QUANTAX EDS
Energy-dispersive X-ray spectrometer for electron microscopy

- User Manual
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1 Introduction

QUANTAX is the ideal all-purpose energy dispersive microanalysis system for industry, research and education. Different system levels and various options are provided for scaling and tuning of QUANTAX to a broad range of analysis tasks and application environments. QUANTAX microanalysis systems are suitable for scanning electron microscopes, transmission electron microscopes, electron beam microprobes, dual beam devices, as well as other X-ray spectrometry applications.

All QUANTAX systems provide state-of-the-art qualitative and true standardless quantitative microanalysis for bulk specimens, polished samples, thin layers, particles, and rough surfaces. This is accomplished by optimized automatic or interactive spectrum analysis methods, fundamental parameter approaches (e.g. P/B-ZAF) and the most exact and comprehensive atomic database available. Different standard-based quantification packages are optionally available. The unique capability of combining standard-based and standardless analysis methods (hybrid quantification) completes the range of analytical possibilities.

An intuitive user interface, the flexible project management package, as well as various options for quick and comprehensive report generation complement the analysis toolboxes. The software tools are tailored to meet both the needs of the novice as well as the experienced user. All QUANTAX systems’ ESPRIT software includes an online help system and supports remote diagnosis and assistance via the Internet.

This manual provides a general software overview and a practical step-by-step description of the most common measurement procedures that can be performed with the QUANTAX EDS system. The integrated assistants and the online help of the ESPRIT software provide additional support. Details on the individual hardware parts and additional technical data are contained in separate manuals. The individual device manuals also contain further references regarding operational safety. Please follow all safety instructions closely to avoid hazards to personal safety and equipment. In case of necessary maintenance, reinstallation, severe computer breakdown, hardware changes, etc. the Bruker customer support or your local supplier have to be contacted for further assistance and instructions.

According to the modular structure of QUANTAX not all parts of this manual may apply in detail to a given installation nor should be taken literally in all cases. For instance, where the general term “electron microscope” (EM) is used, this applies to the whole class of electron-beam or dual-beam systems; SEM includes also STEM, microprobes, or other scanning devices.

The ESPRIT software layout may vary according to the modules installed on your system and therefore may differ slightly from what is shown in the illustrations and figures.
2 Safety Information

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Operation of the QUANTAX EDS microanalysis system is restricted to trained personnel familiar with the system as well as the product documentation, general safety precautions, and laboratory rules. A briefing on safety issues is given at the time of installation or during user training.

Local, state, or federal regulations have to be taken into account additionally to the safety instructions given here. Additional instructions for specific parts of the QUANTAX system - e.g. the X-ray detectors - may be contained in the according manuals.

The QUANTAX EDS microanalysis system may only be used in combination with electron microscopes or similar devices. Any other use beyond that is considered non-intended usage. The operator, not the manufacturer, assumes sole liability for all personal injury and material damage arising from non-intended usage.

2.1 Radiation Safety

The QUANTAX microanalysis system uses the X-radiation that is generated by sample interaction with electrons from the electron source of the microscope or photons emitted by the optional X-ray source. Microscopes and attachments approved by the microscope vendors are generally designed to shield this type of radiation sufficiently.

The QUANTAX XFlash® detector is installed according to the applicable radiation safety regulations. Changes to the original installation and equipment, including flanges, vacuum sealing, support, etc. are strictly prohibited.

In case the XFlash® detector has to be uninstalled, make sure that the original blind flange of the electron microscope vendor is used to seal the sample chamber port. In case the original flange is not available contact the microscope vendor for support.
2.2 Beryllium

**DANGER**

Beryllium is highly toxic!
If a beryllium window is destroyed despite all caution, absolutely all fragments must be carefully collected and disposed of according to regulations.

Beryllium safety issues apply only to X-ray detectors with beryllium X-ray entrance windows. Low energy windows do not contain toxic substances.

Beryllium is toxic if parts are inhaled or swallowed. With X-ray detectors, however, this is only possible in the rare case of a window breaking. Special care must be taken with XFlash® detectors, as they do not contain enough vacuum to suck in the remains of a broken window, as a UHV-Dewar normally does.

In case of an accidental destruction of a beryllium window, gather all fragments thoroughly and collect them using pieces of adhesive tape. The waste has to be labeled and disposed of according to the local safety hazard regulations. Wear a filter mask during cleaning up.

2.3 Electrical Safety

**DANGER**

High voltage inside. Do not remove covers.

All parts have been designed according to the safety requirements for electrical equipment for measurement, control and laboratory use, or the European Low Voltage Directive, respectively. The system must be correctly installed and used only for the purposes it is designed for. Especially grounding of the system or system parts - as performed during installation - must not be changed for any reason. In case of any supplements or replacement parts being installed, supply voltage settings must be checked and adjusted to the local mains voltage.

Certain parts of the system may contain dangerous voltages. No covers need to be removed during regular operation; maintenance should only be carried out by trained and certified personnel.
2.4 Electromagnetic Compatibility

Due to its physical construction, the highly sensitive X-ray detector may not fully comply with common standards regarding electromagnetic immunity of general electronic equipment. This is not a disadvantage under normal circumstances because the QUANTAX system works under the same conditions as electron microscopes and the laboratory environment is designed to meet the requirements of these instruments.

Bruker devices fulfill all requirements regarding active electromagnetic compatibility (emission rules).
3 The QUANTAX EDS System

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3.1 Hardware System Components

Typical QUANTAX EDS system parts
- XFlash® energy dispersive X-ray detector (s)
- Signal processing unit
- Interface and EM control
- QUANTAX server computer
- Optional client workstations
- ESPRIT 2 software package

System components of QUANTAX comprise the X-ray detector, interface hardware, one or more computers, and several software modules. All parts are selected for performance and trouble free interaction. Exchanging any part of the QUANTAX system with non-approved items (e.g. user defined computers) may impair the functionality.

3.1.1 XFlash® Energy Dispersive X-ray Detectors

Single photon counting. Energy dispersive X-ray spectrometers take advantage of the photon nature of light. In the X-ray range the energy of a single photon is just sufficient to produce a measurable voltage pulse at the output of an ultra-low noise preamplifier connected to the semiconductor detector crystal. The individual pulse heights are a statistical measure of the corresponding quantum energy. By digitally recording and counting a great number of such pulses within a so-called multi-channel analyzer (MCA), a complete image of the X-ray spectrum is built up almost simultaneously. This digital quantum counting technique makes the energy dispersive spectrometry extremely reliable.

Bruker XFlash® Detector. The Bruker XFlash® detector is an energy dispersive X-ray detector that works according to the principle of the silicon drift detector (SDD). It uses a special arrangement of drift electrodes and active components on the detector crystal chip to raise the signal strength to a level that can be measured even at room temperature.

XFlash® detectors are maintenance-free, durable, lightweight, completely vibration free, and do not require any consumables.

3.1.2 Signal Processing Unit

The signal processing unit is the heart of the spectrometer electronics. Different auxiliary utilities, like signal inspectors and count rate meters are incorporated in the signal processing unit, as well as all the necessary supply and operational circuits. The data interface connects the spectrometer and the QUANTAX server.
Spectrometry signal processing. Inevitable electronic noise requires the detector signal to be averaged over a certain time for the purpose of pulse height analysis. This averaging or noise filtering is performed by the so-called signal shaper. The necessary time per pulse (filter time) limits the maximum acquisition speed of a spectrometer. The applicable filter times are determined by the properties of the detector noise and the required energy resolution (peak separation).

Pulse throughput. With optimum signal shaper the QUANTAX the XFlash® can output 20 000 to 130 000 cps, depending on the type of detector used (refer to the detector’s hardware manual for specifications). Faster than optimum shapers can be selected at the expense of energy resolution, which is acceptable for ultrahigh-speed applications like element mapping.

The QUANTAX spectrometer electronics is configured according to the chosen detector and QUANTAX system level.

3.1.3 Spectrometry Data Interface and EM Control

Data interface. The proprietary Bruker MegaLink high-speed data interface transfers the spectrometer data in real time to the QUANTAX server. The PCI plug-in board Bruker IO-Scan can connect up to 4 spectrometers simultaneously. Bruker MegaLink is a serial interface protocol featuring full galvanic isolation for improved noise immunity at connection length of up to 30 m. For basic spectrometer functions that do not require real time spectrometry a standard computer interface (RS232) can be used as an alternative.
3.1 Hardware System Components

**QUANTAX scan system.** The PCI plug-in board IO-Scan also contains the necessary hardware for controlling the x/y electron beam deflection as well as two separate digitizers for the image signals of the electron microscope (EM). Position tagged spectrometry data acquisition is provided for advanced line scan and element mapping. Digital signal processing supports the server computer in time critical tasks. Control signals for hardware scan switching are provided as well.

**Switch Box.** The optional switch box allows sharing the external scan interface of the EM between QUANTAX and third party applications. The switching is fully automatic.

**Microscope data transfer.** Microscope data transfer is based on different networking technologies according to the type of microscope. For older brands, serial connections (RS232) are also provided.

**Client-server architecture.** All QUANTAX systems feature client-server architecture. The server part controls the spectrometer and microscope hardware and performs basic data evaluation; the client system is what is visible to the user and provides the graphical user interface (GUI). Client and server can share one computer or run on different computers. Multiple client workstations can connect to a QUANTAX server with network access (LAN).

**Server workstation.** Based on the operating Windows® system, the QUANTAX server can be used as standalone unit or network component. The connections to the optional client workstations are accomplished either via a separate network or via the intranet of the company or lab. With appropriate upgrades it is also possible to connect to wide area networks. The QUANTAX server performs the measurements and primary spectrum mathematics. It also provides the public and private data areas of the QUANTAX user.
3.2 ESPRIT 2

Software

**System requirements.** QUANTAX requires Windows® 7 with a service pack level specified at time of installation. Operating system updates are to be approved by Bruker Nano. Only computers provided or approved by Bruker Nano may be used.

Windows® administrator rights are required for the installation of QUANTAX. Ask your system administrator for help.

**Hardware driver.** During the first startup of the server PC the driver for the IO-Scan plug-in must be installed. The driver is available from the ESPRIT installation CD, which should always be at hand.

**License file.** A user and system specific license file is contained on a separate CD. During the initial start of ESPRIT the user is prompted to provide this CD for registration.

The license file (Licence.pdf) is stored to the server computer; after completion the license CD should be kept in a safe location.

**Software options.** Licenses control general access to the ESPRIT software as well as installed options. Selected options or the complete license can be time-limited. Updated license files obtained from Bruker Nano can be read in the System workspace (see section 4.7). Changes will be enabled after restarting ESPRIT.

The license is also provided as hard copy. The contents can be checked against the file, which can be opened by any pdf-reader or from the System / System workspace by clicking the Show button.

**Hardware key.** The system license is bound to a system specific hardware key coded into the QUANTAX server hardware. Client workstations can receive certification from a licensed server via the network. Standalone PCs (data stations) must be equipped with an optional USB dongle replacing the hardware coded key.

 Do not save the license with a pdf-reader. This procedure destroys the license.
USB Dongle. The QUANTAX dongle for standalone systems (systems without access to a QUANTAX server) fits any USB-port on the regarding PC. A software driver for the dongle must be installed during system installation; the dongle must be present to use the data station.

Install the QUANTAX base system selecting option Data Station (with dongle). Leave the ESPRIT install CD in the drive and plug in the dongle into a convenient USB-port. For details refer to the separate ESPRIT Installation Manual.

Windows® will detect the new hardware automatically and display an installation wizard. Select the options shown in the images below and finish the installation. Note that the dongle does not replace but only enables a valid QUANTAX license.

### 3.3 General Operating Instructions

#### 3.3.1 Detector Maintenance

The detector is the most sensitive part of the spectrometer. According to the type of detector at hand, different operations regulations apply. These must be followed strictly.

**XFlash® detectors.** Bruker XFlash® detectors are maintenance-free. There is no additional wearout caused by permanently powering the detector; nonetheless detectors for light element analysis may exhibit slight parameter changes after very long periods (several weeks) of continuous operation.

**Standby.** Standby mode for the thermoelectrically cooled detector can be chosen between work sessions to prevent unnecessary heating of the detector housing. From the standby mode, the detector can be quickly put back into a stable measuring mode. Standby mode can be entered via the Detector configuration dialog (see section 4.5.5) or by confirming the corresponding option when terminating a work session.
Cooling mode. A mode selection for the detector cooling is provided for XFlash® detectors. Maximum cooling will provide the optimum energy resolution. Thermostat mode will reduce possible changes due to varying environment temperature. Though these changes are normally negligible, thermostat mode is the proposed setting with most QUANTAX applications. Please check the XFlash® Reference manual to decide which of the above options is the most suitable for your purpose.

Detector window. The X-ray detectors are equipped with a beryllium window (detection range starting from sodium) or a light element window. Both kinds of windows are extremely thin, fragile, and contact sensitive. In general, cleaning of the windows is neither necessary nor permissible. If the detector window is contaminated, contact customer support or your local supplier for assistance.

3.3.2 Electronic Units

Setup. Unauthorized changes to the electronic and electric installation are not permitted. Besides safety regulations (see section 3.1.2) also electromagnetic immunity and noise pick up issues are addressed during installation.

Note that electronic parts may have to be adapted to the local mains voltages, if this should change for whatsoever reason. Also replacement parts have to be checked for proper settings before being connected to the mains supply. Details are described in the according reference manual.

Electrostatic damage. All electronic components and computers require the typical operating precautions for electronic products. Especially, all usual preventative measures against electrostatic discharge (ESD) must be carried out. All components of the QUANTAX system are designed for laboratory use only.

Cleaning. Covers of electronic compartments must never be opened. Only dry cleaning (dusting) of the outer compartments is permissible. The detector window must not be cleaned at all.

A broken detector window caused by any user interaction impairs warranty and can result in a total damage of the X-ray detector. Additionally, a broken beryllium window represents a substantial health risk.

The spectrometers are highly sensitive measurement devices, which can be impaired or destroyed by inappropriate use.
3.3.3 Software and Data Handling

**Backing up data.** Even state-of-art, complex and versatile software systems cannot be designed to completely exclude the possibility of damage or impairment by improper operation and/or misuse. Additionally no existing computer system is actually immune against potential hardware or software errors. Appropriate backing-up of all relevant data is strongly recommended. No warranty can be accepted in case of data loss for whatsoever reasons.

