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Operating Manual

ASpect PQ

Software for ICP-OES

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Contents

1 Software ASpect PQ

ASpect PQ is the control and analysis software for following ICP-OES from Analytik Jena:

- PlasmaQuant PQ 9000
- PlasmaQuant PQ 9100

The method parameters for the measurement procedures can be optimized to the specific demands of the sample to be analyzed. The obtained data can be recalculated, exported to various file formats and printed out.

Described software version

This manual help is based on the version ASpect PQ 1.3.

Intended use

ASpect PQ software exclusively serves to control the ICP-OES devices of Analytik Jena and to analyze the data obtained with these devices.

The manufacturer does not assume any liability for problems or damage caused by the non-intended use of ASpect PQ.

ASpect PQ and the device to be controlled by it may only be operated by appropriately qualified and instructed personnel. The user must be familiar with the information given herein and in the user's manual of the device.

1.1 Starting and exiting ASpect PQ

1.1.1 Starting ASpect PQ

▶ Switch on the ICP-OES device and the autosampler.

▶ Click on the ASpect PQ icon on the Windows desktop.

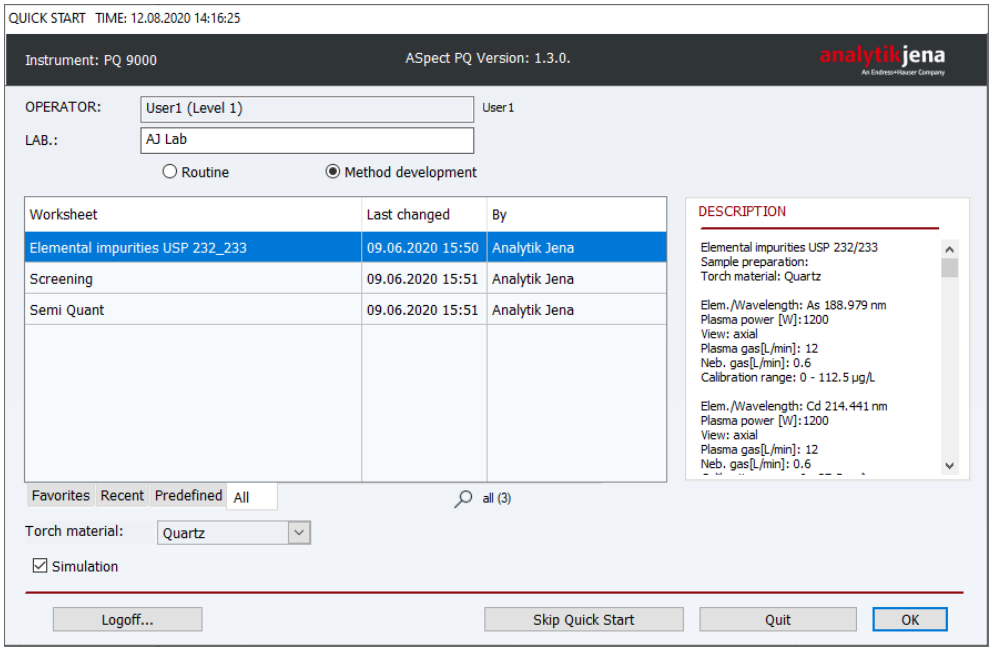
ASpect PQ is started.

▶ If the User Management has been installed, you will be prompted to enter username and password. The ASpect PQ program will only be accessible after successful entry of these data.

After the software starts, the Quickstart (→"Software ASpect PQ" p. 7) opens. Here you have the option of selecting worksheets with preset methods and sequences (→ "Starting with Worksheet" p. 9) or switching directly to the ASpect LS user interface (→ "Starting without worksheet" p. 9).

1.1.1.1 The Quickstart window

After starting the software and logging on a user (only if user management is installed), the **Quickstart** window appears. From here you can load a worksheet or switch to ASpect PQ without any further settings. You can also open the Quickstart window in ASpect PQ with the menu command **File | Quickstart**.



The Quickstart window

Settings in the Quickstart window

The following options and buttons are available in the **Quickstart** window:

Option / Button	Description
Operator	When using the optionally installable user management, the logged-in user is displayed. If the user management is not used, a user can be entered manually here.
Lab	Up to 30 characters can be entered. The last entry is saved and displayed as information in the result logs.
Routine	Start program for routine operation In routine operation, only methods that are enabled for routine operation are displayed.
Method development	Start program completely All settings in method development are activated.
Torch material	Select the used torch material (quartz or ceramic) to adjust the sensitivity of the optical plasma sensor.
Simulation	For training and demonstration purposes, it is possible to operate ASpect PQ without a connected analyzer. When activated, all instrument functions (including measurement acquisition and evaluation) are processed in simulation mode.
[Skip Quick Start]	Switch to the ASpect PQ user interface without selecting a worksheet.
[Quit]	Close Quickstart window and exit ASpect LS.
[OK]	After selecting a worksheet, switch to the ASpect LS interface.

Worksheet table

The worksheet table displays the currently available worksheets. The 4 tabs make it easy to find a worksheet:

Tab	Content
Favorite	Worksheets with the Favorite marker
Recent	Last used worksheets
Predefined	Predefined worksheets from Analytik Jena, which are installed with the installation of ASpect PQ
All	All worksheets
🔍	Use the loupe symbol to filter the worksheets by elements. After a click on the symbol an element list is displayed where you can select an element. You can repeat the selection if you want to search for more elements. If you have selected several elements, all worksheets are displayed that contain at least one of the elements in the deposited method (OR logic).

1.1.1.2 Starting with Worksheet

A worksheet is a folder containing a method and a sequence. Optionally, worksheets can also contain settings for the sample ID and for saving the results file. You can start a measurement immediately with a selected worksheet. If several versions of the method and sequence exist, the newest (current) versions are always used for the measurement.

Install the accessories on the analyzer and then switch on the accessories and the analyzer.

