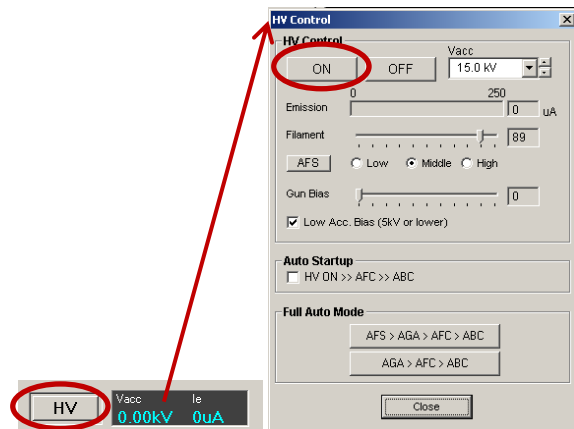
Follow the instruction for specimen exchange (Section 7.2) from point 2 on.

7.2. Specimen exchange

Step

1 When the **accelerating voltage** is ON, click **HV** on the tool bar or OFF in the HV Control dialog box to turn OFF the voltage. The HV Control dialog can be open by clicking in the HV indication/control (blue letters) in the Tool bar.

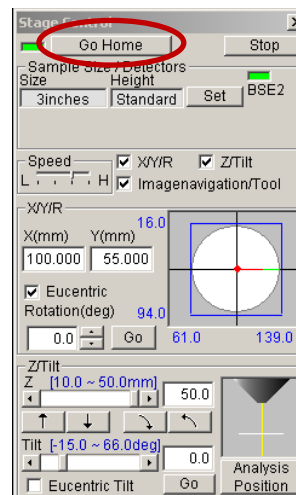
Example/hints



Clicking the HV button will turn the accelerating voltage ON and OFF.

2 Move **the stage to the specimen exchange position** by pressing the **Go Home** button in the Stage Control dialog box. The Stage Control dialog box can be found in Operate.

CAUTION: If the stage is pulled out at other position, the specimen and instrument may be damaged.



3 Press **EVAC/AIR** switch on the evacuation control panel to set it at **AIR**. The LOW lamp comes on and air is introduced into the specimen chamber.



4 Wait until the chamber door opens by itself, pull out the specimen stage and **remove the specimen holder** (if there is any).



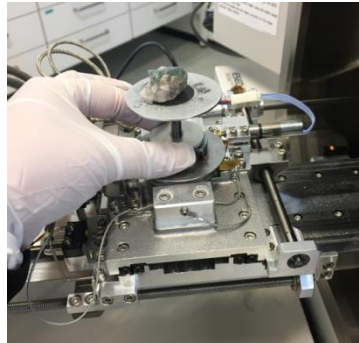
Remember to use gloves when handling the specimen holder to avoid contamination of the specimen chamber.

5 Mount your specimen in an appropriate specimen holder and **determine the specimen size and height (the value in brackets)**.



See Section 6 for details about sample preparation.

6 Set the specimen holder containing the specimen to be observed **onto the stage**.



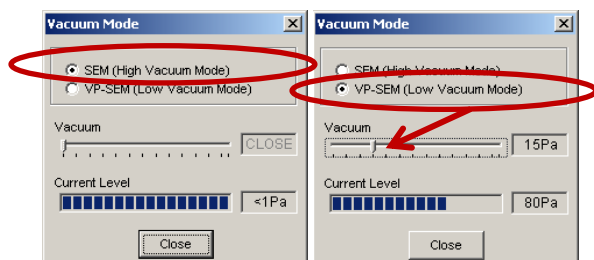
7 Insert the stage into the specimen chamber, close the chamber door and set the **EVAC/AIR switch to EVAC** while holding the chamber door closed for a few seconds.



While waiting to the chamber to evacuate, which can take about 6 minutes (5min 45s), continue to points 8) to 12) in this section.

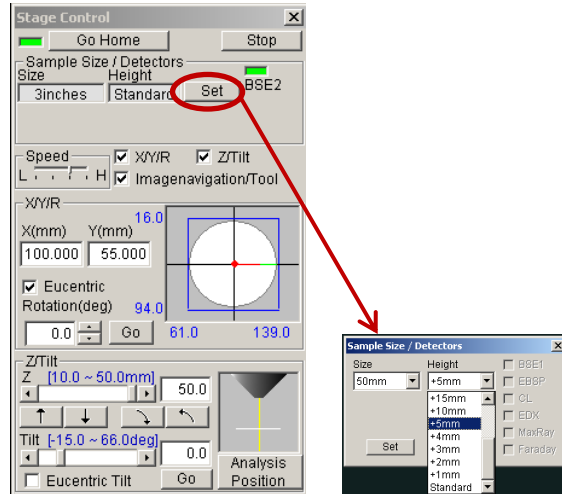
8 Use the **Vacuum Mode** menu to select the vacuum mode you want to work with: SEM (High Vacuum Mode) or VP-SEM (Low Vacuum Mode). The Vacuum Mode menu can be found in Setup or in the Tool bar.

If you select VP-SEM (Low Vacuum Mode) set a vacuum level using the slider at Vacuum in the Vacuum Mode window. The current vacuum level indication will advance toward the set level. It takes



about 2 minutes for the specimen chamber to reach the set vacuum level.

9 Set the specimen size and height by clicking in the button Set in the Stage Control window. The Stage Control menu can be found in Operate.

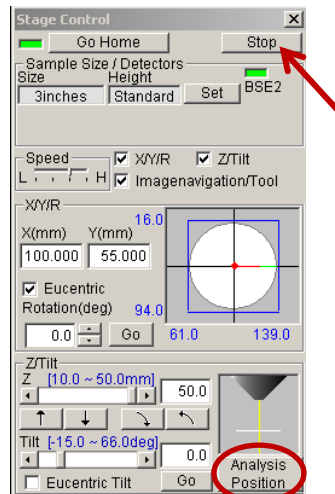


See Section 6.2 for details about sample size and height.