**Maintaining system integrity.** The QUANTAX system is installed on special selected and configured computers. Any change of the system configuration, user status, access rights, or else can impair system integrity. Also new applications, especially multi-media, internet, and game software can interfere with QUANTAX. The internal client-server architecture of QUANTAX depends on Windows® communication services that may be adversely modified by subsequent installations.

**Recovering from errors.** In cases of program lockup due to improper operation or unexpected circumstances a restart of ESPRIT is normally sufficient. In more severe cases a restart of the personal computer and/or the QUANTAX server may be required. More severe system breakdowns - if any - should be handled by trained service personnel.
4 The ESPRIT 2 Software

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4.1 Start-up and Login

The ESPRIT software runs on the QUANTAX server and the client workstations. However the user only has to start the client application; the server software and communication drivers will start and log in automatically.

**Start-up.** To start ESPRIT click the program icon on the Windows® desktop or in the Windows® Start menu. The ESPRIT login screen is displayed prompting user name and password.

**Login.** Enter user name and password and click the button. The password is stored on the local workstation, if the corresponding option is checked. The name of the last user is always retained.

**Multi-user systems.** On multi-user systems several users can access, manage, and evaluate private and shared data simultaneously. The unique user name and password will give access to the private data and settings. Select the server to log in from the pull down menu below after clicking on the arrow symbol of the button.

After logging in with a unique user name the current settings and data will be loaded from the private user profile volume on the QUANTAX server.

**Remote login.** With client-server installations (option LAN) it is possible to log in from any connected remote client workstation. The first user to start a measurement will gain access to the spectrometer and imaging system. This user will be able to perform measurements from the remote workstation. The access to the acquisition hardware remains locked to others as long as this user is logged in.

System functions, especially concerning the measurement system (e.g. calibrations) will affect subsequent users. It is advised to exercise special caution here.

Fig. 4.1-1 ESPRIT Login screen
4.2 User Administration

The QUANTAX communication server controls the user assignments as well as the internal and external client-server communication. Service tools and information for troubleshooting are also provided. The communication server runs on the QUANTAX server computer. It is also present in single computer and standalone systems.

Clicking the Bruker icon in the Windows® system tray of the server computer opens the console of the communication server. With exception of the actions described below no changes to the communication server settings must be made. Unauthorized altering of settings can render the system useless and lead to severe data loss.

New users are to be assigned from the QUANTAX communication server. Double clicking the Bruker icon in the Windows® system tray opens the console of the communication server.

The tab QUANTAX user lists the current assigned users. A new user can be added after clicking the button. A password can be entered (leaving the field empty is permitted) and an optional user description can be added. As user type User, Main user, and Supervisor can be selected.

User has limited access to the ESPRIT System workspace. This user level only allows access to the Appearance tab and can review the connected instruments and license.

The Main user has limited access to the ESPRIT System workspace. This user level has full access to the tabs System, Appearance and can calibrate the connected EDS detectors as well as the imaging system. The Main user has no access to the service settings of the WDS detector and the electron microscope driver.

The Supervisor has full access to all tabs in the ESPRIT System workspace.

Each new user is automatically assigned a folder structure in a private volume of the server. Default methods, a predefined user profile, and demo data are copied to the user folders.
4.2 User Administration

Altering user name and password can be done at any time from the QUANTAX communication server (icon in the Windows® task bar) by clicking Change in QUANTAX user tab.

The user data and profile are automatically copied to the new user data structures. Backups of important data, which are always recommended, are especially advisable when changing user names.

Normally there is no need to delete assigned users. If users have to be deleted, it has to be made sure that all user data to be retained are copied to public volumes.

To delete a user highlight the user name in the list provided in the QUANTAX user tab of the QUANTAX communication server. After clicking the deletion of the user data has to be confirmed separately. Caution, once confirmed, the deletion cannot be revoked! If deletion of the data is not confirmed, the data are retained invisibly; a new user with the same name as a previously deleted will be assigned to the idle data.

The number of assigned measure client ports determines how many users can log in simultaneously to the server computer. Per default only one port is assigned, so that only one user at a time can log in to the server computer. Even when multiple ports are present, the hardware can only be controlled by one user at a time.

Select a new port number in the field Measure client ports and click . Usually, the number following the last assigned port in natural order is adequate. Ask your system administrator for help.

The connection setting for a client workstation is accessed from the ESPRIT login screen. Select the entry Configuration from the Server dropdown list. The QUANTAX SERVER SETTINGS dialog will open.
A random name can be given to a connection. If the client workstation also runs a server, select “Local server” for a name and input network address 127.0.0.1. The port number must match the setting of the communication server (see section above); default is 6478. Ask your system administrator for help with network addresses and port numbers.

4.3 Workspaces

Workspaces are ESPRIT screen areas in which images and spectra are displayed, the analysis is performed and the results are managed. A workspace can be selected by clicking on the corresponding workspace icon. The active workspace icon is highlighted.

- **Assistants**
  gives access to a step-by-step guide for common analysis procedures.

- **Spectra**
  allows spectrum acquisition and analysis of (saved) X-ray spectra.

- **Objects**
  permits point, multipoint and area EDS analysis.

- **Line scan**
  is used to perform qualitative and quantitative line scan EDS analysis.

- **Mapping**
  allows the acquisition of maps: intensity maps and HyperMaps (Bruker’s position-tagged spectrometry tool (spectral imaging)). Mapping also includes the chemical phase analysis tool **AutoPhase**.

- **Imaging**
  allows capturing and processing saved electron microscope images.

- **Feature**
  is used to perform particle analysis and chemical classification. Refer to the ESPRIT Feature User manual for details.
4.4 Display Control

EBSD

gives access to the fully integrated EBSD (electron backscattered diffraction) analysis package. Refer to the QUANTAX EBSD User manual and the EBSD Quick reference guide for details.

Jobs

is the workspace for configuring ESPRIT’s powerful analysis automation tool and running unattended fully automatic analysis tasks.

Scripting

permits writing and running user-defined scripts.

System

is used to adjust global settings and calibrate the X-ray spectrometer and image system. Additional functions: display settings (language, font size); license management; communication with electron microscope and motorized sample stage.

Program window and title bar. The title bar at the top of the ESPRIT program window contains the general control buttons common to the Windows® applications.

The complete ESPRIT window can be resized by dragging and pulling the handle on the bottom right-hand corner or on one of the border lines. To switch to full screen display, click the icon in the title bar; switch back by clicking the icon. The icon minimizes the program window. The icon activates the program help system. Please note that the icon closes ESPRIT completely, not just the active workspace.
To scale individual parts of the program window the splitter (dark grey lines) between different screen areas on the ESPRIT interface can be dragged (a changing mouse cursor indicates availability of this option).

**Full screen / dual screen mode.** An image or a diagram chart can be decoupled from the ESPRIT window and switched to full screen by clicking the full screen symbol on the upper right-hand corner of the corresponding screen area.

If a second monitor is attached, the decoupled window can be dragged and pulled to the second monitor and ESPRIT can be used in extended display mode. Revert from full screen mode by pressing the ESC key or clicking the full screen symbol again.

**Configurator bar.** The configurator bar compiles all attached hardware parts as well as information about the sample and standards, the scan settings and the report and project editor. All parts of the configurator bar are described in detail in section 4.5.

**Image capture chart.** The workspaces Objects, Line scan, Mapping and Imaging have a dedicated chart for controlling the electron image capture. Analysis spots, objects, scan lines, and mapping areas can be defined on the captured image. Image capture is controlled directly from the current workspace. Additionally, a special window for fast image preview is provided. For details please see section 5.3.

One or two of the image detectors (typically the SE and BSE detectors) can be connected to the QUANTAX hardware and selected for image capture.

**Spectrum and Result chart.** The workspaces Spectra, Objects, Line scan, and Mapping have a dedicated chart for displaying spectra and associated spectrum list or results. Details are described in section 5.8.

**Icons and buttons.** A number of icons and buttons associated to the different workspaces and display charts are provided. All icons can be displayed in normal or large style button mode which can be changed by activating/deactivating the corresponding checkbox in the System/Appearance workspace.
4.5 Configurator Bar

Using the triangles at the control buttons opens drop-down menus to enter acquisition and analysis options. Diagram axis labels and column titles can be selected with .

Data lists and thumbnail images will be highlighted by clicking onto them. Multiple items can be selected by using the SHIFT or CTRL key to perform batch processing.

4.5 Configurator Bar

The configurator bar consists of a horizontal row of buttons (configurators) in the upper part of the ESPRIT window that allow the configuration of the hardware components available in the QUANTAX system. The number of configurators that actually appear on the screen depends on the license and may differ from the images shown in this manual.

The individual configurators display relevant adjustable parameters of the sample, standard libraries, X-ray excitation sources, scan unit, spectrometers and other detectors, as well as the report and project tools. They will be explained in detail in the next sections.

The configurator bar is resizable by moving the lower splitter (dark grey line). Pulling the splitter upwards minimizes the configurator bar. When minimized, the individual configurators show only one parameter.

The parameter to be displayed can be set by the user by clicking with the right mouse key on the displayed value in the configurator and selecting the desired parameter in the Select entry menu.
Use the downward-facing arrow in the left bottom corner of a configurator ( or icon, depending on the display mode) to access the corresponding configuration dialog.

4.5.1 Sample Configuration

The Sample configurator is used to display and manage information about the sample. The information displayed is divided into the following parameters:

- Sample type
- Coating type
- Company
- Batch
- Sample

The parameter to be displayed can be set by the user by clicking with the right mouse key on the displayed value in the configurator and selecting the desired parameter in the Select entry menu.

When clicking on the icon in the bottom left corner of the Sample configurator, the SAMPLE PROPERTIES dialog pops up. Before measurement, general properties of the sample can be entered in this dialog. All subsequently collected data (spectrum, image, line scan, map) will contain these parameters. Default input parameters are name and description. Additional user-defined data can be added clicking the icon.

Sample coating. If the sample is coated to enhance electrical conductivity, this can be entered under Sample coating.

- None. Select this option, if the sample is not coated.
- Coating with. Select this option, if the sample is coated. Select the element of the coating material in the periodic table which pops up after selecting this option. The selected element will be considered for the peak deconvolution algorithm but will not be quantified. The display changes to Coating with “Element name”.
- Coating calibration. Select this option, if the sample is coated and a coating correction data file (.ccc) is available. Open the *.ccc file in the popup dialog.

4.5.2 Standards Configuration

The Standards configurator is available, if a standard-based EDS quantification license is
4.5 Configurator Bar

activated. The configurator displays the following information about the system and the standards library:

- Number of standards in the standards library
- Calibration date (of the system factor)
- Calibration time (of the system factor)
- HV
- Geometry

Standards library. When clicking on the icon in the bottom left corner of the Standards configurator, the ADMINISTRATION OF STANDARD LIBRARY dialog pops up. For further details see section 5.12.4.

System calibration. When clicking on the icon in the bottom left of the Standards configurator, the SYSTEM FACTOR CALIBRATION dialog pops up. For further details refer to section 5.10.

The Microscope configurator displays the main parameters of the electron microscope.

- Magnification: Image magnification set on the electron microscope.
- High voltage: Acceleration voltage set on the electron microscope.
- WD (working distance): Sample – pole piece distance [mm].
- Stage X, Y, Z: Microscope stage position coordinates are displayed, if available

The parameter to be displayed can be set by the user by clicking with the right mouse key on the displayed value in the configurator and selecting the desired parameter in the Select entry menu.

When clicking on the icon in the bottom left corner of the Microscope configurator, the MICROSCOPE CONFIGURATION dialog pops up. If the data communication between microscope and ESPRIT is active, the microscope parameters Magnification, High voltage and Working distance will be transferred from the microscope to ESPRIT and displayed in this dialog.

Communication active. This option should be
Because sample tilt affects the take-off angle and hence the Bremsstrahlung background, its value affects the quantification results.

4.5.4 Scan Configuration

The Scan configurator displays the scanning parameters:
- Image resolution: Image size in pixels
- Dwell time: Signal collection time of a single pixel during image acquisition
- Frame time
- Drift qual.: Quality of image shift calculation
- Drift range: Absolute value of current image shift

The parameter to be displayed can be set by the user by clicking with the right mouse key on the displayed value in the configurator and selecting the desired parameter in the Select entry menu.

When clicking on the icon in the bottom left corner of the Scan configurator, the SCAN CONFIGURATION dialog pops up.

Image resolution. The entered value applies to the x-axis of the image. Since the aspect ratio of the image is defined by the microscope and set during installation, the value for the y-axis is set automatically and is not displayed.

Image inputs. If more than one image detector is connected to the QUANTAX scan system, the checked to maintain communication between the microscope and the ESPRIT software, otherwise relevant microscope parameters for image scaling and quantitative analysis will not be transferred to ESPRIT. The communication is set up during installation and automatically enabled after starting ESPRIT.

If the communication is disturbed or not available with the current type of microscope, the microscope data has to be entered manually. To disable automatic data transfer intentionally, uncheck the control box Communication active.

Sample tilt [°]. If the sample is tilted, the value of the sample tilt (in degrees) has to be entered in this field manually.

Additional tilt correction. Check this option, if the sample is tilted and the microscope has no image tilt correction capability. This is recommended when performing EBSD analysis using large tilt angles to correct image distortion of microscope images, element or orientation maps.
The dwell time can only be set in increments of numbers which are a power of two. Direct input of arbitrary numbers is not possible. Use the arrows in the input field.

Very high image resolution will slow down operation.

For Imaging, Mapping and Line scan different timing settings are maintained. However, the electron image captured in the left image window of each workspace always follows the settings for Imaging.

The scan speed can also be set from the dialog that opens after clicking the icon next to the image chart.

Imaging. The Dwell time is the time the electron beam stays on a pixel while capturing an image. Select expanded dwell times to allow filtering and averaging of the image signal and obtain less noisy images. The Line average factor controls the number of times a horizontal line is scanned in the slow scan direction and averaged before proceeding to the next line. Increasing this factor provides noise reduction and expanded image scan time while limiting the increased stress on the sample by maintaining a high scan speed. The line average factor will multiply the total image acquisition time. The total Image capture time is calculated and displayed.