- ▶ Start the software.
  - ✓ The Quickstart appears.
- ▶ Make the necessary entries in the **User** and **Laboratory** fields.
- ▶ Select the torch material.
- ▶ Select the required worksheet in the worksheet table.
- ▶ Click **[OK]**.
  - ✓ The ASpect PQ workspace appears. The method and sequence are already loaded.

Depending on the worksheet configuration, you can now link the method and sequence loaded with the worksheet to a sample ID file or start the measurement directly.

1.1.1.3 Starting without worksheet

Without a prepared worksheet you must load or reconfigure the method, sequence and sample ID for the measurement.

- ▶ Install the accessories on the analyzer and then switch on the accessories and the analyzer.
- ▶ Start the software.
  - ✓ The Quickstart appears.
- ▶ Make the necessary entries in the **User** and **Laboratory** fields.

- Select the **Torch material**.
- Click **[Skip Quick Start]**.

The ASpect PQ workspace appears.

General measurement procedure

The following actions are necessary for a manual or an automatic measurement procedure:

- **Specify the Method parameters** (method development).
- **Create a sequence**. The sequence specifies samples and actions in the intended order of execution. Some sample describing data, such as the name of the sample and its position on the sample tray may also be entered directly and are saved with the sequence.
- For routine analysis it is useful to create a **sample information file** (sample ID). This file contains sample-related data such as sample name, dilution factor and sample tray positions. These data are needed if the concentration is to be back-calculated to the original sample. Sample information files are text files; therefore, they can also be created with external applications.
- **Start** measurement.

The results are instantly written to the result database during the measurement. This central results file is accessed by the integrated data management functions (export, print ...).

After the start of the measurement the result data are entered in the results list. Detailed result presentation (individual values, spectra ...) is accessible by selection of the corresponding sample row. The results obtained last are always appended to the end of the table; overwriting of results is not possible.

Further data analysis is possible by the Reprocessing function. Measured data can be prepared for printing the report or exported.

1.1.2 Opening a second instance of ASpect PQ

If the application is already running, another program instance of this application will be opened in offline mode. In this mode, there is no communication with the device. However, all other functions such as developing methods or loading and analyzing results can be used parallel to the running measurements in the first program instance.

- Start the program in the second instance using the menu item F**ile | Start Offline-Program Instance**.

1.1.3 Locking ASpect PQ

The application can be locked for operation whilst measurements continue to be performed when it is locked. In combination with the optionally available user administration a password confirmation is required to unlock the screen.

- Select the menu item **Extras** | **Lock**.
- To unlock the application click on the padlock icon on the screen.

1.1.4 Exiting ASpect PQ

- ▶ Extinguish the plasma (→ "Igniting plasma/extinguishing plasma" p. 64).
- ▶ Exit the program by selecting the menu item **File / Exit**.
- ▶ If, at this time, method, sequence or sample information data files are open that have not been saved yet, you will be informed accordingly. If you want to save these files, click **[Yes]**.
- ▶ After deactivation of the plasma the ICP-OES device still requires some time for system cooling. If the target temperature has not yet been reached, a progress window is displayed with a message about safe deactivation of the ICP-OES device. Only switch off the ICP-OES device after ASpect PQ has terminated.

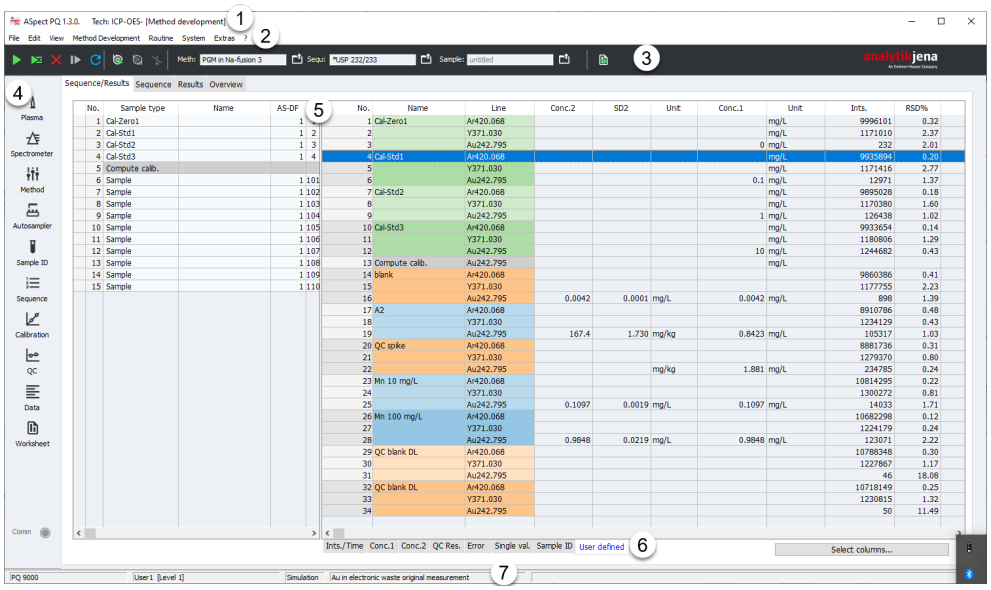
Note

If ASpect PQ is terminated whilst the plasma is burning, the plasma is automatically extinguished after a query!

1.2 General information on operation

1.2.1 The workspace

After starting the ASpect PQ program the window **Main settings** is first opened. After selecting a task you get to the workspace.



ASpect PQ workspace

Main components of the workspace

No..	Beschreibung
1	In the **title bar** you will find information about the software version, the connected device, the technology and (if loaded) the worksheet.
2	Via the **menu bar** you can access all program functions of the software.

3	The **toolbar** contains the buttons for starting and pausing measurement sequences, and displays the currently loaded method, sequence and sample ID file. Click on the button  behind the fields to load the data set. You will also find the button for creating a new worksheet here.
4	Via the **toolbar** you have access to the most important windows (functions) of the software. As soon as one of the windows is open, the corresponding icon turns red. If several windows are open, click the icon again to bring a window to the foreground.
5	In the **main window** ,the sequence and the measurement results are displayed (→ "Displaying results and analysis progress in the main window" p. 72).
6	Some main cards have further **sub tabs**, which are arranged in the lower part of the window.
7	The **status bar** at the bottom of the window displays information about the connected instrument, the user logged in and the name of the currently displayed results database.