10 Turn on the camera unit and monitor. Move the stage to Analysis Position using the Stage Control window.

After clicking in the button Analysis Position, position the mouse on top of the **Stop** button immediately.

Monitor the movement of the Specimen holder inside the chamber. Click Stop immediately if the Specimen Holder is in risk to collide with the BSE detector or any other object inside the chamber.



The Stage Control window can be found in Operate.

11 If you are planning to use the **BSE detector** you can insert it in the specimen chamber now. Use the camera unit to monitor its movement inside the specimen chamber.

In order to move the BSE detector pull up the lock knob. For observation of composition and topographic images insert the BSE detector fully, COMPO/TOPO mode. For observation of 3D images insert the BSE detector to the midpoint position.

CAUTION: The BSE detector is vulnerable to shock. Check the prevention measures that apply to the BSE detector in Section 2.3.



Consult section 3 for details.

12 When the **vacuum meter** on the status bar turns **blue** the accelerating voltage can be applied, meaning you are now ready for image observation.

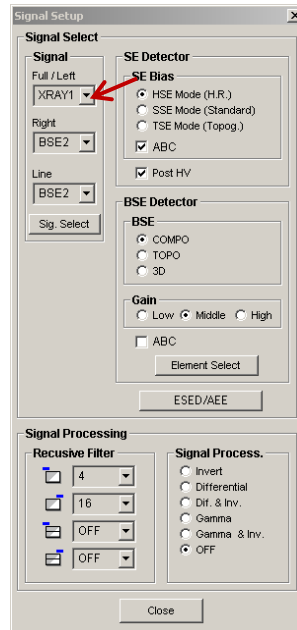


7.3. Image observation (SE and BSE)

Step

1 Select the **input signal** (e.g. SE, BSE, ESED) using the Signal Setup dialog box.

Example/hints



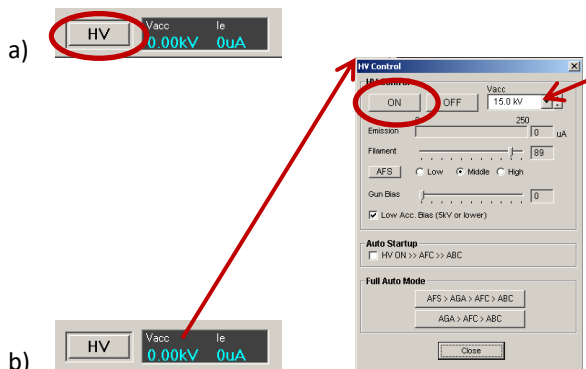
The Signal Setup dialog box can be found in Setup or in the Tool bar.

Both SE and BSE can be observed in high vacuum mode.

In low vacuum mode, BSE signal can be observed the same way as in high vacuum mode, and SE signal can be collected with ESED (environmental secondary electrons detector).

2 Apply a suitable **accelerating voltage** according to specimen and image quality.

- If you want to use the **same accelerating voltage** as in the last observation, click **HV** in the Tool bar.
- If you want to **change the accelerating voltage**, open the **HV Control window** by clicking in the HV indication/control in the Tool bar. Select an accelerating voltage from the V_{acc} dropdown list box.



The accelerating voltage is set at the same value as it was left in the last observation, even after the accelerating voltage is turned off.

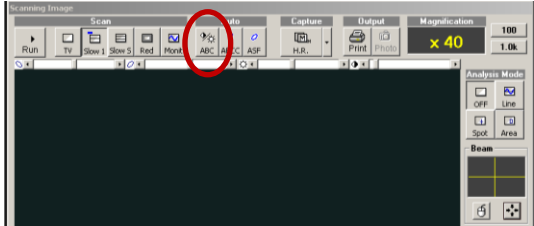
When HV is on, the HV frame turns red and the set accelerating voltage appears on the data display. Check

on the high voltage indication/control area that the emission current is flowing.


3 Adjust the image **brightness and contrast**.

This can be done:

- Automatically** by pressing **ABC** in the Scanning Image window, or selecting ABC in the Operate menu.
- Manually** using the Brightness and Contrast **rotary knobs** in the Rotary Knobs unit.

- 

During the auto adjustment, the image is “frozen” and a message dialog indicating “A.B.C. Mode In Progress” appears on the screen. A buzzer sound indicates that the auto adjustment is completed.

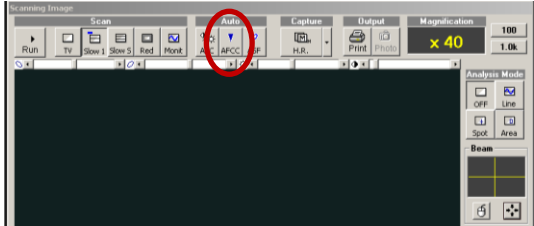
- 

See Section 3 for details.


4 Adjust **the focus**. Focus adjustment is carried out from a status in which the image is barely visible to a status where field selection is possible.

This can be done:

- Automatically** by pressing **AFCC** in the Scanning Image window, or selecting Auto Focus Coarse in the Operate menu.
- Manually** using the Focus **rotary knob** in the Rotary Knobs unit

- 

During the auto adjustment, the image is “frozen” and a message dialog indicating “A.F.C. Mode In Progress” appears on the screen. A buzzer sound indicates that the auto adjustment is completed.

- 

See section 3 for details.
Usually leads to better focus than the automatic option.