Mapping. The Dwell time is the time the electron beam stays on a pixel while acquiring an EDS map. The Line average factor for mapping controls the number of times a horizontal line is scanned in the slow scan direction and averaged before proceeding to the next line. The capture time (Mapping scan time) for one mapping frame is calculated by multiplying the numbers of pixels and lines with the dwell time and the line average factor. The real scan time may be longer due to the time needed for data processing by the computer.

Drift correction. When clicking on the Drift correction button, the IMAGE DRIFT CORRECTION SETTINGS dialog pops up. It is described in detail in section 5.5.

Power synchronization. When the according control box is checked, the scan process is synchronized to the cycles of the AC mains voltage. Power synchronization substantially reduces blurring of vertical lines caused by electromagnetic interference (at the expense of the scan speed). It is applicable for capturing any image or map. However, it is normally only useful in connection with very high magnification of the microscope. A special AC/AC adapter to be connected to the QUANTAX server is provided.

Hardware inputs. The IO scan card provides 8
4.5.5 EDS Configuration

The EDS configurator displays the measuring parameters of the available EDS detector(s).

- Pulse rate (input count rate)
- Energy range
- Maximum pulse throughput
- Temperature
- Real time
- Live time
- Dead time

The parameter to be displayed can be set by the user by clicking with the right mouse key on the displayed value in the configurator and selecting the desired parameter in the Select entry menu.

The rectangle in the upper right corner of the EDS configurator indicates the status of the instrument. A green rectangle means that the detector is activated and ready to use, a red rectangle indicates that the detector is idle. Toggling between activated and deactivated states can be achieved by clicking on the EDS configurator.

When clicking on the icon in the bottom left corner of the EDS configurator, the EDS DETECTOR CONFIGURATION dialog pops up. The following parameters can be set:

**Pulse throughput.** This control box lists the signal processor settings available for the spectrometer hardware. When Automatic is checked, QUANTAX selects the throughput setting according to the current pulse load at the beginning of a spectrum acquisition or measurement.

**Maximum energy.** The available energy ranges are listed. When Automatic is checked, QUANTAX sets the energy range according to the microscope high voltage (kV).

**Standby mode** can be activated from the device control box or when terminating the work session. At start-up, detectors in standby mode will automatically be switched to full operation. Detectors are not switched to standby mode by deselecting them in the configurator bar.
4.5 Configurator Bar

Cooling mode. The XFlash® detectors can be operated in Thermostat mode, where the Peltier cooling operates at constant temperature (default setting) or in Maximum cooling mode, where the Peltier cooling operates at full power.

Detector segments. If the detector has multiple segments (or if several detectors were installed as such), only one EDS configurator is shown in the configurator bar. In this case the detectors or detector segments are listed as Detector segments. The individual detectors can be selected by enabling the respective checkboxes in the detector segments list.

Multiple detectors. Multiple detectors can be registered with individual configurators. In this case, parameters of the spectrometers have to be set individually. When multiple detectors are used, activate the detectors by clicking on the corresponding configurators.

Motorized detector axis. If the EDS detector is equipped with a motorized axis, the icon in the bottom left corner of the EDS configurator (see Fig. 4.5-17) displays and controls the position of the EDS detector. The icon shows one of the two states:

The EDS detector is inserted and is at measurement position, if the icon is displayed. The system is ready for use. When clicking on the icon in this status, the message Detector will be moved to park position pops up and after confirming with OK, the detector will be retracted.

The EDS detector is retracted and in park position, if the icon is displayed. When clicking on the icon in this status, the message Detector will be moved to acquisition position pops up and after clicking on OK, the detector moves to the measurement position.

If additional detectors or X-ray sources are attached, their configurators are displayed in the configurator bar.

EDS detector(s). If additional EDS detectors are used, the parameters of each individual detector can be set in the individual configurators. For details see also the XFlash® Reference Manual.

4.5.6 Additional Configurators
4.5.7 Report

WD spectrometer. If a WD spectrometer is attached to the system, it can be configured under the WDS detector configurator. For details see the QUANTAX WDS User Manual and the XSense Reference Manual.

EBSD detector. If an EBSD detector is connected, it can be configured under the EBSD detector configurator. For details see the QUANTAX EBSD Quick Reference Guide and the eFlash Reference Manual.

X-ray source. If an X-ray tube is connected, it can be configured under the X-ray source configurator. For details see the QUANTAX Micro-XRF User Manual and the XTrace Reference Manual.

The Report configurator shows the number of opened reports, the number of pages, objects and the name of the selected report.

Clicking on the icon in the bottom left corner of the Report configurator opens the report editor. Further details on how to use the report function are described in section 5.21.

4.5.8 Project

The Project tool serves as data clipboard for data transfer between ESPRIT workspaces or save all data added to Project as one file to disk (.rtx file format). Images, spectra, element selection, quantification methods, result tables, map/phase images, line scan data (including point spectra) can be added to and stored in a project.

The Project is either docked into the configurator bar on the top of the screen or docked onto the right side of the screen above the Report/Report preview. The two positions of the Project (and Report) can be toggled by clicking on the icon next to the project name. See section 5.22 for further details.
4.6 Loading and Saving Data and Results

Loading and saving data or adding to project or report can be done in two different ways: using the Import/Export function (icon) or with a right mouse click into the data field.

A local, workspace-relevant or chart-relevant menu pops up providing the options:
- Load
- Open
- Add to project
- Add to report
- Save
- Copy
- I/O
- Print
- Export
- Properties.

Loading data. Data files can be loaded or imported into the current project, into an ESPRIT report or directly into the workspace. To open the file browser for loading data into the workspace, open the according Import/Export menu (icon) and click Load (see also sections 5.21 and 5.22 for information on loading reports and projects, respectively).

Enter a filename or select a file in the list. A preview image and an info box will be displayed, if applicable.

The private data volume on the QUANTAX server is selected via the topmost icon labeled with the user name. To open the shared server folder or access the local or network drives, click the button labeled Public or a drive letter in the list below.

Any volume can contain an unrestricted number of folders and subfolders. To go one level up, click the icon in the headline or use the Backspace button.

A list of file types selects multiple files for browsing. Only file types selected under File type are shown in the browser.

Spectra can be loaded to spectrum charts; images can be loaded to the processing area of the Imaging...
workspace or to the Report. Other data types including result tables and free texts can be imported to the Report or to the current Project. Multiple spectra can be selected for loading by holding the SHIFT or CTRL key when clicking onto the file list.

**Saving data to files.** To save ESPRIT items (spectra, images, etc.) open the Import/Export menu by clicking the Import/Export icon in the according spectrum chart or image chart. Decide whether a graphic representation or data file is appropriate and click Save below the corresponding headline. A browser for saving files will open. The same selection of volumes and folders is provided as for loading data.

In the file browser enter or edit the file name and check the desired data format.

**Graphical data output.** Graphic export is possible using common graphical file types or the Windows® clipboard. To export graphical data to files use the option Save under the subsection Graphic (Image, Map image). To exchange graphics via the Windows® clipboard, click Copy in the according subsection of the Import/Export menu or in the local menu after a right mouse click into the corresponding window area.

**Projects.** Storing data in a project and saving the whole project to a project file is the recommended way of storing data. Working with projects is described in section 5.22.

**Result tables/lists.** Qualitative and quantitative results are automatically included in spectra files. To save result tables separately, open the spectrum chart Import/Export menu (Import/Export icon) and Save under Result list/tables or use the local popup menu after a right mouse click into the result table.

**Element selection.** Element selections are a special data type within QUANTAX that control the display of line markers and provide element lists for spectrum quantification, line scan and mapping. Element selections can be saved to file, transferred between workspaces using Copy and Paste or added into a project (see also section 5.22).

**Twain export.** Images or other data from ESPRIT can be exported via the standardized interface Twain export. It is enabled only when data is being requested by image processing applications.
4.7 System Functions

Attention!
Any changes in the system settings will influence the global ESPRIT software. The user must proceed with caution when altering the system settings!

Clicking the button in the main menu gives access to the System workspace. This workspace is divided into seven sections (tabs): System, Appearance, Spectrometer, Imaging, WDS, Microscope and Stage.

![System workspace](image)

Fig. 4.7-1 Workspace System

4.7.1 System

The section System provides product information, user license administration, and control of the spectrometer settings. All items in this section are generally only of interests to Bruker service personnel. Unless explicitly requested, please do not alter any parameters within the system assignments.

System report. This area lists all relevant system settings. The report is valuable for troubleshooting and service. If requested by Bruker service personnel, the report can be exported via the icon.
The product registration is only valid for the specified system number. The system number of the current hardware is also listed in the product registration area.

Export system files. This dialog allows for export all system relevant setting files as a .zip file.

Product information. The current version of the ESPRIT software is displayed here. Please relay this information to Bruker Service upon service enquiry.

Product registration. The product registration area shows the owner’s registration information. Further details can be accessed by clicking on the button. This will launch the PDF reader to show the current product license. If a new license file is issued, the file in the ESPRIT software can be updated by clicking the button. A copy of the license file is stored to the QUANTAX server’s profile folder. The license will be checked automatically by every time the ESPRIT software is started.

Devices. The assignment and setup of the X-ray spectrometer(s) and other devices are normally only performed during system installation.

The assignment is completely automatic. Each detector to be assigned must be connected to either Bruker MegaLink port, a RS232 port, an USB port or via Ethernet of the QUANTAX server and powered on when the button is clicked. All available ports are automatically scanned for valid connections. Once the spectrometer is located, the ESPRIT software must be restarted in order to register the newly found device.

After the restart of the ESPRIT software the available detectors are listed within the Configurator bar (refer to section 4.5).

Clicking the button opens a dialog for setting the type of spectrometer, the type of the radiation entrance window, and the geometry parameters. These entries are loaded from the detector description file or the detector data sheet.

The motorized detector support can be activated here. In order to record the acquisition position, click the button and follow the wizard.

The Command field next to the button performs low-level command functions. This is for troubleshooting purposes by system engineers.
4.7 System Functions

4.7.2 Appearance

The Appearance tab allows the user to configure settings related to the display of the ESPRIT software.

The subsection Languages allows the user to change the display language. Select a language and confirm by clicking Accept.

The GUI style allows the user to change the ESPRIT interface to the dark color scheme for use in low light conditions (e.g. in TEM rooms).

The subsection Save image options permits the user to reduce the quality of the images saved in jpeg format, in order to keep to the file size low. For images saved in TIFF format, two types of gray palettes can be selected: Normal TIFF assigns black to 0, and Inverted TIFF assigns white to 0.

In the subsection Miscellaneous options the user can activate/deactivate the Large style buttons mode. If the checkbox is activated, the name of the workspace or function is shown on the icon, e.g. / . The Auto close delay option defines the time the mouse-over hint messages of the icons are displayed.

The Colors for overlay objects in different workspaces can be selected in this subsection. Overlay objects are objects (points, lines, rectangles, ...) to e.g. extract and display spectral information from the mapping data cube.

In the Chart settings tab, font sizes and properties of the spectrum chart can be modified.

To use a fixed color setting for a particular chemical element, click on the element symbol on the periodic table to change the elements properties. If no specific color is defined, a random color is assigned.
The option **Excluded elements in ‘Finder’** should be checked by default. This allows to see all possible elements when using the finder, even those that are excluded for the Auto ID (the Auto ID works best especially for noisy spectra, if the user excludes some elements that are not expected in the sample). The box should only be unchecked, if the excluded elements should not be shown in the Finder.

In the **Table settings** tab, the user can specify the default settings for the export of tables.

In the **Spectrum tables** tab, it is possible for the user to select the type of information the quantification table should display post quantification.

### 4.7.3 Spectrometer

**Attention**

The spectrometer calibration is stored in the hardware and affects all subsequent uses and users.

> Do not interfere while the automatic calibration is running. If necessary, click the button to terminate the sequence.

> The calibration timing schedule should be adjusted to the laboratory regulations or practice and constraints defined by the application.

Spectrometer calibration refers to the realignment of the energy axis (abscissa) on the EDS spectrum using a calibration sample (more later) with known composition. The calibration is recommended for high precision quantitative analysis, or in instances where the line markers do not match the peak positions, or if the auto identification fails.

**Pulse throughput and Range selection.** The user can choose the different combinations of pulse throughput and energy range for spectrometer calibration. For each combination, the calibration data is maintained separately.

**Calibration.** Follow section 5.2 to perform energy channel calibration. It is possible to return to predefined settings by clicking the button if the peaks in the spectrum deviate severely from theoretical values.

**Calibration report.** The calibration report lists all relevant calibration settings of the QUANTAX system. If requested, please provide this document to the Bruker service personnel.
4.7 System Functions

4.7.4 Imaging

The **Imaging** tab gives access to image and electron microscope (EM) stage calibration.

**Image input.** The image input links the communications between ESPRIT software and the EM. The settings here are microscope dependent and should only be modified by Bruker service personnel.

**Image calibration.** The image calibration is performed to calibrate the pixel values within the ESPRIT software. It is possible to calibrate based on the electron microscope scale bars, but user should ensure that the pixel values on the microscope is up to date before performing image and stage calibration in the ESPRIT software.

A standard calibration grid or any specimen that has features of known geometry can be used for image calibration. After capturing an EM image, locate the features of the image by the green overlay line. To adjust the line, click on the line once to show the sizing handles, then drag the handles to fine tune the position of the end points of the line.

Once confirmed, enter the correct/certified values of the line length in µm. Click the **Accept** button to store the according calibration value.

**Stage calibration.** This option calibrates the stage movement alongside the SEM image for montage purposes. Before stage calibration, user must ensure that the line length (pixel calibration) is properly calibrated.

To start calibration, capture a SEM image, then open the acquisition settings by clicking on the **Acquire** button. The settings that can be changed here include the percentage of overlap (it is advised to use the same overlap value for actual montage acquisition in other modes), 2 x 2 or 3 x 3 images. Click on the **Acquire** icon again to confirm the settings.