1.2.2 The Help function

Help on the operation of ASpect PQ is available via the menu item **? / Help topics**. While working with Aspect PQ windows you can activate context-sensitive help by pressing the function key **[F1]**.

The program pops up brief information (tool tips) on buttons of toolbar and icon bar and other buttons as well as on the table headers in windows **Method**, **Sequence** and **Sample ID** while you move the mouse pointer across the button.

1.2.3 Overview of menu bar, toolbar and icon bar

The menu bar is arranged at the top edge of the Aspect PQ workspace. It allows all operating actions of software to be started. Menus and buttons not accessible for the current contents of the workspace appear grayed out. Some menu items, such as the print function, are displayed dependent on other windows being open.

Functions in the menu bar

Menu item	Description
File	■ Create, open and save methods, sequences and sample information data ■ Open results data ■ Delete methods and sequences ■ Export spectrum data ■ Start offline or online program instances ■ Print active window or protocol ■ Open report design mode ■ Open the window **QUICKSTART** ■ Exit the application ■ Directly open the last opened methods and sequences.
Edit	■ Copy and insert content of text and input fields ■ Copy selected rows of the results list to the clipboard ■ Delete the content of the results list

View	■ Open and closing windows showing graphs and information during the analysis process e.g. signal curves ■ Select the scale of the signal axis for graphs
Method Development	■ Activate windows required for method development ■ Record overview spectrum
Routine	■ Start, stop and break measurement ■ Reprocess measurement data ■ Extinguish plasma ■ Wash system
Extras	■ Open the windows **Data** and **Options** ■ Start a search for individual samples ■ Show lines table ■ Print the current screen image ■ Perform maintenance of the cooling mobile ■ Lock workplace
System	■ Available when the optional "21 CFR Part 11 Com-pliance ASpect PQ" module is installed ■ Configure user administration ■ Change password ■ View Audit Trail ■ Sign results
?	Online help and information on software version.

Toolbar

The buttons in the toolbar are mainly used to start/pause and continue the (sample) sequence measurement. In the toolbar fields the currently loaded methods, sequences and sample IDs are displayed.

Tools	Description
	Start sequence measurement.
	Measure a highlighted row in the sequence.
	Pause running sequence measurement.
	Continue paused sequence measurement.
	Recalculate results, e.g. after measuring an additional sample.
	Start/stop the pump at the ICP-OES device.
	Speed up the pump (washing the sample path).
	Extinguish the plasma.
	Open files. Saved methods, sequences or sample IDs can be loaded to the program and used for the current analysis.

	Create a new worksheet.

Icon bar

The icon bar provides quick access to the key functions of the program ASpect PQ. Clicking on the icon opens the window with the corresponding program function. After installation has completed the icon bar is located near the left screen margin. However, it can be moved anywhere.

Icon	Description
	Check atomization: ■ Igniting/extinguishing the plasma ■ Setting the gas flows ■ Check the pump for the sample conveyance to the vaporizer ■ Adjustment of the transfer optics
	Check spectrometer functions: ■ Device data ■ Test of wavelength corrections ■ Start a measurement on a test wavelength ■ Start a continuous measurement for device optimizations ■ Optimization of plasma power and nebulizer gas
	Create and display method parameters
	Set autosampler parameters and check operation.
	Enter and display sample data.
	Set measuring frequency.
	Display and edit calibration function.
	Configure and view quality control.
	■ Data administration ■ Print results and manage protocol templates ■ Selection of measuring units ■ Database for stock standards and QC samples
	Manage the worksheets Open a worksheet

1.2.4 Frequently used control elements

Various button, mouse and keyboard functions are used in Aspect PQ, which always have the same or very similar meanings.

These control elements are described here in general. Specific information is given, where necessary, in the description of the respective windows.

General Buttons

The function of icon buttons is indicated by means of tool tips displayed when the mouse pointer rests on the corresponding button.

Button	Description
[OK]	Closes the window and accepts the settings.
[Cancel]	Closes the window rejecting possibly changed settings.
[Accept]	Accepts the settings without closing the window.
[Close]	Closes the window; settings are not saved permanently.
[Open]	Opens a selection window for loading a file or data record.
[Save]	Opens a selection window for saving a file or data record.
	Opens a selection dialog box, e.g. for file path selection.
	Opens the **Print** window. From this window, you can print out the contents of the active document window or export it to a file.

Tables

In some of the windows, values are entered directly into a table. Dependent on the type of entry, the table cell behaves like an input field, a selection list, or a spin box for a restricted numerical value range with arrow keys.

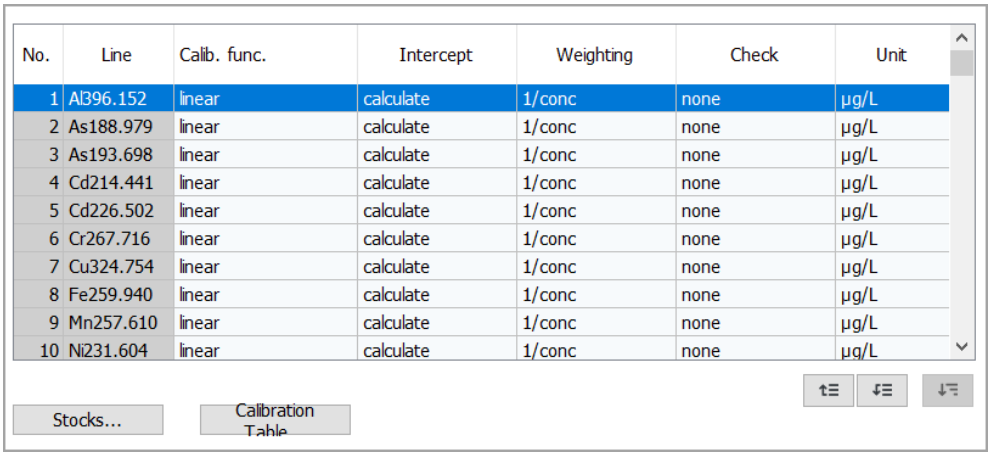
- To select a row of a table, click on the corresponding row in the first table column highlighted by a gray background. Afterwards, you can move the line cursor with the **[↑]** and **[↓]** buttons.
- To change the width of a column move the mouse pointer to the corresponding border line in the column head until it turns into a double-headed arrow. Keeping the left mouse button depressed, you can then drag the border line to adjust the desired width.