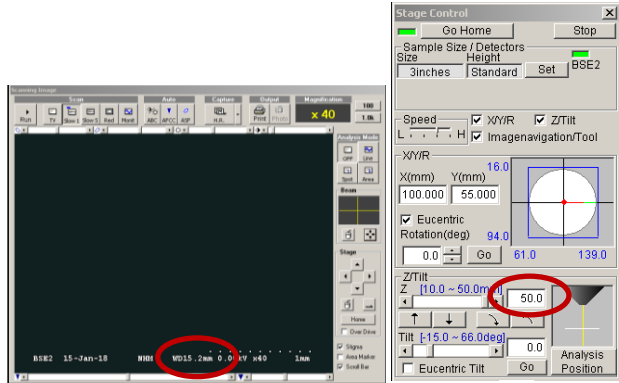
Important: It is good practice to focus at higher magnification than the one needed for your observations.

Note: Comparing the focus WD and the specimen WD one can check for discrepancies:

- If the focus WD value is higher or equal to the specimen WD, the specimen height was calculated correctly and there is no risk of

accidentally running the specimen into a detector or other objects inside the chamber.

- If the focus WD is lower than the specimen WD, may have been an error in the measurement of the specimen height and there is a risk of collision with detectors or other objects inside the chamber. Move the specimen to the exchange (home) position, remove it, and measure its height again (see section 7.2 for more details on specimen exchange and measuring the specimen height).

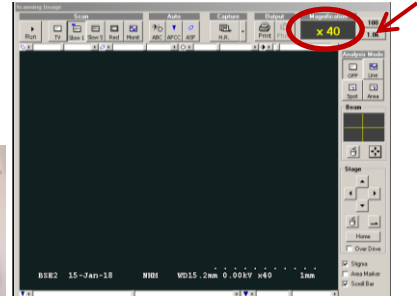


When the sample is in focus, the **focus WD** (working distance) is indicated in the **Scanning Image window**. The focus WD corresponds to the distance from the objective at which your sample is in focus.

In the **Stage Control window** is indicated the **specimen WD**, which is the distance at which the software calculates that your specimen is from the objective, based on the specimen height that you measured and input in the software (Section 6.2). If there was an error in the measurement of the specimen height and/or input of the data in the software, the real distance at which the specimen is from the objective is going to differ from what is indicated in the software increasing the risk of collision.

5 Set a suitable **magnification** using the Magnification **rotary knob** in the Rotary Knobs unit. The magnification is indicated in the Scanning Image window.

For setting to a **pre-set magnification**, press the pre-set magnification buttons on the Scanning Image window.



The **minimum magnification** is restricted by the WD accelerating voltage and scan speed.

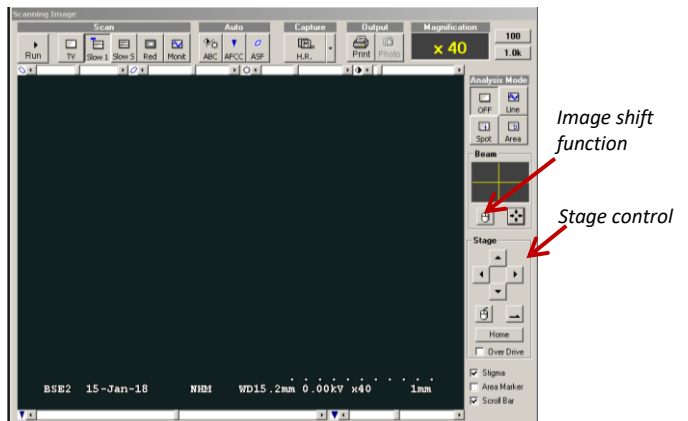
The **maximum magnification** is restricted by WD and accelerating voltage.

The magnification is set at the same value as it was left in the last observation, even after the HV is turned off.

6 Select a suitable **visual field** for your analysis.

First, at a setting near the minimum magnification, search the specimen using the **Trackball** or **Stage Control menu** (Section 3.5), then gradually increase the magnification and select a visual field.

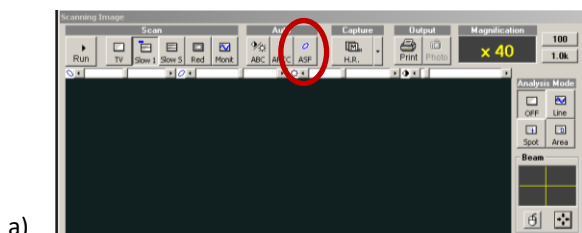
At a **high magnification** (several thousand or higher) it is convenient to use the **Image Shift function** (electrical field shift). This function is found in the Scanning Image window (Section 3.5).



7 Astigmatism may need to be corrected for observing images at magnifications higher than 2000 times.

This can be done:

- a) **Automatically** by pressing **ASF** in the Scanning Image window, or selecting Auto Stigma in the Operate menu.
- b) **Manually** using the Stigmator/Alignment **rotary knobs** in the Rotary Knobs unit.



- a) During the auto adjustment, the image is “frozen” and a message dialog indicating “A.S.F. Mode In Progress” appears on the screen. A buzzer sound indicates that the auto adjustment is completed.



- b) Usually leads to better astigmatism correction than the automatic option. See Section 3.3 for details.

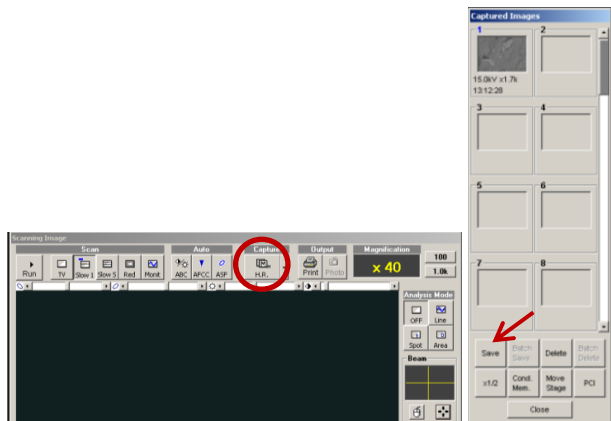
Remember that to achieve a good astigmatism correction you should start and finish by adjusting the focus. Using the reduced field option (Red button in the Scanning Image window) can be useful during astigmatism correction.