For best results, it is recommended to switch off the scan rotation on the SEM to ensure that the stage movement matches the scan direction. Click the **Acquire** button to start image acquisition. A progress bar will show the progress of the image acquisition.
Once all images have been acquired, fine tune the overlap in the X/Y directions or the rotation and press ENTER on the keyboard. The correction in X will only change the overlap in the X-direction, the same applies to Y. The rotation will change the relative rotation between adjacent fields. In the image, the red curly brackets highlight the misaligned overlaps. Adjust the overlaps and rotation in small steps. Care should be taken to focus not on the corners of the image overlaps but at the straight edges (in the corners the image distortion is at its worst).
4.8 The Help System

ESPRIT features a multi-level help system. Descriptions are provided for all major control elements. Dialog boxes pop up, if manual interaction is required. Warnings to prevent performing undesired actions are displayed to the user.

Program help. The comprehensive user manual can be accessed from the program window using the icon.

Context sensitive help. The mouse cursor is context sensitive. When moving the mouse over objects, e.g. buttons and labels containing short explanations are displayed.

Assistants. Interactive assistants provide step-by-step instructions for common analytical tasks.
5 Step-by-Step Guides

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This chapter gives the analyst an overview of the most important tools of the ESPRIT software, permitting a quick start to perform analyses with the QUANTAX EDS system.

The procedures described here assume standard (default) ESPRIT settings. They also refer to the use of a scanning electron microscope (SEM) or a scanning transmission electron microscope (STEM) or similar. Differences in the analysis of bulk samples (mainly SEM and FIB) and electron transparent samples (TEM, STEM, sometimes SEM or FIB) are pointed out.

5.1 Preparatory Steps

This section describes the actions needed prior to starting the analytical work with the QUANTAX EDS system.

<table>
<thead>
<tr>
<th>Step</th>
<th>Examples/hints</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Prepare the sample and place it in the microscope.</td>
<td>e.g., embedding, polishing and carbon coating, TEM sample preparation</td>
</tr>
<tr>
<td>2. Adjust high voltage and working distance (SEM) or eucentric height (TEM) on the microscope. Switch on the beam. Adjust magnification.</td>
<td>Choose high voltage (HV) based on overvoltage of elements to analyze.</td>
</tr>
<tr>
<td>3. Start ESPRIT and log in.</td>
<td>Refer to section 4.1.</td>
</tr>
<tr>
<td>4. Use the icon in the bottom left corner of the Microscope configurator to open the MICROSCOPE CONFIGURATION dialog and check the settings.</td>
<td>WD, Magnification, HV values must match values set on the microscope (see section 4.5.3). Device status indicator should be green.</td>
</tr>
<tr>
<td>5. Use the icon in the bottom left corner of the EDS configurator to open the EDS CONFIGURATION dialog.</td>
<td>Normal operation mode should be selected. For Pulse throughput and Maximum energy use Automatic settings. Change it on demand.</td>
</tr>
<tr>
<td>a) Set Pulse throughput and Maximum energy.</td>
<td>Check operation temperature. Device status indicator should be green (see section 4.5.5). The input count rate should be chosen according to the analytical task.</td>
</tr>
<tr>
<td>b) Check Detector temperature.</td>
<td>The sample name applies for all further measurements.</td>
</tr>
<tr>
<td>6. Check input count rate and dead time for the analysis.</td>
<td>Refer to section 5.7, 5.14, 5.15 or 5.17.</td>
</tr>
<tr>
<td>7. Use the icon in the bottom left corner of the Sample configurator to open the SAMPLE PROPERTIES dialog and set sample name and description.</td>
<td></td>
</tr>
<tr>
<td>8. Proceed with required analysis.</td>
<td></td>
</tr>
</tbody>
</table>
Fig. 5.1-1 Preparatory steps for measurements with the ESPRIT software
5.2 Energy-Channel Calibration

Correct energy-channel calibration is a prerequisite for reliable qualitative and quantitative analysis results. Calibration should be checked on a regular basis or after re-start of the signal processing unit (SVE).

**Step**

1. Select the [System] workspace and the [Spectrometer] tab.

2. Set a calibration sample into the analysis position.

3. Set the beam current to produce an intermediate count rate.

4. Select element and spectral line for calibration.

5. Click [Test] to check the current energy channel calibration and deviation.

6. Select [Pulse throughput] and [Energy range] under [Settings to calibrate].

7. Choose accuracy and click [Start].

8. Click [Yes] to save new calibration data.

**Examples/hints**

- Do not confuse energy-channel calibration with system factor calibration (relevant for standard-based quantification; for details see section 5.12.3 and 5.12.4).

- Use a sample with any element in the mid energy range (Mn – Zn) without peak overlap and sufficient concentration. Preferably use single element standards (e.g. pure copper) to avoid peak misidentification, improve speed and accuracy.

- Use microscope controls. Set appropriate magnification on a homogeneous sample area.

- Use Kα lines whenever possible.

- The test corresponds to the current Pulse throughput and Maximum energy displayed in the EDS configurator. The acceptable deviation value is between + 5 eV and - 5 eV. If not, proceed with step 6.

- Recommended value is Medium.

- The calibration data is not user specific. Changes in calibration will affect all users of the ESPRIT software.
Fig. 5.2-1 Spectrometer calibration
5.3 Image Capture

This section describes how to capture electron microscope images with the ESPRIT software.

### Step

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Select a workspace (<em>Objects, Line scan, Mapping, or Imaging</em>).</td>
</tr>
<tr>
<td>2</td>
<td>Use the icon in the bottom left corner of the Scan configurator to open the SCAN CONFIGURATION dialog.</td>
</tr>
<tr>
<td>3</td>
<td>Set image resolution (pixel).</td>
</tr>
<tr>
<td>4</td>
<td>Set dwell time [µs].</td>
</tr>
<tr>
<td>5</td>
<td>Set line average.</td>
</tr>
<tr>
<td>6</td>
<td>Click and set image contrast and brightness on the microscope.</td>
</tr>
<tr>
<td>7</td>
<td>Click on the button to set capture parameters and Image number or Automatic numbering.</td>
</tr>
<tr>
<td>8</td>
<td>Capture an image.</td>
</tr>
<tr>
<td>9</td>
<td>Image legend parameters can be edited using the icon.</td>
</tr>
<tr>
<td>10</td>
<td>Toggle between two image channels if available.</td>
</tr>
<tr>
<td>11</td>
<td>Use the image chart icon to a) Save image b) Add image to project c) Add image to report.</td>
</tr>
</tbody>
</table>

### Example/hints

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>These workspaces allow capturing an EM image.</td>
<td></td>
</tr>
<tr>
<td>The parameters in steps 3, 4, and 5 affect the total Image capture time.</td>
<td></td>
</tr>
<tr>
<td>Use histogram settings and oscilloscope overlay tools if needed.</td>
<td></td>
</tr>
<tr>
<td>The image will have the name set in the Sample configurator. Choose Single to acquire one image frame, Continuous to update image frames or Sliding average to average a number of frames.</td>
<td></td>
</tr>
<tr>
<td>The image name can also be changed here after capture.</td>
<td></td>
</tr>
<tr>
<td>Note that the image channel names can be changed in the System workspace under Microscope to e.g. SE, BSE, HAADF... (see section 4.7.6).</td>
<td></td>
</tr>
<tr>
<td>Alternatively, click with the right mouse button into the image or drag and drop it to Project or Report. To perform image processing, refer to section 5.6.</td>
<td></td>
</tr>
</tbody>
</table>
Fig. 5.3-1 Image capture
5.4 Image Extension

This section describes the automatic capturing of multiple microscope images using sample stage movement. The images are put together into a mosaic image instantly.

**Step**

1. **Select a workspace** *(Objects, Line scan, Mapping, or Imaging)*.
2. Set image resolution and dwell time.
3. Click \(\text{Preview}\) to adjust image contrast and brightness.
4. Click \(\text{Capture}\) on the \(\text{Capture parameters}\) button to open the \(\text{Capture parameters}\) menu.
5. Set **Image number** or **Automatic numbering**.
6. Select **Activate** in the \(\text{Image extension}\) submenu.
7. Set **Width** and **Height**.
8. Click the extended image.
9. Use the image chart icon to:
   a) **Save** image
   b) **Add** image to project
   c) **Add** image to report.

**Example/hints**

These workspaces allow capturing an EM image.

Refer to section 5.3.

Use histogram settings and oscilloscope overlay tools if needed.

E.g., 2 x 3 means that 2 images in x direction and 3 images in y direction will be captured. The current stage position is in the center of the mosaic image.

Alternatively, click with the right mouse button into the image or drag and drop it to Project or Report. If an analysis of this area is required, follow the corresponding guide (section 5.14, 5.15 or 5.17).
Fig. 5.4-1 Image extension
5.5 Drift Correction

The drift correction tool compensates sample drift when performing long and/or high-resolution acquisitions.

**Step**

1. Select a workspace (Objects, Line scan, Mapping, or Imaging).
   
   Set measurement conditions and before starting the analysis proceed as follows:

2. Use the icon in the bottom left corner of the Scan configurator to open the SCAN CONFIGURATION dialog.

3. Click to open the IMAGE DRIFT CORRECTION SETTINGS dialog.

4a. When performing a Line scan or Objects analysis, select Current input and Correction interval.

4b. When acquiring a Map, the correction time is equal to the Mapping scan time.

5. Click on to change parking position of the beam.

6. Click on to activate drift correction.

7. Start data acquisition by clicking the button in the current workspace.

8. Right click on to display current image shift.

**Example/hints**

These workspaces allow capturing an EM image and beam control.

Refer to the guides in section 5.3, 5.14, 5.15 or 5.17. When acquiring a Map, select a smaller map area than the captured image. Use Fixed or Variable options under Map area (see step 6 in 5.17).

Use Current input and 60 seconds as initial value for Correction interval. If the analysis stops due to extensive drift, decrease this value. Use Current value as Dwell time to capture the same quality image as during the analysis or use a lower Special value than used for Imaging to save time.

If image drift is extensive, reduce Image capture time by decreasing Mapping Dwell time and Line average.

Use a parking position outside of the analysis area to avoid beam induced contamination.

Right click to change parking conditions.

The system starts acquisition and captures an image after each correction interval set in Step 4.

The latest correction image, the latest calculated drift vector (arrow) and the quality of drift correction are displayed. When the drift exceeds the limit which can be corrected by the system, the acquisition stops automatically. Acquired data remains in the workspace.

The starting image records the initial acquired image when the acquisition starts. The image is then compared at intervals set by the user.

The quality plot records the pixel shift in X and Y directions of the image obtained at the latest interval as compared to the original image.
Fig. 5.5-1 Image drift correction
## 5.6 Image Processing

This section describes the workflow for the imaging workspace.

<table>
<thead>
<tr>
<th>Step</th>
<th>Example/hints</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Select the <img src="image" alt="Imaging workspace" /> workspace. Refer to section 5.3. Alternatively, load a saved image by using the workspace icon of the Imaging workspace or drag an image from Project and drop it into the Edit tab in the image chart. Proceed with step 4.</td>
</tr>
<tr>
<td>2</td>
<td>Capture <img src="image" alt="Capture an image" /> an image. The captured image appears in the Edit tab and is ready for processing.</td>
</tr>
<tr>
<td>3</td>
<td>Select the <img src="image" alt="Filter tab and click" /> Filter tab and click <img src="image" alt="Take image" />. Perform filtering and build filter list.</td>
</tr>
<tr>
<td>4</td>
<td><img src="image" alt="Select type of processing from the tab list: Filter" /> Select type of processing from the tab list: Filter, Segmentation, Panorama, Measurements. Perform image masking based on grayscale intensity, Offline compilation of mosaic images, Perform dimension measurements on images using the <img src="image" alt="Draw tool and display results as table" /> tool and display results as table. Drag intermediate images to the clipboard (shown as thumbnails at the bottom of the workspace).</td>
</tr>
<tr>
<td>5</td>
<td><img src="image" alt="Apply filters or perform other operations." /> Apply filters or perform other operations.</td>
</tr>
<tr>
<td>6</td>
<td><img src="image" alt="Use the workspace icon to" /> Use the workspace <img src="image" alt="Save processed image" /> icon to a) Save processed image b) Add processed image to project c) Add processed image to report. Use .rti file format to save all available EM images, processed image, result tables, and drawn items into one file. Alternatively, drag and drop the processed image to Project or Report.</td>
</tr>
<tr>
<td>7</td>
<td><img src="image" alt="Use the image chart icon of the Edit tab to" /> Use the image chart <img src="image" alt="Save processed image" /> icon of the Edit tab to a) Save processed image b) Add processed image to project c) Add processed image to report. Use .bmp, .jpg, .png, or .tif file format. Alternatively, drag and drop the processed image to Project or Report.</td>
</tr>
<tr>
<td>8</td>
<td><img src="image" alt="Use the thumbnail bar icon to" /> Use the thumbnail bar <img src="image" alt="Save processed image" /> icon to a) Save processed image b) Add processed image to project c) Add processed image to report. Alternatively, drag and drop the processed image to Project or Report.</td>
</tr>
</tbody>
</table>
Fig. 5.6-1 Image processing
5.7 Spectrum Acquisition

This section describes the workflow for spectrum acquisition within the Spectra workspace. It is recommended to acquire a preview spectrum before proceeding with analysis in various workspaces.

**Step**

Choose appropriate measurement conditions on the electron microscope.

1. Select the workspace.

2. Click the button to acquire a live spectrum.

3. Click on the button to set timing parameters in the Preview timing submenu.

4. Click on the button:
   a) Set **Acquisition parameters**
      - **Automatic**
      - **Manual**
      - **Real Time**
      - **Live Time**
      - **Counts**
   b) Set **Automatic quantification**
   c) Activate **Cyclic acquisition**
   d) **Spectrum numbering**.

**Examples/hints**

Refer to the previous sections 5.1, 5.2, and 5.3.

The live spectrum appears in the spectrum window. Click the button again to stop acquisition.

Set acquisition time.

- **Fast** (50,000 counts) for major elements,
- **Precise** (250,000 counts) for minor elements,
- **Exhaustive** (1,000,000 counts) for elements close to the detection limit.

If **Manual** is selected, the acquisition has to be stopped manually by clicking the button.