In input fields, the following functions are additionally available:

- [F2] activates the edit mode. In this mode, the **[←]** and **[→]** keys are used for editing character by character. Renewed pressing of **[F2]** reactivates the standard mode where the cursor keys are used to navigate between the cells.
- Text can be copied to the Windows clipboard and re-inserted via the menu **Edit | Copy** and **Edit | Insert** or the key combinations [Ctrl+C] and [Ctrl+V].

Buttons accessible in tables

Button	Function
[Append]	Appends a new table line to the end of the list.
[Insert]	Inserts a new table line before the selected line.
[Delete]	Deletes the selected table line. Several table rows can be selected by holding down the Ctrl or Shift key and clicking with the mouse.
	Shifts up the selected table line by one position. **Note**: A table line must be completely selected before it can be moved. To do this, click on the number of the line in the first column of the table.
	Shifts down the selected table line by one position.
	Transfers the value of the active cell to all following table lines of the same sample type (samples, standards, QC etc.). With the checkbox **inc. enabled** (inc. means increment) this value will be incremented automatically, e.g. Sample001, Sample002 ...



No.	Line	Calib. func.	Intercept	Weighting	Check	Unit
1	Al396.152	linear	calculate	1/conc	none	µg/L
2	As188.979	linear	calculate	1/conc	none	µg/L
3	As193.698	linear	calculate	1/conc	none	µg/L
4	Cd214.441	linear	calculate	1/conc	none	µg/L
5	Cd226.502	linear	calculate	1/conc	none	µg/L
6	Cr267.716	linear	calculate	1/conc	none	µg/L
7	Cu324.754	linear	calculate	1/conc	none	µg/L
8	Fe259.940	linear	calculate	1/conc	none	µg/L
9	Mn257.610	linear	calculate	1/conc	none	µg/L
10	Ni231.604	linear	calculate	1/conc	none	µg/L

Example of a table

Graphs

In graphs, you can open a context menu by clicking the right mouse button. This menu provides options for copying either the graph or the entire window to the Windows clipboard.

In several graphic windows, additional icon buttons are accessible:

Icon	Function
	Activates the zoom mode. With the left mouse button pressed you can select an area of the graph to be zoomed in.
	Deactivates the zoom mode and resets the graph to the original scale.
	Activates the text mode. With the left mouse button pressed it is possible to select an area for a window for adding text to a graph. A double click on existing text opens the window for modifying or deleting the text. With a combination of Ctrl key and right mouse button existing text can be moved.
	Activates the selection mode in signal or spectral plots. Clicking the left mouse button adds labels to the measuring points; pressing Shift+right mouse button deletes all labels.

Function keys

Icon	Function
[F1]	Activate the context-sensitive help.
[F2]	Edit table cells.
[F5]	Start printing a screen image.
[F6]	Measure the selected row of the sequence (Menu item **Routine / Run selected sequence row**).
[F7]	Display additional graphical windows (e.g. signal curve).
[F8]	Close additional graphical windows.
[F10]	Switch over for the operation by keyboard between menu bar of the work area and results window.
[F11]	Continue a previously halted measurement (menu item **Routine / Continue**).
[F12]	Start or stop the measurement process (menu items **Routine / Start sequence** and **Routine / Stop**).

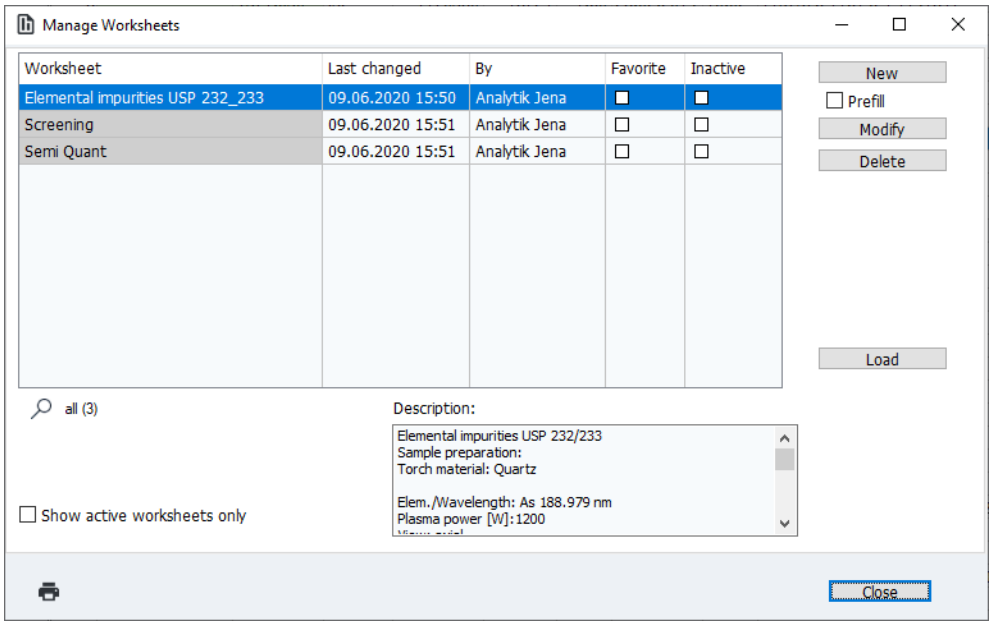
Choosing a printer	If you have already set up a Windows default printer, this printer will be used in ASpect PQ.