Note: Astigmatism correction can be challenging. Contact the laboratory manager if you need help with this step.

8 Record a photo by pressing the button **H.R.** in the Scanning Image window.

The **photography settings** can be adjusted prior to the capture in the menu Photo Condition that opens by pressing the arrow next to the HR button. The conditions are stored and do not need to be input every time.

Select a photo and click **Save** in the Captured Images dialog to save the selected photo. All photos will be saved in the SEM computer. All photos shall be saved in the folder D:\User data under a folder with your name (e.g. D:\User data\Waldemar Brøgger).



H.R. Capture can also be found under Scan in the Menu bar (Section 3).

When clicking H.R., the image disappears and then is newly displayed from top to bottom. When the scan reaches the bottom line, a buzzer sounds, and the Photo Condition dialog opens (if not opened yet). The Captured Images dialog can also be opened by clicking in the first button of the Tool bar (Section 3) or under Image(s) in the Menu bar (Section 3).

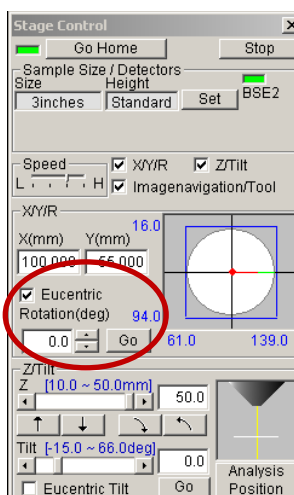
In the course of photographing, all functions except photo recording cancellation will not work.

No more than 16 images can be active in the Captured Images dialog. After saving the images of interest in the right folder delete the images from the Captured Images dialog using the button Delete or selecting several images at the same time and clicking Batch Delete.

9 Rotating the stage may be necessary for some observations. This is done in the **Stage Control** menu.

The current angle of rotation is indicated in black. Input a target value and press Enter, the stage will be driven to the specific angle. Using the two arrow buttons, rotation can be adjusted manually.

The **eucentric rotation** function can be turned on (checked) to assure that the visual field will not escape when the stage is rotated. When the eucentric rotation function is selected the rotation menu is changed. Key in a target value or selected using the arrow buttons, then press the button Go.

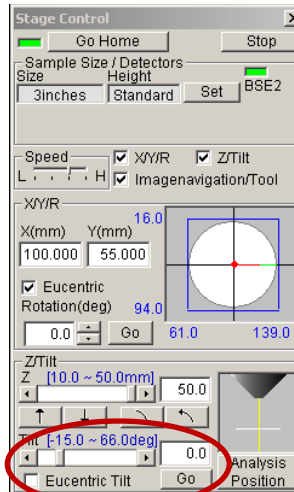


It is recommended to decrease the magnification to approx. 250x before attempting eucentric rotation.

10 Tilting the stage can also help some observations.

The current tilting angle is indicated in black. Input a target value and press Enter or adjust it by adjusting the slider and press the Go button. Then, the stage will be moved to the specific position. Using the two arrow buttons, tilting can be adjusted manually about 0.5° at the time.

The **eucentric tilt function** can be turned on (checked) to assure that the visual field will not escape when the stage is tilted.



Note: Tilt range is sample size dependent. It is recommended to decrease the magnification to approx. 250x before attempting eucentric tilt.

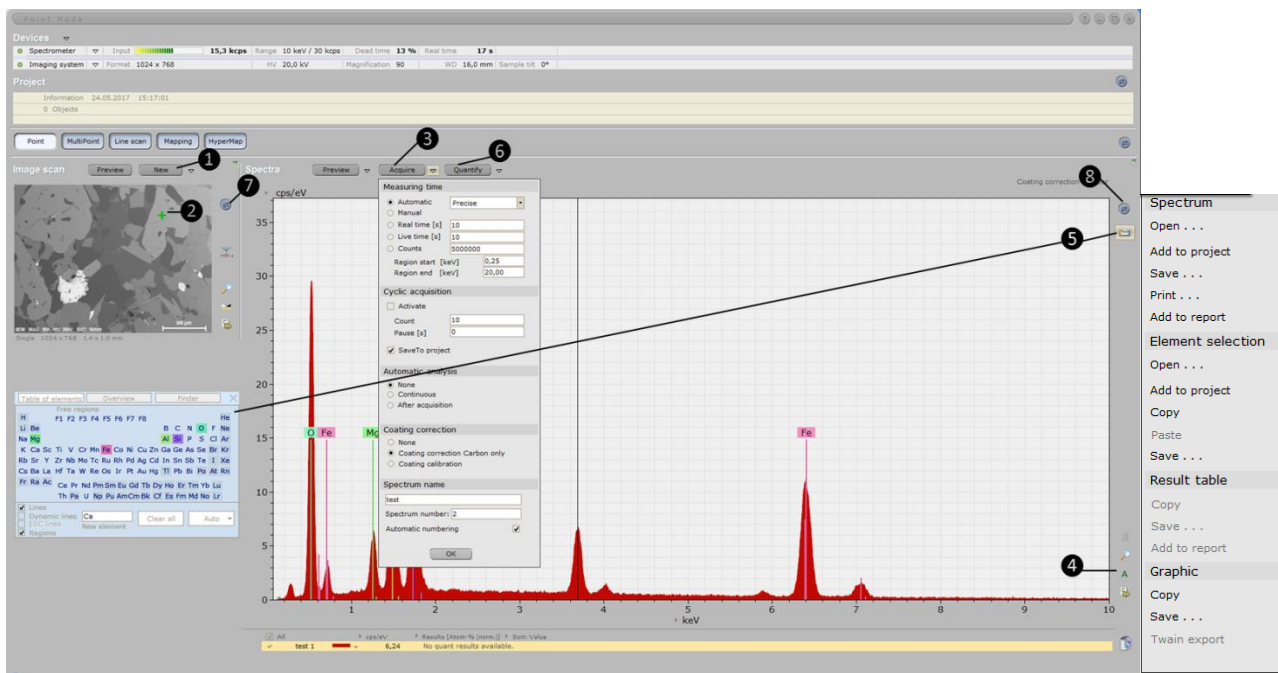
The **eucentric tilting function may require sample calibration**. Please contact the laboratory manager if the visual field is systematically lost when the stage is tilted.