- The acquisition will stop after the time entered in the dialog box has elapsed.
- The acquisition will stop after the dead time corrected acquisition time has elapsed.
- The acquisition will stop after the predefined number of counts is recorded.

If **Continuous/After acquisition** is selected, the spectrum is quantified during/after acquisition. Click to access the method editor (see description in 5.12.1). Load a quantification method using the button.

- This option acquires several spectra with identical settings from the same area. Set Cycle count and Pause [s]. Use Add to project to automatically add acquired spectra to project.

**Automatic numbering** can be activated. The numbering will start with the entered Spectrum number.
5. Acquire a spectrum.

6. Use the workspace icon to
   a) Add data to project
   b) Add data to report

7. Use the spectrum chart icon to
   a) Save spectrum (or result list, element selection, graphic)
      b) Add item to project
      c) Add item to report

*optional, license-based

The acquired spectrum appears in the spectrum chart and the quantification results in the spectrum list (refer to section 5.8). The spectrum can be further processed (element identification, quantification).

Alternatively, drag and drop the spectrum from the spectrum list to Project or Report. The project has to be saved manually (see section 5.22).

Highlight spectra in the spectrum list to save several spectra. Alternatively, right click into the spectrum. Various file formats are possible*. To include all meta data use the .spx format. Refer to section 5.8 and 5.12.1.

Alternatively, drag and drop the spectrum from the spectrum list to Project or Report. The project has to be saved manually (see section 5.22).

*optional, license-based

Fig. 5.7-1 Spectrum acquisition
5.8 Using the Spectrum Chart

This section describes the features of the Spectrum chart, available in the Spectra, Objects, Line scan, and Mapping workspaces. The assigned numbers do not indicate that a certain order needs to be followed.

**Step**

The spectrum list shows:

1a Spectrum type: excitation and/or analysis method

1b Spectrum name

1c Spectrum color

1d Options

1e Factor: scaling factor of y-axis

1f Results.

2 Multiple spectra can be selected (checkbox) or highlighted by clicking on the spectrum line (gray outline).

3 To delete a spectrum press the DEL key on the keyboard or on the spectrum chart.
   
   To scale spectrum diagram or zoom, use either:

4a Mouse scroll wheel to change x-scale

4b Click and hold mouse scroll wheel to move spectrum area

4c Use CTRL key + left mouse click into the spectrum diagram + drag the mouse to scale x- and y-axis

4d Right click on x- or y-axis: scale values can be entered manually

Example/hints

The spectrum name has to be unique in the spectrum list. It can be changed after clicking on the name.

Click on the color bar to toggle between filled and unfilled display. Click on to select the color.

Click to select Pulses, cps, Net counts, energy resolution of peak selected by the cursor (FWHM fit).

Available when Individual scaling is checked in the menu.

Click to select Spectrum information, identification or Display of quantification results (Mass-%, Mass-% (norm.), Atom-%, Stoich.-%, Stoich.-% (norm.).

Checkbox selects spectra for display, highlighted spectra can be processed and exported. To highlight several spectra use SHIFT or CTRL keys and left click on the spectra. When exporting multiple spectrum results, the result table contains methodical error values (Sigma).

Alternatively, drag and drop spectrum from the spectrum list onto the icon.
4e Use the icon for Automatic scaling (spectrum fills display area)

4f Use the icon to zoom in the spectrum window.

5 Use the icon or right click on the spectrum chart and select Properties… to change Spectrum display properties.

6 Click next to the x-axis dimension.

7 Click next to the y-axis dimension.

8 Right click on the x or y scale bar.

9 Double click on a spectrum in the spectrum list to access Spectrum properties (Results, Sample info, Detector, Parameter, and Spectrum).

10 Click the icon to select the desired operator.

11 Click the icon to select or de-select an element.

12 Select the tab in the periodic table.

13 Highlight a region in the spectrum (by dragging the spectrum cursor with the right mouse button pressed) and select a free region (F1-F8).

14 Click the icon on the right side of the spectrum chart and choose the location where the reference spectra are located. Then click .

15 Use the spectrum chart icon to select Add to project in the Element selection section.

Set a rectangle by holding the left mouse key to zoom into the area of interest.

To normalize multiple spectra for comparison, select Individual scaling and Automatic and select an energy region with right mouse button.

Toggle between energy (keV) and channel.

Toggle between cps/eV or channel and pulse/eV or channel.

Adjusting the x and y scaling.

Quantification results, sample info, detector (detector parameters), parameter (Acquisition parameters), spectrum (Energy calibration data) can be here retrieved.

The SPECTRA ARITHMETIC dialog opens. Available from the dropdown list are sum, absolute difference, relative difference, quotient, maximum and minimum. The resulting spectrum will be added to the spectra list.

Any element can be selected or de-selected just by clicking the according symbol.

The Finder option supports the identification of unknown peaks in the spectrum. Place the spectrum cursor over the center of a peak or highlight the peak range by dragging the cursor with pressed right mouse button to display a list of all possible elements. The first element of this list is that one with the highest probability of actually being present in the sample.

Up to 8 spectral regions can be assigned to monitor non-analytical peaks or background levels.

SEARCHING FOR SIMILAR SPECTRA dialog opens. Found spectra will be added to the spectrum list in the dialog. Use the sensitivity slider (cross correlation factor) to optimize search results.

Add the element selection to the project.
Fig. 5.8-1 Features in the spectrum chart
5.9 Sample Coating Correction

This section describes how to use sample coating correction in two ways:
1. Set an element (e.g. carbon) as coating material and omit the net counts originating from it.
2. Measure a reference coating spectrum and subtract this from measured spectra.

<table>
<thead>
<tr>
<th>Step</th>
<th>Example/hints</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Set an element (e.g. carbon) as coating material and omit the net counts of this element in the quantification process:</strong></td>
<td></td>
</tr>
<tr>
<td>1. Click the icon in the bottom left corner of the Sample configurator to open the SAMPLE PROPERTIES dialog.</td>
<td>The <strong>Select element</strong> dialog pops up. The selected element will be deconvolved during the quantification, but not quantified during automatic quantification in subsequently acquired spectra, line scans, and maps.</td>
</tr>
<tr>
<td>2. Select option <strong>Coating with</strong>.</td>
<td></td>
</tr>
<tr>
<td>3. Click on an element.</td>
<td><strong>Make sure that the sample does not contain the same element as the coating material.</strong> <strong>Refer to section 5.12.2.</strong></td>
</tr>
<tr>
<td><strong>Acquire a coating correction file of a coated sample and use this to subtract from all subsequent spectrum acquisitions:</strong></td>
<td></td>
</tr>
<tr>
<td>4. Acquire a spectrum of a coated sample (as coating reference).</td>
<td>The <strong>Save coating correction data</strong> dialog appears. This file contains the net counts from the coating reference sample.</td>
</tr>
<tr>
<td>5. Perform quantification of the spectrum.</td>
<td></td>
</tr>
<tr>
<td>6. Click on an element in the result list.</td>
<td></td>
</tr>
<tr>
<td>7. Click on the icon.</td>
<td></td>
</tr>
<tr>
<td>8. Save the .ccc file.</td>
<td></td>
</tr>
<tr>
<td><strong>Apply the saved .ccc file for coated samples</strong></td>
<td></td>
</tr>
<tr>
<td>9. Click the icon in the bottom left corner of the Sample configurator to open the SAMPLE PROPERTIES dialog.</td>
<td>The <strong>LOAD COATING CORRECTION DATA</strong> dialog opens. Make sure that the coating reference sample and the unknown sample are coated identically.</td>
</tr>
<tr>
<td>10. Select option <strong>Coating correction</strong>.</td>
<td></td>
</tr>
<tr>
<td>11. Load the previously saved .ccc file.</td>
<td></td>
</tr>
</tbody>
</table>
5.9 Sample Coating Correction

Fig. 5.9-1 Sample coating correction – Step 1 to 8

Fig. 5.9-2 Sample coating correction – Step 9 to 11
## 5.10 System Factor Calibration/ Beam Current Setting

For standard-based quantification, a probe current-dependent system factor has to be determined. There are two ways to proceed depending on sample type and whether a picoamperemeter / beam current monitor is available or not. The system factor value is stored in the ESPRIT software and saved along with the following measurements until the software is closed. The quantification of electron transparent samples with the Zeta-factor method requires the knowledge of beam/probe current.

### System Factor Calibration

<table>
<thead>
<tr>
<th>Step</th>
<th>Example/hints</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Move the calibration sample into the analytical position and adjust microscope parameters.</td>
<td>Use a single element calibration sample (Co, Mn, Cu). Select a homogeneous area for spectrum acquisition. Follow section 5.11.1.</td>
</tr>
<tr>
<td>2 Load or create a standards library.</td>
<td></td>
</tr>
<tr>
<td>3 Click the Standards configurator’s calibration icon to open the SYSTEM FACTOR CALIBRATION dialog.</td>
<td></td>
</tr>
<tr>
<td>4 Select the material of the Calibration sample from the Select the sample to use for calibration drop-down list.</td>
<td>After the calibration is finished the message System is now calibrated appears. Do not change microscope parameters afterwards. Spectra that are obtained after this calibration will contain the measured system factor.</td>
</tr>
<tr>
<td>6 Click to calibrate the system factor.</td>
<td></td>
</tr>
<tr>
<td>7 Close the SYSTEM FACTOR CALIBRATION dialog.</td>
<td></td>
</tr>
<tr>
<td>8 Proceed with measurement of standard samples.</td>
<td></td>
</tr>
</tbody>
</table>
5.10 System Factor Calibration/ Beam Current Setting

Use this guide for electron transparent samples and for bulk samples when a picoamperemeter / beam current monitor is available to measure the beam current.

**Step**

1. Measure beam/probe current.

2. Load or create a standards library.

3. Click the Standards configurator’s calibration icon to open the SYSTEM FACTOR CALIBRATION dialog.

4. Enter probe/beam current value manually in the System factor field and press <Enter>.

5. Close the SYSTEM FACTOR CALIBRATION dialog.

**Example/hints**

*Use a Faraday cup or a calibrated electron screen.*

*Follow section 5.11.1 for bulk samples or sections 5.11.2 and 5.11.3 for electron transparent samples.*

*For Zeta quantification of electron transparent samples, the probe current needs to be known and entered in pA.*

*The system factor is now updated with the beam/probe current and measured spectra will contain this information.*
Fig. 5.10-2 Beam current setting
## 5.11 Standards Library

### 5.11.1 Maintain Standards Library for Bulk Samples

This guide describes how to create and maintain a standards library for the standard-based quantification of bulk samples.

<table>
<thead>
<tr>
<th>Step</th>
<th>Example/hints</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Create/open a standards library</strong></td>
<td><strong>See Fig. 5.11-1.</strong></td>
</tr>
<tr>
<td>1 Click the icon in the bottom left corner of the Standards configurator to open the ADMINISTRATION OF STANDARD LIBRARY dialog.</td>
<td>Load a previously saved *.esl file which corresponds to the current detector geometry parameters. Proceed with step 5.</td>
</tr>
<tr>
<td>2 Use the standards library icon to:</td>
<td>The question “Do you want to load a spectrum measured with corresponding system settings?” pops up.</td>
</tr>
<tr>
<td>a) <strong>Load</strong> a standards library or</td>
<td>If you are using the software as Data station (not connected to a microscope). The LOAD SPECTRUM dialog pops up. Load a spectrum .spx which was acquired with the corresponding detector geometry.</td>
</tr>
<tr>
<td>b) <strong>Select New</strong> to create an empty standards library</td>
<td>If you are using the ESPRIT software connected to the microscope.</td>
</tr>
<tr>
<td>i. <strong>Select Yes</strong></td>
<td></td>
</tr>
<tr>
<td>ii. <strong>Select No.</strong></td>
<td></td>
</tr>
</tbody>
</table>

**Update standards library with standard spectra**

<table>
<thead>
<tr>
<th>Step</th>
<th>Example/hints</th>
</tr>
</thead>
<tbody>
<tr>
<td>3 Perform system factor calibration.</td>
<td>Follow section 5.10.</td>
</tr>
<tr>
<td>4 Move the reference sample into analysis position and acquire a reference spectrum (within the Spectra workspace).</td>
<td>Follow section 5.7 to acquire a spectrum. <strong>Important:</strong> Use automatic spectrum numbering or rename spectra after acquisition.</td>
</tr>
<tr>
<td>5 <strong>Quantify</strong> the reference sample with a standardless quantification method (within the Spectra workspace).</td>
<td>Load a standardless quantification method or deselect <strong>Use standards</strong> under Quantification model in the method editor. <strong>Important:</strong> Set the quantification method to (interactive) to access the quantification dialog. Follow section 5.12.1.</td>
</tr>
<tr>
<td>6 Click <strong>Add to standards</strong> in the quantification dialog.</td>
<td>See Fig. 5.11-2. <strong>EDIT STANDARD PROPERTIES</strong> dialog pops up. Confirm the assignment for all chemical elements to be referenced to that standard. If there is an element missing, it needs to be added in the <strong>QUANTIFICATION</strong> dialog under <strong>Settings/Elements</strong>.</td>
</tr>
</tbody>
</table>
7 Add reference concentrations and click.

8 Close quantification dialog with OK.

Repeat step 4 to 8 until all standard samples are processed.

**Edit existing standard values**

9 Click the icon in the bottom left corner of the Standards configurator to open the ADMINISTRATION OF STANDARD LIBRARY dialog.

10 Click on an element to select it.

11 Select the standard spectrum assigned to the selected element.

12 Click to change standard concentrations.

13 Click to re-quantify the standard spectrum.

The assigned spectrum appears in spectrum chart.

The EDIT STANDARD dialog pops up, where the assigned concentrations can be changed.

The QUANTIFICATION dialog pops up, where the re-quantification of standard spectrum can be performed. Follow section 5.12.1.

Fig. 5.11-1 Creating and maintaining standards library
Fig. 5.11-2 Quantification dialog
5.11.2 Maintain Standards Library for Electron Transparent Samples – Cliff-Lorimer Factors

This guide describes how to check and calculate theoretical Cliff-Lorimer factors for standardless quantification of electron transparent samples using the standards library dialog.