2 Managing worksheets

A worksheet is a folder that combines a method and a sequence. In addition, settings for a sample ID and for results data can be stored in a worksheet. You can start the measurement of the sequence directly from a loaded worksheet (→"Starting with Worksheet" p. 9).

You can create, change, delete, deactivate or load worksheets. You can find the functions for this in the **Manage Worksheets** window.

You open the **Manage Worksheets** window by clicking on in the toolbar.



Manage Worksheet window

Buttons / Options	Description
[New]	Create new worksheet
Prefil	An already loaded sequence and method are transferred to the worksheet.
[Change]	Edit selected worksheet
[Delete]	Delete selected worksheet
[Load]	Load selected worksheet for measurement
Show active worksheets only	Hide all worksheets in the table that are marked **inactive.**
Decription	Description of the selected worksheet This information is stored when the worksheet is created.

The following information about the worksheets is displayed in the table:

Column	Description
Worksheet	Worksheet name

Last changed	Date of the last modification of the worksheet
By	User how made the last modification The name of the user is taken from the Quick Start.
Favorite	If activated, the worksheet is displayed on the **Favorites** tab in the **Quick Start** window
Inactive	If activated, this worksheet is not displayed in the Quick Start However, a worksheet marked as inactive can be loaded from the **Manage Worksheets** window.

2.1 Creating a new worksheet

- To create a new worksheet, open the **Manage worksheet** window by clicking on  in the icon bar and click **[New]**.

  Alternatively, you can click on  in the toolbar.

  ✓ The **New worksheet** window appears.

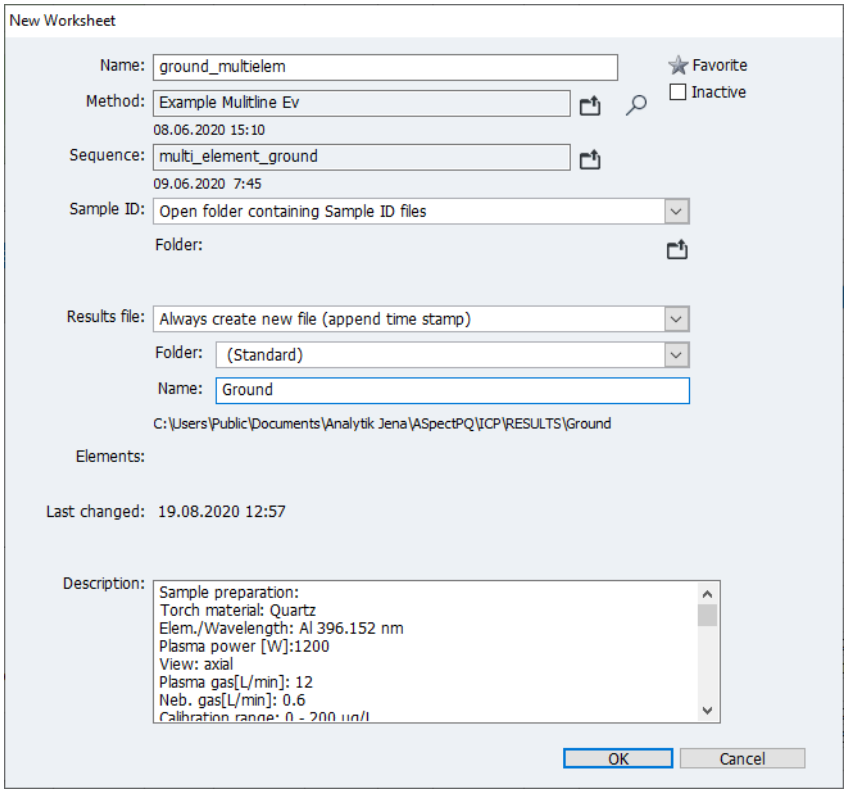
- Select a method and a sequence.

  Note: In a sequence, additional methods can be loaded as actions.

- Optionally, you can enter specifications for saving the results file and the use of a sample ID file and edit the description (see below "Elements in the New worksheet window").

- Exit the window with [OK].

  ✓ The new worksheet appears in the **Manage worksheet** window and can be loaded.



New Worksheet window

Elements of the New Worksheet window

Field / Option	Descrption
Name	Enter the worksheet name
Method	Method specified in the worksheet Open the database window with and select the method.
Sequence	Sequence specified in the worksheet Open the database window with and select the sequence.
Sample ID	Optionally, you can make settings for loading a sample ID file: **(none)**: No settings are stored for the sample ID file. **Open folder containing sample ID files**: After loading the worksheet, a folder is opened in which the sample ID file is available. Click and select the folder. **Load sample ID file**: When the worksheet is loaded, a sample ID file is automatically loaded. Click and select the file. You can also use the wildcards "*" and "?" to define a file mask.

Result files	Optionally, you can make settings for saving the results. **(none)**: Measurement starts with **Start measurement** window, in which the name of the results file and the storage location are assigned. **Always create new file (append timestamp)**: Result files of a sequence are always saved in a new file. The file name consists of a fixed component (name) and the time stamp of the measurement. Select a folder in which the file is saved and enter a name. **Create and append to file**: The result file is created the first time the sequence is started. At each subsequent sequence start, the results are appended to this file.
Description	In the **Description** field, some analysis parameters extracted from the method are displayed by default. You can edit this information as you wish and thus provide concrete information on how to use the worksheet. The entries appear in the Quick Start and in the **Manage Worksheets** window for a selected worksheet.
Favorite	With a click on the star, you can mark the worksheet as a favorite: Yellow star: Favorite Grey star: No favourite
Inactive	If activated, the worksheet will not be shown in the Quick Start

2.2 Editing worksheets

You can edit all settings in an existing worksheet.