7.4. Chemical analysis (EDS)

In this section we describe the most common operations within Objects workspaces. For advanced operations such as Fully Quantitative Line Scan, Fully Quantitative Mapping and Phase Analysis talk with the laboratory manager or consult the document Quantax Quick Reference Guide available at the Geo lab webpage <https://www.nhm.uio.no/english/research/infrastructure/geo-lab/other-equipment/>.

7.4.1. Point Analysis (automatic standardless)

Step	Examples/hints
1 Click New to acquire an image.	
2 Adjust measurement point.	
3 Click Acquire .	
4 When acquisition is finished click A to adjust the scale of the spectrum (if necessary).	
5 Open the Interactive identification tool (Periodic Table of the Elements). Click Auto to perform an automatic search of the elements present. You can also select the elements manually.	
6 Click Quantify and follow the instructions in the new window to perform a quantification of the elements present in your sample.	
7 Click the button Export next to the image to save the image .	
8 Click the button Export next to the spectrum to save the spectrum, the table data, and the spectrum image (graphic) .	Save the spectrum in .spx, the table data in .xls. Note that if you have analyzed several points you can save an excel table with all results by selecting all spectrums lines (need to be all yellow) and them pressing the Export button and Save table.



7.4.2. Multi point analysis

Step

- 1 Click **New** to acquire an image.
- 2 Position any desired number of points, draw any number of rectangles, ellipses or polygons.
- 3 Use **Select all** to highlight all objects.
- 4 Check if automatic analysis after acquisition is selected by clicking in the arrow next to the button **Acquire**.
- 5 Click **Acquire** to start spectrum analysis.
- 6 When acquisition is finished click **A** to adjust the scale of the spectrum (if necessary).
- 7 Open the **Interactive identification tool** (Periodic Table of the Elements). Click **Auto** to perform an automatic search of the elements present. You can also select the elements manually.
- 8 Click **Quantify** and follow the instructions in the new window to perform a quantification of the elements present in your sample.
- 9 Click the button **Export** next to the image to **save the image**.
- 10 Click the button **Export** next to the spectrum to **save the spectrum, the table data, and the spectrum image (graphic)**.

Examples/hints

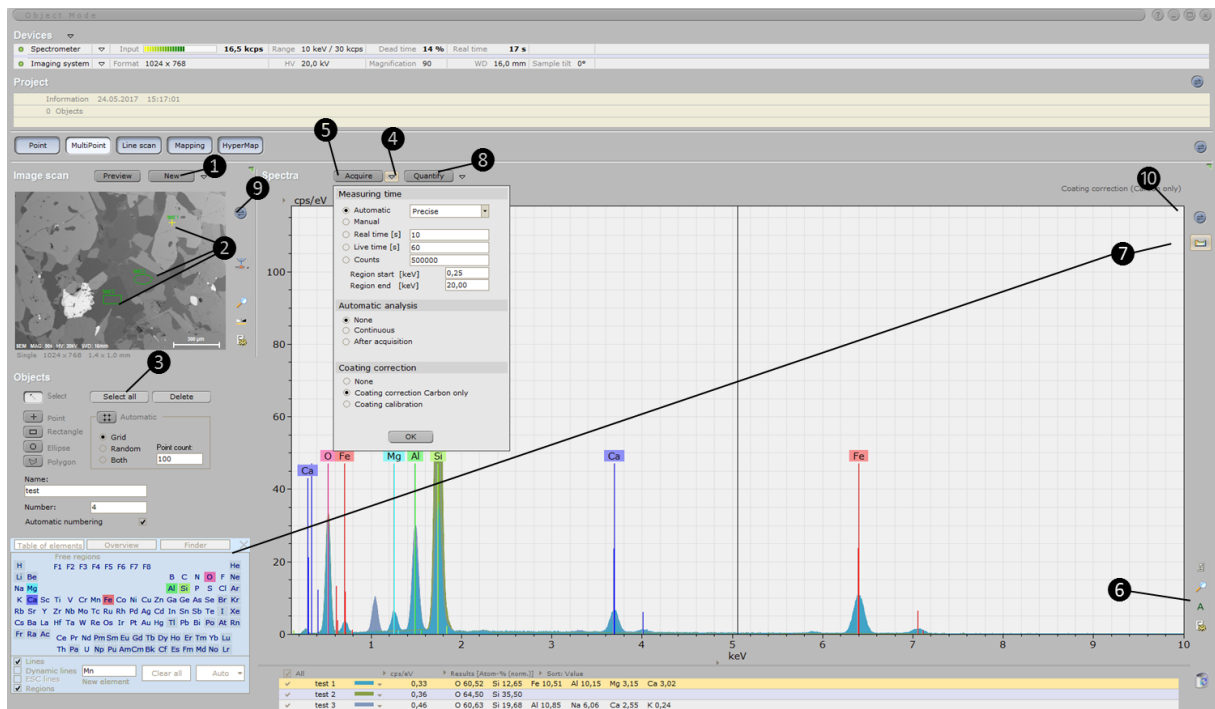
Objects are automatically analyzed in sequence, the exact procedure depends on the analysis mode settings.