### Step 1

1. Click the icon in the bottom left corner of the **Standards configurator** to open the **ADMINISTRATION OF STANDARD LIBRARY** dialog.

### Example/hints

These parameters have to match the parameters of measured data and/or settings of your EDS configuration on the microscope. A spectrum measured with the correct system settings will also contain the respective values. These can be checked in the file or by double click on the text line below the spectrum chart. The geometry parameters of a measurement system can be checked in the workspace by clicking on and should only be changed by Bruker service. If the geometry parameters do not match, proceed with step 3. If they match, proceed with quantification as described in section 5.13.1 or 5.13.2.

### Step 2

2. Check the detector geometry parameters.

### Step 3

3. Use the **standards library** icon to

   a) Load a standards library or

   b) Select **New** to create an empty standards library

   i. Select **Yes**

   ii. Select **No**.

### Step 4

4. Check **NEW STANDARD LIBRARY** geometry parameters, enter a name and click **OK**.

### Step 5

5. Click on **Cliff-Lorimer Factors** to review.

Load a previously saved *.esl file which corresponds to detector geometry parameters. Proceed with step 5.

The question “Do you want to load a spectrum measured with corresponding system settings?” pops up.

If you are using the software as Data station (not connected to a microscope). The **LOAD SPECTRUM** dialog pops up. Load a spectrum *.spx which was acquired with corresponding detector geometry.

If you are using the ESPRIT software connected to the microscope.

Check or update values of Energy, Elevation angle, Azimuth angle, Tilt angle, Beam entrance angle. They should match the values of your microscope/detector configuration (see step 2).

The **EDIT CLIFF-LORIMER FACTORS** dialog pops up: Cliff-Lorimer factors for K-, L- and M-lines are listed. Zoom the graph: left click into it and use the arrow keys on keyboard.
5.11 Standards Library

6* Click on a Cliff-Lorimer value to edit it manually.

7 Use the standards library icon to save the standards library.

* steps can be omitted

It is advised to calculate theoretical factors or set standards as described in section 5.13.2.

Save the standards library (containing Cliff-Lorimer factors) as .esl file.

Fig. 5.11-3 Maintain standards library for Cliff-Lorimer factors
5.11.3 Maintain Standards Library for Electron Transparent Samples – Zeta Factors

This guide describes how to set up the Zeta factor standards library. Quantification of electron transparent samples with the Zeta factor method requires knowledge of the beam current in [pA] for standard and “unknown” sample and sample thickness of the standard sample.

**Experimental Zeta Factors**

Follow this guide to update Zeta factors for the elements contained in the standard sample.

<table>
<thead>
<tr>
<th>Step</th>
<th>Example/hints</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Set up a Cliff-Lorimer standards library.</td>
</tr>
<tr>
<td>2</td>
<td>Select the spectra or the workspace and acquire or open a spectrum of the reference/standard sample. The composition and thickness of the sample has to be known. The beam current during measurement also has to be known and has to be entered as described in 5.13.3 or, if offline, as spectrum parameter: double click on the highlighted line of the active spectrum in the spectrum list (below the spectrum) to open the SPECTRUM PROPERTIES dialog and enter the beam current in pA as system factor in the Parameter tab.</td>
</tr>
<tr>
<td>3</td>
<td>Follow the standardless quantification steps 6 to 13 to identify elements, set background regions and deconvolution.</td>
</tr>
<tr>
<td>4</td>
<td>Select Zeta factor method under Quantification model.</td>
</tr>
<tr>
<td>5</td>
<td>Click Add to standards.</td>
</tr>
<tr>
<td>6</td>
<td>Enter reference element concentration values and sample thickness for standard sample.</td>
</tr>
<tr>
<td>7</td>
<td>Click Add to standard library.</td>
</tr>
</tbody>
</table>

The **EDIT STANDARD PROPERTIES** dialog pops up.

Review Beam current. If missing or incorrect, enter the correct value and also enter the correct value in the spectrum Parameter tab (see step 2). The density will be calculated internally using the sample composition, if changed manually, the thickness will change, respectively.

The **VALIDATION** dialog pops up with new and old Zeta factors. Initial Zeta factors have the same values as theoretical Cliff-Lorimer factors (and are considerably smaller).
5.11 Standards Library

8. Click [OK] to close the VALIDATION dialog.

9. Click [OK] to close the QUANTIFICATION dialog.

The updated Zeta factors for elements contained in the reference sample will be marked red in the Zeta factor list.

NOTE: Save the updated standards library.

If the “unknown” sample contains other elements than the standard sample, proceed with the “Fit Zeta Factors to Standard” procedure below.

Fig. 5.11-4 Maintain standards library for Zeta factors

**Fit Zeta Factors to Standard**

Follow this guide to update Zeta factors for all elements by performing a polynomial fit based on Zeta factors of elements set using standard sample(s).

**Step**

1. Set up Zeta factors for the standard sample.

2. Click the [ ] icon in the bottom left corner of the Standards configurator to open the ADMINISTRATION OF STANDARD LIBRARY dialog.

3. Click [Zeta-Factors].

**Example/hints**

Follow section 5.11.3 “Experimental Zeta factors” and see Fig. 5.11-4.

The EDIT ZETA FACTORS dialog pops up. The list shows Zeta factors for standard sample in red (default initial values are theoretical Cliff-Lorimer factors). To view the graph, left click into it and zoom by using the arrow keys.
4* Click on a Zeta-factor value to edit it manually.

5 Click [Fit to Stds] to perform a polynomial fit based on the already existing standard composition values.

6 Press [OK] to confirm the new Zeta factors.

7 Use the updated Zeta factors for quantification.

* It is advised to proceed with step 5.

A polynomial is fitted on the standard elements Zeta factors. Zeta factors for elements not contained in the standard sample(s) are interpolated.

Follow section 5.13.3.

Fig. 5.11-5 Fitting the Zeta factors to standard
5.12 Quantitative EDS Analysis of Bulk Samples

5.12.1 Using the Method Editor

This section describes how to use the Method editor and how to set up a quantification method for the automatic quantification of multiple spectra. Setting up a quantification method is also important to turn a HyperMap into a QMap (see section 5.18).

<table>
<thead>
<tr>
<th>Step</th>
<th>Example/hints</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Select the Spectra, Objects, Line scan or Mapping workspace.</td>
<td>In the Line scan and Mapping workspaces, spectra are accessible under the Spectrum tab.</td>
</tr>
<tr>
<td>2 Click on the Button and select Load... to load a quantification method.</td>
<td>Select a quantification method (eg. Default, Oxides, Standards or TEM). Alternatively drag and drop a quantification method from the project to the spectrum chart.</td>
</tr>
<tr>
<td>3 Click on the Button and a) Select Edit... b) Double click on the method name next to the button.</td>
<td>This opens the METHOD EDITOR to set up a method before performing the quantification.</td>
</tr>
<tr>
<td>4 Use the Elements menu to a) choose how peaks will be labelled b) access element properties c) change settings for the AutoID (when using the Search additional elements option).</td>
<td>Use spectrum elements will use element markers that you have previously set in the spectrum via the icon. Use list elements will only use the elements that you have selected (bold) in the periodic table within the method editor. Search additional elements will perform an AutoID (see also step 4c). Double click or right click on an element to access the element properties dialog. Use this dialog to set Deconvolution only or to change spectral line for quantification. Set Minimum concentration to an appropriate value to avoid that the AutoID finds too many peaks (especially when spectra are noisy). Elements with a gray background in the periodic table are excluded from the AutoID. To include them click on the specific element. Use this list to change or review element parameters. The background fit area can be set to Automatic or Preset.</td>
</tr>
<tr>
<td>5 Use the Element overview list menu to display non-default settings for selected elements.</td>
<td></td>
</tr>
<tr>
<td>6 Use Background settings menu to select background fit areas and fit model</td>
<td></td>
</tr>
</tbody>
</table>
a) Use [Auto] to generate an automatic background fit

b) Activate [+] and drag with right mouse key in the spectrum diagram to set a background fit area manually.

7 Select the **Deconvolution settings** menu.

8 Select the **Quantification model** menu.

9 Use the **Additional settings** menu for
   a) Setting high voltage correction
   b) Setting sample tilt angle correction
   c) Selecting fast quantification.

10 Click [A] to perform automatic high voltage or sample tilt angle correction.

---

**Fig. 5.12-1 Using the method editor**
5.12.2 Standardless Quantification

This section describes the steps of automatic standardless quantification and quantification using the quantification dialog.

<table>
<thead>
<tr>
<th>Step</th>
<th>Examples/hints</th>
</tr>
</thead>
</table>
| 1    | **Select** the workspace and **Acquire** a spectrum or **Open** a spectrum.  
   a) **Use** the Spectrum chart icon to **Open** a spectrum.  
   b) **Use** the Spectrum chart icon to **Open** a spectrum.  
| 2    | Click on the **Quantify** button and select **Load...** to load a quantification method.  
| 3    | Click on the **Quantify** button and select **Method mode**.  
| 4    | Click on the **Quantify** button and select **Edit...**  
| 5    | Start quantification by clicking the **Quantify** button.  
| 6    | **Edit** quantification parameters using the quantification dialog  
   a) **Use** to drop down the different settings menus  
   b) **Click on** to access/hide the settings of the quantification method.  
| 7    | **Use the Elements tab** to identify elements.  
| 8    | The **Element finder** displays elements with spectral lines at the cursor position in the spectrum diagram.  

Select an element by clicking on the ID in the periodic table. Double click or right click on an element to access the element properties dialog. Use this dialog to set Deconvolution only or to change spectral line for quantification.

Use right click and drag mouse to select a spectral region: all elements are listed in the Finder which have spectral lines in the selected spectrum region.
9 The **Element overview list** displays non-default settings for selected elements.

10 Use **Background settings** to set background fit areas and fit model
   a) Use [Auto] to generate an automatic background fit
   b) Activate [Add] and drag with right mouse key in the spectrum diagram to set a background fit area manually.

11 Select **Deconvolution settings**.

12 Select **Quantification model**.

13 Use **Additional settings** for
   a) High voltage correction
   b) Sample tilt angle correction
   c) Selecting fast quantification.

14 Review spectrum chart.

15 Review quantification result table.

16 Click
   a) [Load] to load a quantification method
   b) [Save] to save current settings as a quantification method
   c) [To project] to add a method with current settings to project
   d) [Apply] to update the active quantification method with the current quantification settings.

17 a) Use the Spectrum chart [Export] icon to export or save spectrum diagram
   b) Use the Results [Copy, Save or Add to Project] icon to Copy, Save or Add to Project or Add to report.

18 Click [OK] to close the **QUANTIFICATION** dialog.

---

**Use this list to change or review element parameters.**

**Deactivate Add button to use spectrum diagram for Element finder (see point 8)**

**Use Series fit as default.**

**Use PB/ZAF as default. Use Phi(Rho,Z) for light elements.**

**Click [ ] to perform automatic high voltage or sample tilt angle correction, if needed.**

**Compare deconvolution results with measured spectrum.**

**Use this to load a previously saved or a default quantification (*.mtdx) file.**

**Use this to save the current settings as quantification method (*.mtdx) file.**

Alternatively, use [ ] on the button.

**Use this to add current settings to project as a quantification method file.**

**Use this to transfer current quantification settings to method editor.**

**Use this to export deconvolution results or background-subtracted spectra.**

The results will be displayed in the spectrum list. The display options of the spectrum diagram are described in section 5.8.
5.12 Quantitative EDS Analysis of Bulk Samples  QUANTAX EDS

Fig. 5.12-2 Automatic standardless quantification
5.12.3 Standard-based Quantification with a Temporary Reference

This guide describes how to perform standard-based quantification without calibration of system factor, measuring reference and unknown samples with same microscope/beam conditions.

**Step**

1. Prepare an “unknown” sample, reference sample(s) and a sample for system factor calibration. Select workspace.
2. Calibrate the system factor.
3. Select the Spectra or Objects workspace.
4. Move the reference sample into analytical position and acquire a spectrum.
5. Click at the button and select Load...
6. Set Method mode to Interactive ( ).
7. Click.
8. Click Add to standards.
9. Enter reference concentrations and click Add as temporary reference.
10. The VALIDATION dialog pops up. Close it with.
11. Click OK to close the QUANTIFICATION dialog.
12. Repeat steps 4-11 for other reference samples.
13. Move the “unknown” sample into the analytical position.

**Example/hints**

“Unknown” sample, reference sample(s) and sample for system factor calibration must be available at the same time in the SEM chamber.

For details see section 5.10. Do not change microscope parameters afterwards.

Follow 5.7 to acquire a spectrum. Use automatic spectrum numbering or rename spectrum after acquisition.

Load a quantification method.

The QUANTIFICATION dialog opens. Set quantification parameters (Element ID, EDS background, Deconvolution) as needed.

**EDIT STANDARD PROPERTIES** dialog opens

**NOTE:** The reference values are TEMPORARILY stored until the ESPRIT software is closed. They will not be added into the standards library. For advanced standard-based quantification options see section 5.12.4.

Do not change any relevant microscope settings (beam current, focus, high voltage...)

If automatic quantification during or after acquisition is enabled (see 5.7), the spectrum is quantified automatically and the results appear in the result list.
5.12 Quantitative EDS Analysis of Bulk Samples

15 Click on the **Quantify** button and select **Load...**

16 Click **Quantify**.

17 Use the **button of the result table to Copy, Save or Add to project or Add to report.**

Load a standard-based quantification method e.g. **Linemaker Standards.mtdx**. Alternatively, open the Method editor and select **Use standards** under **Quantification model**.

...unless automatic quantification during or after acquisition is enabled.

Save the modified quantification method for further use. Use **on the **button or **Save** in the quantification dialog.

Fig. 5.12-3 Standard-based quantification with temporary reference
5.12.4 Standard-based Quantification with Standards Library

Standard-based quantification using a standards library allows the measurement of standard and unknown samples at different times using beam current dependent system factor.

**Step**

1. Click the icon in the bottom left corner of the Standards configurator to open the ADMINISTRATION OF STANDARD LIBRARY dialog and
   - Load a standards library
   - Create a New standards library.