- Open the **Manage Worksheets** window by clicking on in the toolbar.
- Select the worksheet and click on **[Change]**.
  - ✓ The Edit Worksheet window appears.
- Make the changes in the same way as you create a new worksheet.

2.3 Deleting worksheets

You can delete worksheets.

- Click on in the toolbar to open the **Manage Worksheets** window.
- Select the worksheet and click on **[Delete]**.
  - ✓ The worksheet is deleted after a confirmation prompt.

2.4 Loading worksheets

You can load a worksheet in the **Quick Start** (→ "Starting with Worksheet" p. 9) or in the **Manage Worksheets** window:

- Open the Manage Worksheets window by clicking on in the icon bar.

- Select the worksheet in the table with a mouse click and click on **[Load]**.
  - ✓ The worksheet is loaded and sequence is displayed in the main window.

Depending on the worksheet configuration you can now link the method and sequence loaded with the worksheet to a sample ID file or start the measurement directly.

Notice

When loading a worksheet, the current versions of the method and sequence are always used.

Notice

If you load a method or sequence that differs from the worksheet, the settings for the results file and sample IDs in the worksheet are reset.

3 Methods

In Methods the parameters required for an analysis are stored:

- Selection of analysis lines
- Parameters for line analysis
- Plasma and spectrometer settings
- Type of sample supply
- Calibration parameters
- Statistical analyses
- Settings for quality control and assurance
- Settings for quality control and ensuring quality
- Settings for output of the measurement results

The method forms the basis for a measuring sequence which defines the sequence of sample measurements and other actions within an analysis (→ "Sequences" p. 53). Saved methods can thus be used for analyses with different sequences.

The **Method** window is opened by clicking on in the icon bar. The last active method is displayed. If no method has been loaded since program start, the window displays contain the main settings or are empty.

3.1 Creating, saving and loading methods

Methods are saved in a database. If the method parameters of an existing method are changed and these changes saved under the same name, a new version of the method is created. The existing method can therefore not be overwritten or be unintentionally deleted in this way. Further information on managing methods can be found in section "Managing methods and sequences" p. 124.

3.1.1 Create new method

When creating a new method you can make use of default settings, parameters of a saved method or current method parameters.

▶ Select the menu item **File | New Method** and enable one of the three options and open the corresponding **Method** window:

Option	Meaning
Based on default parameters	Open a window for the entry of new method parameters (only with editable default settings for calibration and statistics).
Based on current parameters	Open the **Method** window with the currently set method parameters.
Based on saved method	Open the **Load method** window. After selecting a method, its parameters are displayed in the **Method** window.

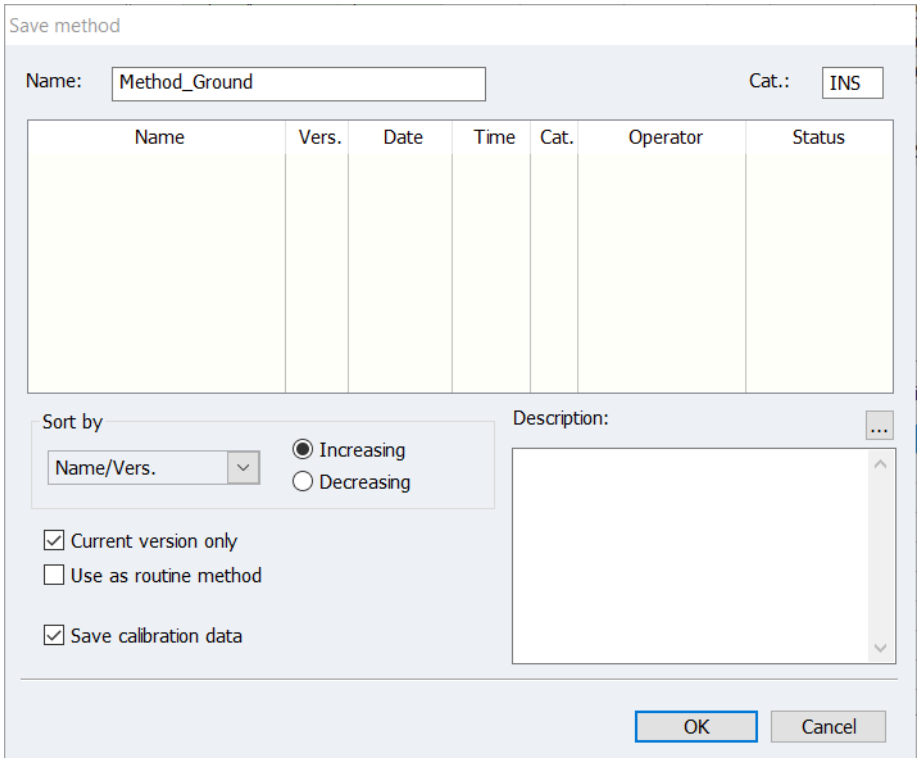
- Alternatively click on  or select the menu item **Method Development | Method**, to open the **Method** window with the current parameters.
- Apply the necessary method settings (→ "Method parameter settings" p. 26).
- Enable the set method parameters with the buttons **[OK]** or **[Accept]** for the subsequent analysis.

3.1.2 Save method

After entering the method parameters save the method to the database:

- In the **Method** window click on **[Save]** to open the **Save method** window.

  Alternatively select the menu item **File | Save | Method**.



Window Save method

- Make the following settings:

Option	Entry / setting
Name	Enter the method name.
Use as routine method	If enabled, the method is available in the program mode **Routine** (→ "The Quickstart window" p. 7).
Table	Overview of existing methods Using the options in the group **Sort by** you can sort methods by various criteria. If the option **Current versions only** is enabled, only the method with the latest version will be shown for methods bearing the same name.
Description	Optionally enter further explanations for the method.

Cat.	Optionally enter a category (three characters) for further identification and sorting the methods.
Save calibration data	Existing calibration graphs are saved with the method and can be used for subsequent analyses.

▶ Save the method with [OK].