If after the automatic search there are peaks that were not identified, place the marker on top of that peak and press **Finder** to get a list of suggested elements.

Save the spectrum in .spx, the table data in .xls.

Note that if you have analyzed several points you can save an excel table with all results by selecting all

spectrums lines (need to be all yellow) and then pressing the Export button and Save table.



7.4.3. Line fast scan

Step

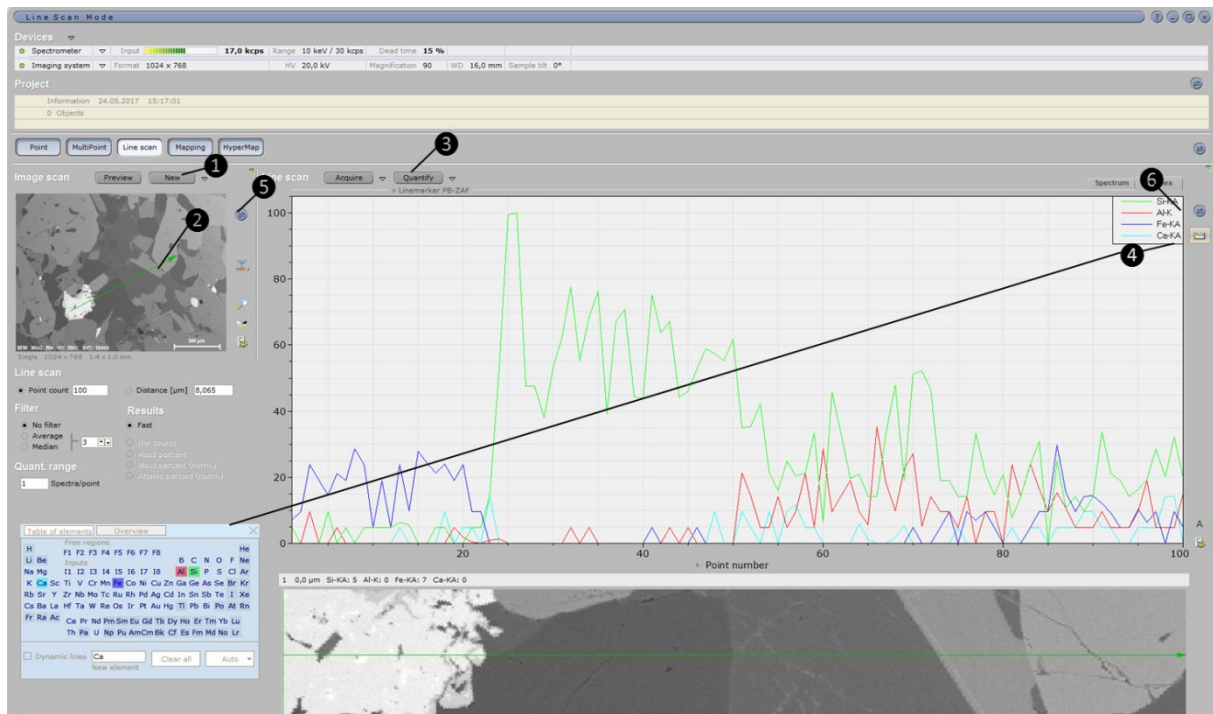
- 1 Click **New** to acquire an image.
- 2 Adjust the scan line if desired.
- 3 Select elements of interest using the **Interactive identification tool** (Periodic Table of the Elements).
- 4 Click **Acquire**.
- 5 Click the button **Export** next to the image to **save the image**.
- 6 Click the button **Export** next to the line scan to **save the line scan, the table data, and the line scan image (graphic)**.

Examples/hints

If the sample is of unknown composition perform step 4 first and then identify the elements by using the **Interactive identification tool** (Periodic Table of the Elements).

Adjust the acquisition parameters by clicking in the arrow next to the button **Acquire**.

Save the line scan .spx, the table data in .xls.



7.4.4. Mapping (fast)

Step

- 1 Click **New** to acquire an image.
- 2 Adjust the map area if desired.
- 3 Select elements of interest using the **Interactive identification tool** (Periodic Table of the Elements).
- 4 Click **Acquire**.
- 5 Adjust colors and image controls.

Examples/hints

Mapping elements has to be carried out before starting the acquisition.

Terminate acquisition when image quality is sufficient.

The acquisition parameters can be adjusted by clicking in the arrow next to the button Acquire.