2. Perform system factor calibration.

3. Select the or workspace and
   - Acquire a spectrum
   - Use the Spectrum chart icon to open a spectrum.

4. Click on the button and select Load… to load a quantification method.

5. Click .

6. Use the button of the result table to Copy, Save or Add to Project or Add to report.

**Example/hints**

Follow section 5.11.1 to measure standard samples and update the standards library.

Refer to section 5.10.

This workflow also applies to any spectrum chart available in the ESPRIT software.

Follow section 5.7.

 Optionally, drag and drop a spectrum from the Project into the spectrum workspace.

Select a standard-based quantification method, e.g.: Linemarker Standard.mtdx.

Follow section 5.12.1. Make sure, that Use standards is selected under Quantification model in the method editor panel.

Save the modified quantification method for further use. Use on the button or Save in the quantification dialog.
5.12 Quantitative EDS Analysis of Bulk Samples

Fig. 5.12-4 Standard-based quantification with a standards library
# 5.13 Quantitative EDS of Electron Transparent Samples

## 5.13.1 Standardless Cliff-Lorimer Quantification

This guide describes the steps of standardless quantification of electron transparent samples.

<table>
<thead>
<tr>
<th>Step</th>
<th>Example/hints</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Load or create a standards library containing theoretical Cliff-Lorimer factors. Follow section 5.11.2.</td>
</tr>
<tr>
<td>2</td>
<td>Select the or workspace and a) Acquire a spectrum b) Use the Spectrum chart icon to open a spectrum.</td>
</tr>
<tr>
<td>3</td>
<td>Click on the button and select <strong>Load...</strong> to load a quantification method.</td>
</tr>
<tr>
<td>4</td>
<td>Make sure that geometrical EDS detector and HV parameters of spectrum and Cliff-Lorimer factor database (standards library) match.</td>
</tr>
<tr>
<td>5</td>
<td>Click on the button and select <strong>Method mode</strong>. Interactive method Automatic method.</td>
</tr>
<tr>
<td>6*</td>
<td>Click on the button and select <strong>Edit...</strong></td>
</tr>
<tr>
<td>7</td>
<td>Start quantification by clicking the button.</td>
</tr>
<tr>
<td>8</td>
<td>Edit quantification parameters in the quantification dialog.</td>
</tr>
</tbody>
</table>

Optionally, drag and drop a spectrum from the Project into the spectrum chart. The spectrum chart in the Mapping workspace can also be used. Select a TEM quantification method based on Cliff-Lorimer. Alternatively drag and drop a quantification method from the project to the spectrum chart. Two different quantification methods can be loaded, the active one is displayed in bold.

If the information message “Cliff-Lorimer / Zeta factor: Wrong primary energy in standards library” pops up, update the standards library as described in 5.11.2.

Refer to section 5.12.1.

If quantification is set to automatic ( ), the quantification results appear in the spectrum list (see section 5.8 step 1f).

If quantification is set to interactive ( ), proceed with step 8.

The left dialog part has the same functionality as the method editor. All changes have an immediate effect on the quantification results displayed in the result table. Refer to section 5.12.1.
9 Use the icon of the result table to Copy, Save or Add to Project or Add to report. Save the modified quantification method for further use. Use on the button or the button in the quantification dialog.

Fig. 5.13-1 Standardless Cliff-Lorimer quantification
5.13.2 Standard-based Cliff-Lorimer Quantification

This guide describes the Cliff-Lorimer quantification of electron-transparent samples using standard samples.

**Step**

1. Load or create a standards library containing theoretical Cliff-Lorimer factors.

2. Select the or workspace and
   a) Acquire a spectrum
   b) Use the Spectrum chart icon to open a spectrum.

3. Follow step 3 to 7 of standardless Cliff-Lorimer quantification to identify elements, set background regions and ensure correct deconvolution.

4. Click .

5. Enter reference element concentration values for standard sample.

6. Click .

7. Select an element as reference.

8. Click to close the dialog.

9. Click to close the dialog.


**Example/hints**

*Follow section 5.11.2.*

Acquire or open a spectrum of the reference/standard sample. The sample should contain elements which are present in the “unknown” sample.

Refer to section 5.13.1. Set quantification Method mode to Interactive ( ) to access the dialog.

The dialog pops up.

Use Mass% or Atomic-%.

The dialog pops up with new and old Cliff-Lorimer factors.

The CL-factor of this element will not be changed. Advice: Keep a higher atomic number value as reference.

The modified Cliff-Lorimer factors will be marked red in the Cliff-Lorimer factor list in the standards library (see section 5.11.2).

NOTE: Save the updated standards library.

Follow section 5.13.1. The process is the same as for standardless Cliff-Lorimer factors. The updated standard-based Cliff-Lorimer factors will be used for the quantification.
Fig. 5.13-2 Standard-based Cliff-Lorimer quantification
### 5.13.3 Standard-based Zeta Quantification

This section describes how to perform standard-based quantification of electron transparent samples using beam current and thickness of standard sample as reference values.

<table>
<thead>
<tr>
<th>Step</th>
<th>Example/hints</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Click the <strong>Standards configurator</strong>’s calibration icon ![ICON] to open the <strong>SYSTEM FACTOR CALIBRATION</strong> dialog. Follow section 5.10 for electron transparent samples. Enter the beam current in [pA] units. Follow section 5.11.3.</td>
</tr>
<tr>
<td>2</td>
<td>Measure and enter the beam current value as system factor.</td>
</tr>
<tr>
<td>3</td>
<td>Check or Load the appropriate Zeta factors.</td>
</tr>
</tbody>
</table>
| 4    | Select the **Spectra** or **Objects** workspace and:  
  a) ![Acquire](Icon-Acquire.png) a spectrum  
  b) use the Spectrum chart ![Spectrum Chart](Icon-Spectrum-Chart.png) icon to open a spectrum  
  c) drag and drop a spectrum from project to Spectrum chart. |
| 5    | Click ![Quantify](Icon-Quantify.png) button and **Load...** a quantification method. Enter the beam current as spectrum parameter: double click on the highlighted line of the active spectrum in the spectrum list (below the spectrum) to open the **SPECTRUM PROPERTIES** dialog and enter the beam current in pA as system factor in the **Parameter** tab. Select a TEM quantification method. Alternatively drag and drop a quantification method from the project to spectrum. |
| 6    | Set the quantification method to **interactive ( )**. |
| 7    | Click ![Quantify](Icon-Quantify.png) button to access the **QUANTIFICATION** dialog. See section 5.12.1 to optimize quantification and to save or export quantification method and results. Calculated thickness is shown below the result table. Density is calculated based on element composition. If density is known, it can be entered and sample thickness will change correspondingly. |
| 8    | Set **Quantification model** to Zeta factor method and perform quantification of “unknown” sample. |
| 9    | Click ![OK](Icon-OK.png) to close the **QUANTIFICATION** dialog. |
Fig. 5.13-3 Standard-based Zeta quantification
5.14 Object Analysis

This section describes the steps of user-defined area analysis with image capture using the Objects workspace.

**Step**

1. **Select the workspace**.
2. **Capture an image**.
3. **Select the desired object type**.
4. **Draw an object in the captured image**.
5. **Use** to highlight all objects.
6. **Click at the button to set acquisition parameters**.
7. **Acquire a spectrum**.
8. The acquired spectra will appear in the spectrum list.
9. Use the workspace icon to
   a) **Save object data**
   b) **Add data to project**
   c) **Add data to report**.
10. Use the spectrum chart icon to
    a) **Save spectrum** (or Result list, Element selection, Graphic)
    b) **Add item to project**
    c) **Add item to report**.

**Example/hints**

Refer to section 5.3.

Objects types are point, rectangle, ellipse, and polygon or a grid of points (optional grid, random, both).

The object can be edited (moved or resized) when the icon is activated. Click to delete selected objects.

If automatic quantification is desired, check if automatic analysis. After acquisition or Continuous is selected.

Objects are automatically acquired in sequence. Progress is indicated by the acquisition bar. If Manual is selected, the acquisition has to be stopped manually.

Refer to section 5.8 for further reference.

Use .rto file format. This file type contains all available EM images including objects and corresponding spectra.

Alternatively, drag and drop data to Project or Report.

Alternatively, click with the right mouse button into the image or spectrum.
Fig. 5.14-1 Object analysis
5.15 Line Scan

This section describes the steps of line scan analysis using the Line scan workspace.

<table>
<thead>
<tr>
<th>Step</th>
<th>Example/hints</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Select the workspace.</td>
</tr>
<tr>
<td>2</td>
<td>Capture an image.</td>
</tr>
<tr>
<td>3</td>
<td>Highlight the line and drag and adjust the endpoints to the desired position.</td>
</tr>
<tr>
<td>4</td>
<td>Set <strong>Point count</strong> of the line scan.</td>
</tr>
<tr>
<td>5</td>
<td>Click at the <strong>Acquire</strong> button to set <strong>Scan time</strong>.</td>
</tr>
<tr>
<td>6</td>
<td>Acquire a line scan.</td>
</tr>
<tr>
<td>7</td>
<td>Use the <strong>Elements</strong> icon to identify elements.</td>
</tr>
<tr>
<td>8</td>
<td>Select elements in the thumbnail bar by ticking the boxes below the individual element images to display their profiles in the <strong>Profiles</strong> tab.</td>
</tr>
<tr>
<td>9</td>
<td>Use right mouse key in the scan image to extract region of interest spectrum from the line scan.</td>
</tr>
</tbody>
</table>

10 Use the workspace **icon** to
   a) **Save** line scan
   b) **Add data to project**
   c) **Add data to report**.

Alternatively select Distance [µm] between measurement points.

Choose **Automatic**, **Manual** or **Measurement time [s]**

The line scan is automatically terminated if **Automatic** or **Measurement time [s]** is selected. When **Manual** is selected, acquisition can be stopped manually by clicking the **Acquire** button. One click terminates scan after finishing the last scan (the button changes into **Stop**), a second click terminates the measurement immediately.

Elements are automatically identified by **Auto ID**. Add and delete elements by clicking on the **Element ID** or use the **Finder** in the Spectrum tab. Identified elements are displayed in the line profile thumbnail bar. The element selection can be changed at any time during or after the line scan acquisition.

Extracted spectrum (named as **Range**) appears in the **Spectrum** tab. (optional step). The **Scan** spectrum in the spectrum tab presents the sum spectrum of the line.

Use .rtl file format to save line scan data including EM and scan images and point spectra.

Images(s), composite element profiles and scan image will be transferred. Confirm the pop-up window **Do you want to save point spectra too?** by clicking **Yes** to transfer spectral data.
5.15 Line Scan

11 Use the Profiles tab icon to
   
a) Save data
   
b) Add data to project
   
c) Add data to report.

12 Use the thumbnail bar icon to Add Line scan to report.

13 To quantify a line scan proceed with section 5.16.

Save the composite element profile as .bmp, .jpg, .png, .tif file format including Composite element profile, Scan image or both.

Alternatively, right click into the profile and use the local Line scan menu. Confirm the pop-up window Do you want to save point spectra too? by clicking Yes to transfer spectral data.

Highlight elements in profile thumbnails. Select Individual element profiles to add separate profiles for each element. Select Composite element profile to add all selected elements in one diagram and the scan image.

Fig. 5.15-1 Line scan acquisition
5.16 Quantitative Line Scan (QLine)

This section describes how to quantify a line scan.

<table>
<thead>
<tr>
<th>Step</th>
<th>Example/hints</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Select the workspace.</td>
<td>See section 5.15.</td>
</tr>
<tr>
<td>2. Acquire a line scan or Load a stored line scan file (.rtl) from the workspace menu, or retrieve it from the Project folder.</td>
<td>Use the Scan spectrum of the Line scan to optimize a quantification method. Save this method to disk or add to project and drag and drop from the project into the Profiles tab. See section 5.12.1 for details. For electron transparent samples use a suitable Cliff-Lorimer or Zeta method.</td>
</tr>
<tr>
<td>3. Click on the QLine button and a) Load… an optimized quantification method</td>
<td>To enhance spectra statistics, use values 3, 5, 7, 9, 11, 13 to bin neighboring point spectra.</td>
</tr>
<tr>
<td>4. Click to start the quantification of the line scan database.</td>
<td>Select from: Counts, Net sum, Mass percent, Mass percent (norm.) and Atomic percent (norm.).</td>
</tr>
<tr>
<td>5. Use the icon in the Profiles tab to change profile display options.</td>
<td>When adding the data into the project, or saving the line scan .rtl file format, the point spectra for each point in the line scan can be added to the Project by confirming the pop-up window Do you want to save point spectra too?</td>
</tr>
<tr>
<td>6. Use the workspace icon to a) Save the line scan</td>
<td>Use .rtl file format to save quantified line scan data including scan image and point spectra.</td>
</tr>
<tr>
<td></td>
<td>b) Add data to project</td>
</tr>
<tr>
<td></td>
<td>c) Add data to report.</td>
</tr>
<tr>
<td>7. Use the Profiles tab icon to a) Save data</td>
<td>Save as .bmp, .jpg, .png, .tif file format including Composite element profile, Scan image or both.</td>
</tr>
<tr>
<td></td>
<td>b) Add data to project</td>
</tr>
<tr>
<td></td>
<td>c) Add data to report.</td>
</tr>
</tbody>
</table>
Use the thumbnail bar icon to Add Line scan to report. Highlight elements in profile thumbnails. Select Individual element profiles to add separate profiles for each element. Select Composite element profile to show all selected elements in one diagram.

Fig. 5.16-1 Quantitative line scan
5.17 Map Acquisition

This section describes the acquisition of maps using the Mapping workspace. Maps can be saved as element distribution images or datacube (HyperMap data)*.

**Step**

1 Select the [Mapping] workspace.
2 Click [Image resolution, Mapping dwell time and Line average].
3 Click [Capture] and adjust image.
4 Click [Capture parameters] menu.
5 An image.
6 Click [Acquire] button and set parameters for Map time, After measurement and Map area.
7 [Acquire] a map.
8 Use the workspace icon to
   a) **Save** Map data
   b) **Add data to project**
   c) **Add data to report**
9 Use the chart icon of Map tab to
   a) **Save** map image (Element selection, Settings, Result table)
   b) **Add item to project**
   c) **Add item to report**

**Example/hints**

Use high count rate for good count statistics.