On doing so, the method will be saved to the database. If you choose an existing method name, the existing method will not be overwritten, but a new version created in the database. To remove methods from the database, they have to be deleted explicitly (→ "Managing methods and sequences" p. 124)!

Note

The method is also saved in the result file of the measurement. After having opened the results file, you may also reproduce the method.

3.1.3 Load method

Method parameters can be loaded both from the method database and from an existing results file.

Loading from the database

▶ Open the database window with one of the following alternatives:
  – in the toolbar click on the folder icon next to the field **Method**
  – select the menu item **File | Open method** or
  – in the **Method** window click on **[Open]**.

▶ Choose the desired method from the list.

▶ In the field **Cat.** you can limit the displayed methods by selecting a category. If you want to see all methods, delete the entry in the field **Cat.**

▶ Enable the checkbox **current version only** if only the method with the latest version is to be shown for methods bearing the same name.

▶ Open the **Method** window with **[OK]**.

Loading from a results file

The method can be extracted from a results file displayed in the main window.

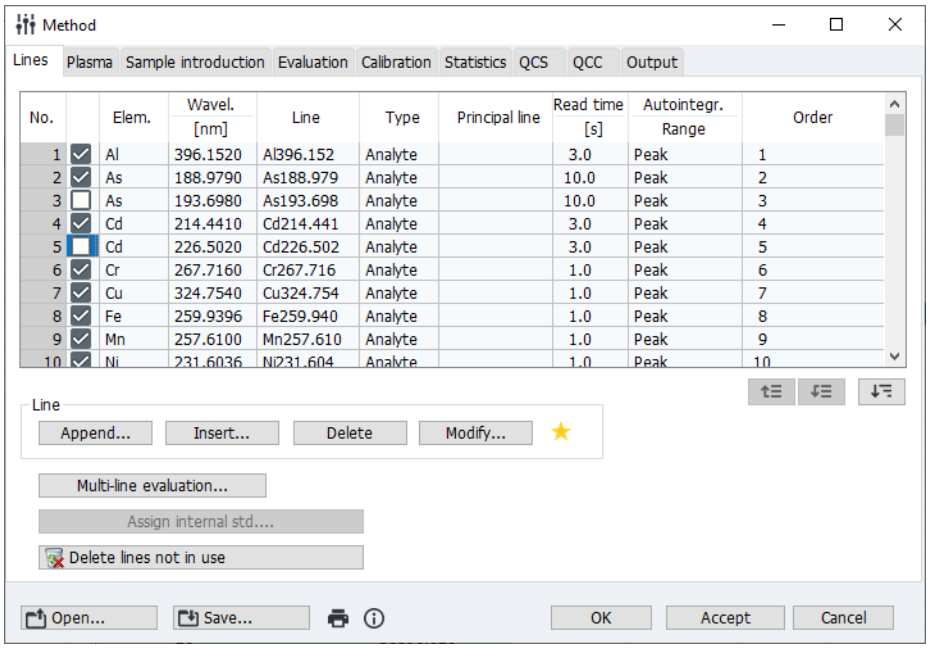
▶ Right click on any sample.

▶ In the context menu select the item **Load method from result**.

▶ After a query whether current method parameters should be overwritten the method can be displayed by clicking on  .

3.2 Method parameter settings

3.2.1 Selection of analysis lines – Lines tab



Window Method / Lines

Line table parameters

Table column	Description
No.	Sequence of selected lines in the table
☑ / ☐	Only available in the mode **Method development** The marking eases method development, where several lines of an element are measured at the beginning and then the appropriate line is selected. If an element line with a check mark is activated, this line is used and measured for the analysis. Deactivated lines are excluded in the following analysis and are not measured. Deactivated lines are not yet explicitly deleted from the line table.
Elem.	Element icon of the element to be analyzed
Wavel. [nm]	Wavelength of analysis line in nm
Line	Name of the analysis line. In the main settings the name of the line consists of the element symbol and the wavelength. However, the name can be edited freely and must be unique.
Type	Selection between **Analyte** (line to be analyzed) and **Int. Standard** (internal reference line)

Principal line	Display with which analysis line the current line is measured simultaneously (simultaneous measurement). The measuring time can be shortened by measuring lines that are close together with one spectrometer setting. After clicking on **[Multi-line evulation]**, the possible combinations are displayed. (→ "Measuring lines simultaneously" p. 30).
Read time	Total measuring time for an analysis line
Autointegr. Range	The integration time is automatically chosen for optimum exposure of the CCD detector and to avoid over-exposure. With over-exposure the charge absorbed by a pixel spills over to adjacent pixels and causes measuring errors (blooming effect). To determine the integration time the area under consideration must be selected: **Spectrum** – The integration time is optimized for the highest peak within the spectral range of the line. This is the default option and leads to a safe result. **Peak** – The integration time is optimized for the analysis peak. When selecting this option the dynamic range of the CCD detector is used optimally for the analysis. It must, however, be ensured that no higher peak is present in the immediate vicinity of the analysis pixel. In this case the measuring result could be distorted by the blooming effect. **Detector** – The integration time is adapted to the highest peak on the detector. In this option no area of the detector is over-exposed; it may be possible that the pixels of the analysis peak are not optimally exposed.
Order	Analysis order. The measuring order can be freely defined. **Note:** After highlighting a number the numbers are assigned to the subsequent rows in ascending order after clicking on . The element lines can thus be arranged in the desired measuring order in the table with and ; enter "1" under Order in the first row and assign the measuring order to all other analysis lines in ascending order with .

Buttons in the group Lines

Using the buttons **[Append]**, **[Insert]** and **[Modify]** you add additional analysis lines to the line table or edit a selected line (→ "Tables" p. 15). After clicking on one of these buttons the window **Select Element/Line** for further entries. Using the button **[Delete]** you delete a selected or several analysis lines from the method.