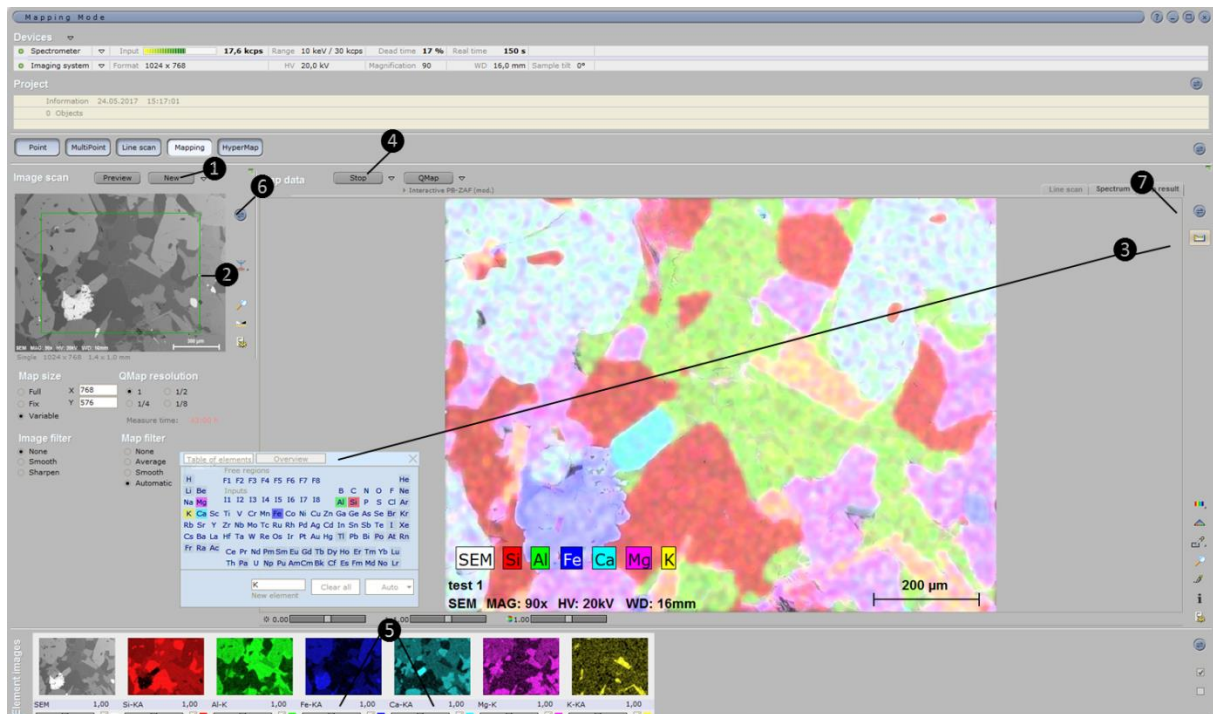
Image mixing can be changed at any time.

Try different image mixing modes for optimum results.

6 Click the button **Export** next to the image to **save the image**.

7 Click the button **Export** next to the map to **save map data and result table**.

Save the map in .spx, and the table data in .xls.



7.4.5. HyperMap

Step

- 1 Click **New** to acquire an image.
- 2 Adjust the map area if desired.
- 3 Set up acquisition by clicking in the arrow next to the **Acquire** button.
- 4 Select elements of interest using the **Interactive identification tool** (Periodic Table of the Elements).

Examples/hints

Preferably use manual start/stop or cyclic acquisition (total duration depends on pixel dwell time set for the image system).

In contrast to classical mapping you can select and deselect elements any time, also during acquisition.

5 Click **Acquire**.

Manual start/stop: for reasonably reliable quantification don't terminate acquisition before pixel spectra contain at least 5000 counts.

6 Adjust colors and image controls.

Image mixing can be changed at any time.

Try different image mixing modes for optimum results.

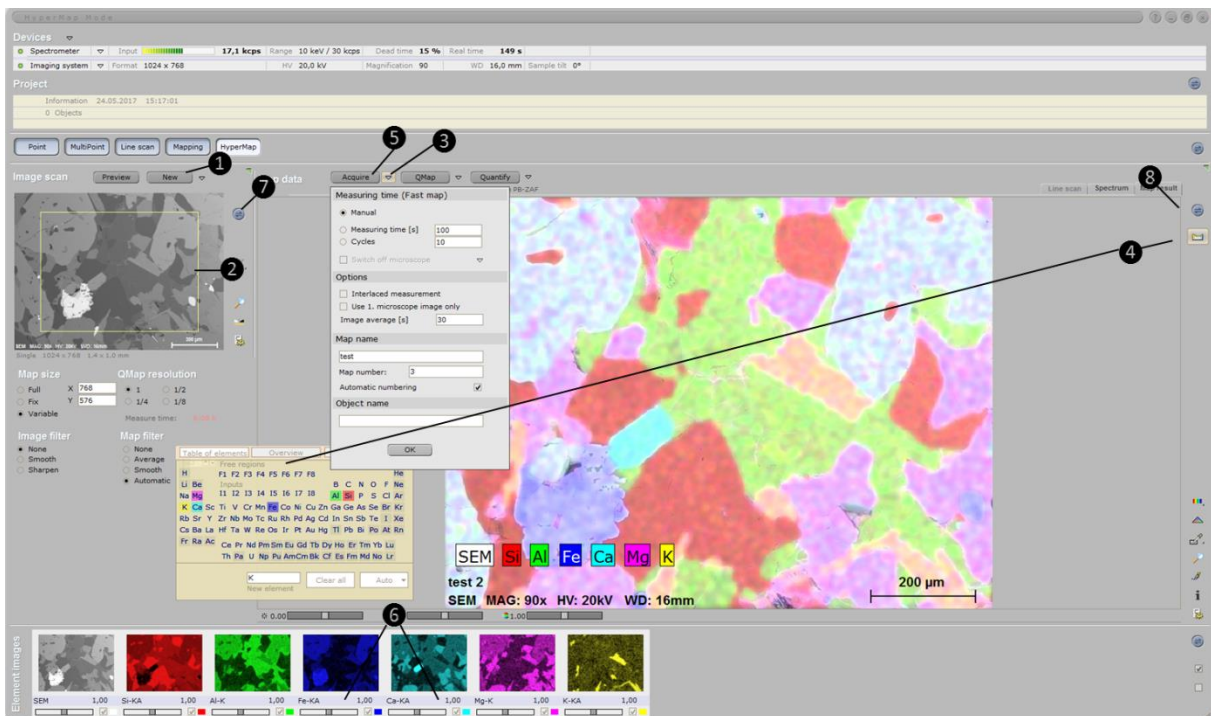
7 Click the button **Export** next to the image to **save the image**.

8 Click the button **Export** next to the map to **save Hypermap database, map data and result table**.

Save the map in .spx, and the table data in .xls.

9 Process Hypermap data as desired.

You can perform quantitative analyses, line scans, phase analysis, etc.