Image resolution defines the pixel resolution.

Adjust brightness, contrast and magnification on the microscope.

Adjust Capture parameters for the image: set single or continuous (see section 5.3 for further details).

Mapping is automatically terminated if Measurement time [s] or Cycles is set. When Manual is selected acquisition has to be stopped manually by clicking the button. One click terminates scan after finishing the last frame, a second click terminates the measurement immediately.

The processing of map data is described in section 5.19.

Use file format*:

.bc:\ to save HyperMaps (datacube, hyperspectral data set). Spectra for each pixel are saved. Further processing is possible only, when data is saved as a .bc file.

.rtm: To save element distribution images without point spectra.

.raw: To save hyperspectral datacube for further processing with third party softwares (NIST Lispix format).

Note: Only the EM image, composite element images and Map spectrum will be added to a Project and Report, not the whole hyperspectral database (HyperMap).

Use image file formats (.bmp, .png, .jpg, .tif) to save the composite element image. Alternatively, click with the right mouse button into the map.

Composite element image will be added to Project or Report.
10 Use the thumbnail bar icon to
   a) **Save Images**

   b) **Add images to project**

   c) **Add images to report**.

11 Process Map data as desired.

*optional, license-based*

---

**Fig. 5.17-1 Map acquisition**
5.18 Quantitative Mapping (QMap)

This section describes the quantification of mappings.

<table>
<thead>
<tr>
<th>Step</th>
<th>Example/hints</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Select the <strong>workspace</strong>.</td>
<td>Good spectrum statistics may improve the quantification accuracy.</td>
</tr>
<tr>
<td>2 a) <strong>Acquire</strong> a map or b) open a .bcf file.</td>
<td>Use a previously optimized method (presetting element IDs, background fit regions and quantification algorithm).</td>
</tr>
<tr>
<td>3 Click <strong>Load</strong>... an appropriate quantification method.</td>
<td>Use tile size of 3x3, 5x5 or 9x9 pixels to reduce processing time and enhance count statistics. The <em>Estimated time</em> to complete the QMap will be displayed.</td>
</tr>
<tr>
<td>4 Set <strong>QMap options</strong>.</td>
<td>QMap will terminate automatically after the last pixel has been quantified.</td>
</tr>
<tr>
<td>5 Click to start the quantification of the map.</td>
<td>See <strong>Map Display settings</strong> and refer to section 5.19.</td>
</tr>
<tr>
<td>6 Adjust colors and image controls.</td>
<td>Quantification results appear in the Map result list.</td>
</tr>
<tr>
<td>7 Use the <strong>Tool</strong> to extract quantification results of regions of interest.</td>
<td>The pseudo colors display presents numerical quantification values. Choose one element only for pseudo color display. If several elements are selected, the map displays summarized concentration values.</td>
</tr>
<tr>
<td>8 Click on to switch to pseudo colors display.</td>
<td>Select Net sum, Mass %, normalized Mass % or Atomic %. Select Counts to display non-quantified intensity maps.</td>
</tr>
<tr>
<td>9 Set <strong>Result type</strong> in Map display settings submenu.</td>
<td>Save the quantified datacube (Hypermap) as .bcf file. Alternatively, drag and drop composite element image to project or right click into the map and use the local Mapping menu.</td>
</tr>
<tr>
<td>10 Use the workspace <strong>icon</strong> to a) <strong>Save</strong> Map data b) <strong>Add</strong> data to project c) <strong>Add</strong> data to report.</td>
<td>Alternatively, click with the right mouse button into the map image.</td>
</tr>
<tr>
<td>11 Use the chart <strong>icon</strong> in <strong>Map</strong> tab to a) <strong>Save</strong> Map image (Element selection, Settings, Result table) b) <strong>Add</strong> item to project c) <strong>Add</strong> item to report.</td>
<td></td>
</tr>
</tbody>
</table>
12 Use the thumbnail bar icon to

a) Save Images

b) Add images to project

Individual element images of selected thumbnails will be saved.

Composite and individual element images will be added to the Project.

Individual element images will be added to the Report. Alternatively, click with the right mouse button into the thumbnail bar.

c) Add images to report.

Fig. 5.18-1 Quantitative mapping
5.19 Map Processing

This section describes the online and offline processing of HyperMap data (datacube/.bcf files*).

**Step** | **Example/hints**
--- | ---
1. Select the workspace. | Select elements by clicking on the Auto ID or use the Finder in the Spectrum tab. Identified elements are displayed in the map thumbnail bar.
2. Use the icon to open the periodic table and identify elements. | Optionally use or on the right of the thumbnail bar.
3. Select/Deselect element(s) in the thumbnail bar by ticking/unticking the boxes below the individual element images to display their distribution in the large/mixed map image. | Alternatively, click with the left mouse button into the Map display bar.
4. Click to open the Map display settings submenu and adjust | Applies filters on the overlay EM image.
   a) Image filter | Applies filters on the element distribution map.
   b) Map filter | Use Online deconvolution to display deconvolved elements distribution maps.
   c) Result types | Use sliders to apply image brightness, gamma correction and color saturation.
   d) Color control | Use Enhanced to improve the display of EM image and element map overlay.
   e) Map color mixing. | Choose one element only for pseudo color display. Right click on the icon to adjust Palette settings. Note: If the map is not quantified, the values are normalized to the highest displayed intensity value.
5. Click on to switch to pseudo colors display. | View and quantify the extracted point/area spectra in the Spectrum tab.
6. Extract point/area spectra from the map: | Select point, line, rectangle, ellipse or polygon.
   i. Right click on to access object tool list | Object spectra appear in Spectrum tab.
   ii. Select the preferred tool | Line profile appears in the Line scan tab. Enhance line profile statistics: use + and – keys to set line width. Line scan can be transferred to the Line Scan workspace via the Project. To quantify a map or a line scan, see section 5.18 or 5.16.
   iii. Click on to activate the object tool | Identification of locally enriched elements with low total concentration. The MaximumPixelSpectrum is displayed in the Spectrum tab.
   iv. Move the cursor to the area of interest, press left mouse key and drag cursor to expand object.
7. Click on to generate a MaximumPixelSpectrum
5.19 Map Processing

8 Select the Chart tab to see element correlations or element histogram.

9 Use the workspace icon to
   a) Save Map data

b) Add data to project
c) Add data to report.

10 a) Use the chart icon in the Map tab to Save the map image (Element selection, Settings, Result table)

b) Use the chart icon in the Charts tab to Save the charts image

c) Use the chart icon in the Spectrum tab to Save the spectrum.

11 Use the thumbnail bar icon to
   a) Save images

b) Add images to project
c) Add images to report.

*optional, licence-based

Choose Color settings, Mode and displayed Elements in the Chart settings below the Chart diagram. Make sure to have good count statistics in the map.

Use file format*:

.bcf: to save HyperMaps (datacube, hyperspectral data set). Spectra for each pixel are saved. Later offline (re-) processing is only possible when data is saved as a .bcf file.

.rtm: To save element distribution images without point spectra.

.raw: To save hyperspectral datacube for further processing with third party softwares (NIST Lispix format).

Note: Only the EM image, composite element images and Map spectrum will be added to a Project and Report, not the whole hyperspectral database (HyperMap).

Use image file formats (.bmp, .png, .jpg, .tif) to save composite element image. Alternatively, click with the right mouse button into the map. The options Add to project and Add to report are also available.

The options Add to project and Add to report are also available.

Alternatively, click with the right mouse button into the thumbnail bar. The options Add to project and Add to report are also available.

Individual element images of selected thumbnails will be saved. To select them mark the individual element images with left mouse-click + SHIFT or CRTL key.

Composite and individual element images will be added to Project.

Individual element images will be added to Report. Alternatively, click with the right mouse button into the thumbnail bar.
Fig. 5.19-1 Map processing
5.20 Phase Analysis

The AutoPhase tool calculates images that reveal the local distribution of different phases in a sample.

**Step**

1. Select the [Mapping] workspace and acquire a map or open a bcf. or rtm. file.

2. Select the [Map] tab and the elements of interest for phase calculation in the thumbnail bar.


4. Use the [Calculate phases] button to open the corresponding menu and activate [Automatic update] option.

5. Click [Calculate phases] button.

6. Use [AutoPhase settings] sub-menu to open it.

7. Select the AutoPhase [Method]

   i. **PCA**

   PCA (Principal Component Analysis) is a specific type of vector analysis reducing the dataset to the most relevant chemical information. It starts its operation with an n-dimensional “element” space. The intensity of every element selected in the map thumbnail bar (below the left-hand side Map tab) spans one axis of this space. Map pixels that contain the same chemical information form data clouds which are separated by the PCA dimension reduction algorithm.

   The **Clusters** method compares the composition of neighboring pixels (by running through the map pixel by pixel).

   The phases are user-defined by manually defining objects using drawing tools accessible through the [objects] icon.

   Use the sliders to change the values.

   ii. **Clusters**

   iii. **Objects**

8. Select and adjust the **Options** for the phase calculation.
i. **Sensitivity**

Represents how similar (high sensitivity) or different (low sensitivity) the chemical compositions of the distinguished phases are.

ii. **Area**

Represents the minimum size of a phase, ranging from 0 to 1 area % of the map image.

iii. **Edges.**

Represents the sensitivity used to connect small phases.

Choose area %, area pixels and area µm/mm. You can rename P1, P2, … by selecting a phase from the list and clicking on its name. Rename and press <ENTER>.

Optionally use [all] or [none] on the right of the thumbnail bar.

9 Phase results will appear in the Phase result list.

10 Select or deselect the phases in the thumbnail bar.

11 Click on a phase thumbnail to highlight it and

a) Merge phases using the icon

b) Extract a spectrum of a highlighted phase using the icon.

12 Use the workspace icon to

a) **Save Map data**

b) **Add data to project**

c) **Add data to report**.

13 Use the Phases tab icon to

a) **Save Phase data**

b) **Add Phase data to project**

c) **Add Phase data to report**.

14 Use the thumbnail bar icon to

a) **Save Phase images**

b) **Add Phase images to project**

c) **Add Phase images to report**.

Several phases can be merged into a single phase by highlighting the particular phases in the clipboard (press CTRL or SHIFT key to highlight multiple thumbnails) and pressing the icon.

The spectrum will be displayed in the Spectrum tab.

Save datacube (Hypermap) as .bcf file or map data as .rtm file.

Alternatively, drag and drop data to the Project or Report.

Alternatively, right click into composite image and use the Phases menu.

Individual phase images of selected thumbnails will be saved.

Composite and individual phase images will be added to Project.

Individual phase images will be added to Report.
Fig. 5.20-1 Performing phase analysis
5.21 Creating a Report

This section describes how to create a report based on the data obtained or processed in all workspaces of QUANTAX ESPRIT.

**Step**

1. Select a workspace and perform an analysis or process data.

**Example/hints**

Reports can also be composed from files or project entries.

**Add data to report:**

2a. Use the workspace icon and select **Add to report**

2b. Use the chart icon and select **Add to report**

2c. Use the thumbnail bar icon and select **Add to report**.

3. Use the icon in the bottom left corner of the report configurator to open the **Report**.

4. Edit/ review the report
   a) Use **Zoom** to set display
   b) Use **Properties** to set object layer order
   c) Use **Objects** to add text
   d) Use **Tools** for drawing.

   e) Use the icon in the **Report** editor to import data.

5. Rearrange order of items.

6. Use the **Report** icon to **Save as report**.

7. Use the **Report** icon to **Export to WORD**.

8. Click the **Print** button to print the report.

**Example/hints**

All results contained in the workspace will be added to the report.

Alternatively, right click into the chart and use the local menu.

Available in the **Mapping** or **Line scan** workspace. Alternatively, a right mouse click into the thumbnail bar opens the corresponding local menu.

The **Report preview/Report** is either docked to the right side of the screen or to the configurator bar. The display can be changed using the icon in the **Project** tool.

Objects are displayed as layers. Uncheck **Fixed** when moving an object. Check **Pass on** to show object on every page.

**Draw Point, Arrow, Line, Rectangle, Circle/Ellipse.** Set fill/color/size options for selected element.

Open image, spectrum or text.

Drag objects (maps, images, spectra…) and move them to the desired position.

Alternatively, use the **Report Preview** icon. The report is saved as .rpt.

If Microsoft Word © is available.

Check page **Setup** before printing. Use printer or generate a .pdf file.
5.21 Creating a Report

Fig. 5.21-1 Creating a report
5.22 Managing Projects

This section describes how to use Project to manage and archive data obtained or processed by ESPRIT. Use Project as a data clipboard to transfer data between the workspaces or save all data added to Project as one file to disk. Add images, spectra, element selection, quantification methods, result tables, map/phase images, line scan data (including point spectra).

**Step**  

1. Use the Project icon to  
   a) Generate a New Project  
   b) Open…a Project file from disk.

2. Select a workspace and perform analysis or process data.

**Add data to project:**

3a. Use the workspace icon and select Add to project

3b. Use the chart icons and select Add to project

3c. Use the thumbnail bar icon and select Add to project

3d. Drag and drop an item from the current workspace/chart to the Project

3e. Use the Project icon to Import an item from the hard disk.

**Retrieve an item from Project to a workspace:**

4. Drag and drop an item from Project to the current workspace/chart.

**Save project to hard disk:**

5. Use the Project icon to Save the project to hard disk.

**Example/hints**

- An empty project list is generated.
- All results contained in the workspace will be added to the project.
- Alternatively, right click into the image chart or result chart and use the local menu.
- Available in the Mapping workspace.
- The item appears in the Project list.
- Use the icon to toggle the position of Project. It is either docked to the right side of the screen or to the configurator bar. Double click an item in the project to review.
- Use this function as a data clipboard for data transfer between workspaces of ESPRIT.
- The project is saved as .rtx file. Use Save to overwrite the current project and use Save as... to add a new name.
- Note: (mod.) is displayed after the project name if an opened project file has been modified and the current changes are not saved.
Please note:
Items in the project are not automatically saved to the hard disk! Save the project file manually to archive your data.
Do not use Project to store HyperMap data. The current map display can be added to the project, but not the HyperMap data (point spectra). HyperMaps should be saved as described in 5.17.

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