7.4.6. Creating a report

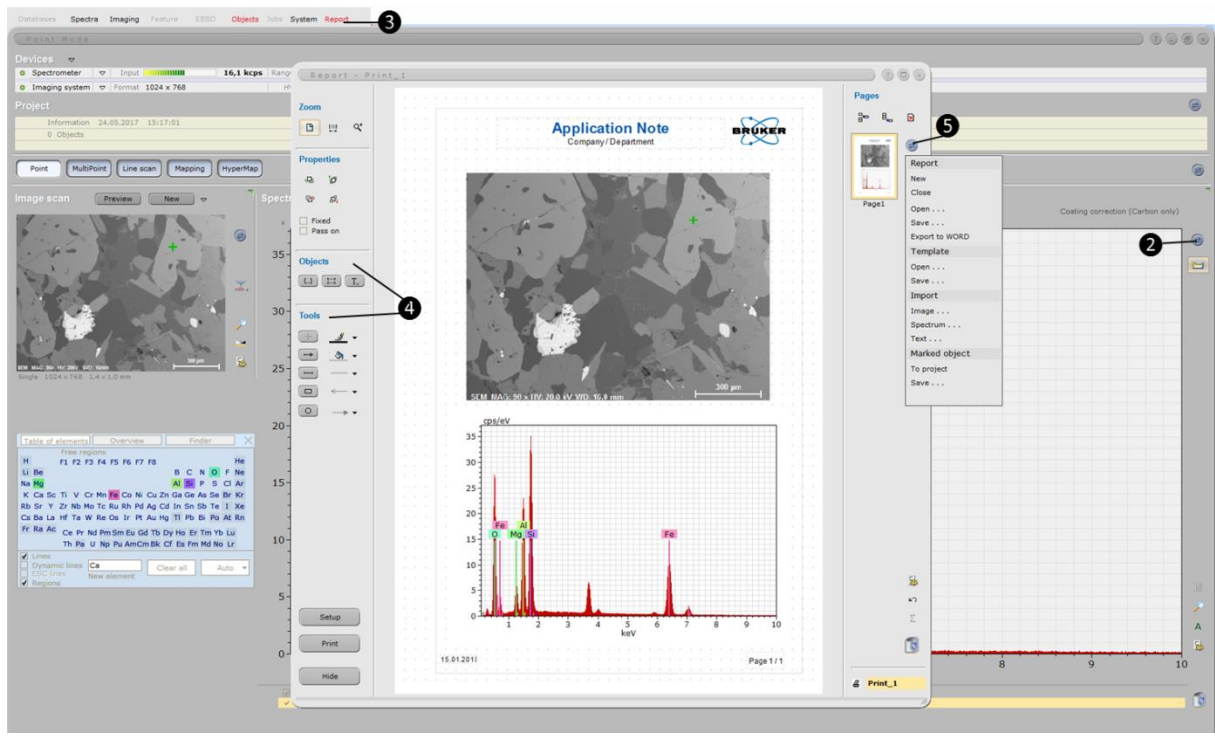
Step

- 1 Perform an analysis in a workspace of the Objects group.
- 2 Select **Add to report** from the **Export** menu.
- 3 Open the **report editor**.

Examples/hints

4 Add texts or legends, draw objects, or rearrange items as desired.

5 Save your work using the Report **Export** menu. Possible to save in word format.



7.5. Saving data

High resolution images will be saved in the **SEM computer** (Section 7.3). All files shall be saved in the folder *D:\User data* under a folder with your name (e.g. *D:\User data\Waldemar Brøgger*). There is a shortcut to this folder in the computer desktop.

Data from chemical analysis (e.g. spectra, data tables, images, maps, etc.) will be saved in the **EDS computer** (Section 7.4). All files shall be saved in the folder *C:\Quantax\Users\Edx\Data* under a folder with your name followed by EDS (e.g. *C:\Quantax\Users\Edx\Data\Waldemar Brøgger_EDS*). There is a shortcut to this folder in the computer desktop.

USB devices are NOT allowed in any of the SEM computers.

A copy of your data will be transferred to the following folder *\Økern-GeoLab\GeoLab_Users data_Temporary storage\“yourname”*

The data will be available from Monday the week after your booking and for a period of one month. Contact the lab manager or deputy if you do not have access to this folder or if you need your data earlier.

7.6. Shutdown

Step

Examples/hints

1 When the **accelerating voltage** is ON, click HV on the tool bar or OFF in the HV Control dialog box to turn **OFF** the voltage.

2 **Move the stage to the specimen exchange position** by pressing the **Go Home** button in the Stage Control dialog box.

CAUTION: If the stage is pulled out at other position than the specimen exchange position (Home), the specimen and instrument may be damaged.

3 Press **EVAC/AIR** switch on the evacuation control panel to set it at **AIR**. The LOW lamp comes on and air is introduced into the specimen chamber.

4 Pull out the specimen stage and **remove the specimen holder**. Remember to use gloves when handling the specimen holder to avoid contamination of the specimen chamber.

5 Select **SEM (High Vacuum Mode)** from the Vacuum Mode menu in Setup.

6 **Insert the specimen stage into the specimen chamber** and set the **EVAC/AIR** switch to **EVAC**.

7 Wait until the **HIGH lamp** comes on and the vacuum meter on the status bar turns blue (about 6 minutes).

8 Pull out the **BSE detector**.

9 Fill in the end time in the **logbook** (Excel file in the EDS computer) and save the document.

10 **Close the EDS program** but **leave the SEM program open**. **DO NOT turn off the computers**.

11 **Remove your sample(s)** from the **specimen holder(s)**. **Put the specimen holder(s) away** in the drawer. **Clean and organize** any material that you may have used to sample preparation. **Trash your gloves**.

If you need more details how to perform these instructions consult Section 7.2.