Additional buttons

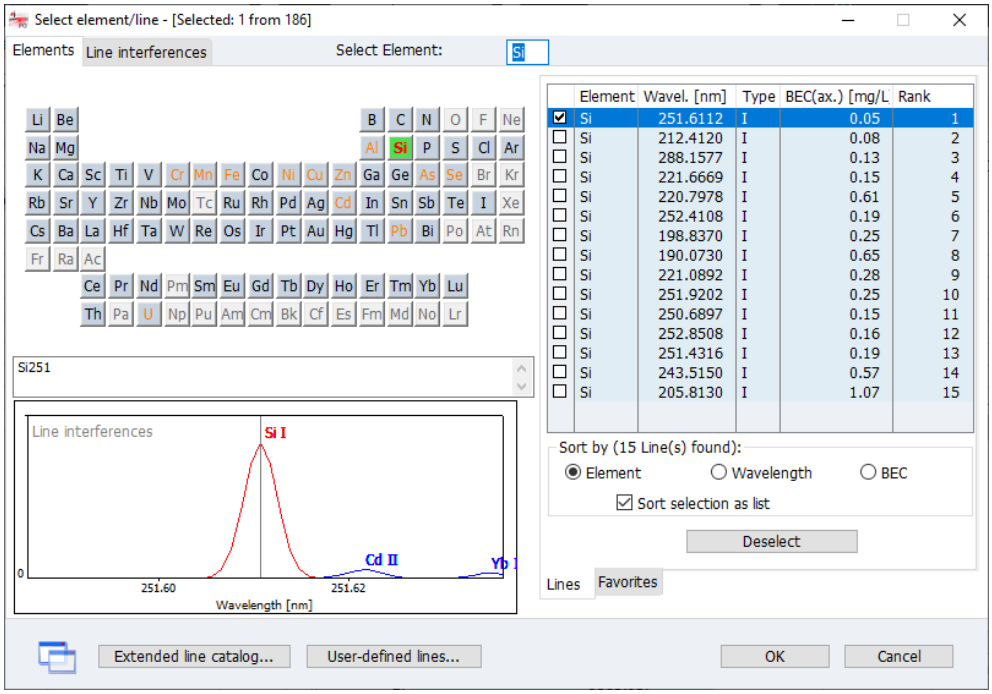
Button	Description
Multi-line evaluation	Analysis lines that are detected together with a monochromator adjustment can be measured simultaneously (→ "Measuring lines simultaneously" p. 30).
[Assign Internal std.]	Combine and correct analysis lines with an internal standard (→ "Assigning internal standards" p. 31).

[Delete lines not in used]	Only available in the program mode **Method development**. Delete all disabled lines from the method list. **Note:** Methods can only be saved and used as routine methods if all lines in the line table are being used.

3.2.1.1 Inserting analysis lines into the line table

Select the window Elements/Line

The analysis lines are selected in the window **Select Element/Line**. The **Elements** tab contains the periodic system with all elements analyzable with the ICP-OES technology (dark gray buttons and black element symbols). "Grayed out" elements are not available. The **Line interferences** tab shows the known possible interferences for a selected line with relative sensitivities.



Window Select Element/Line

The spreadsheet **Favorites** contains a preselection of lines with the recommended applications (key words). When selecting these lines, optimized method parameters are simultaneously transferred to the method. You can also add your own lines to these favorites (→ "Defining own line favorites" p. 31).

The spreadsheet **Lines** contains all selectable lines with the following information:

Table column	Description
Element	Element
Wavel. [nm]	Analysis wavelength
Type	Atomization type: I Atom line II Ion line

BEC(ax.)	Typical BEC value of the analyte line. The BEC value (background equivalent concentration) is the concentration of the analyte producing an intensity equivalent to the background. A lower value corresponds to a higher sensitivity. The BEC values were determined under the following conditions: axial monitoring, output 1200 W, plasma gas flow 12 L/min, auxiliary gas flow 0.5 L/min, nebulizer gas flow 0.6 L/min.
Rank	Ranking of usability of the analysis line. The usability of an analysis line depends both on the sensitivity and on the possible interference from adjacent lines of other elements. The further forward a line is in the ranking, the better the chances of achieving good results with the analysis line.

Using the options **Element, Wavel.** or **BEC** you can sort the line table in ascending order by chemical symbol, wavelength ore BEC.

If the option **SORT SELECTION AS LIST** is enabled, the lines are inserted into the line table of the method in the sorting order of the list (**sort by**). If the option is disabled, the lines are inserted in the order they are selected.

Selecting lines

- In the window **Method / Line** click on **[Append]** or **[Insert]**.

  The window **Select Element/Line** opens.
- In the periodic system click on an element symbol (gray buttons are selectable elements). This only displays the lines of the selected element in the line table / favorites table.
  Alternatively, enter the element symbol into the field **Select Element**.
  Delete the entry in the field **Select Element** to display the entire element list in the line table.
- In the spreadsheet **Favorites** select the lines for your application or enable the checkboxes of the desired lines in the line table.
- Change to the **Line interferences** tab, enable the checkboxes of the desired lines in the line table and check them for known interferences.

  Note:
  When working through the methods select several lines for each analyte.
- Continue until you have selected the lines for each analyte. Exit the window with [OK].

  The selected lines are transferred to the window **Method / Line**.

Extended line catalog

After installation the line list contains a preselection of analysis lines. This can be supplemented by analysis lines from the extended line catalog.

- In the window **Select Element/Line /Elements** click on **[Extended line catalog]**.
- In the list select the lines by mouse click.
  The selection is removed by a second mouse click on an individual line. **[Cancel selection]** removes all selections.
- By clicking on **[Add to line list]** the selection is transferred to the line list.

Note

The lines added from the extended line catalog cannot be removed from the standard catalog.

Creating and editing own analysis lines

You can create your own analysis lines in ASpect PQ.

- In the window **Select Element/Line /Elements** click on **[User-defined lines]**.
- In the window **Edit lines** enter the data for the **Element** and the **Wavelength** and select the **Type** in the list field.
- Transfer the entries to your own line list with **[Add]**.
- With **[Close]** your own lines are transferred to the line list.

Own lines can be edited and removed again from the line list.

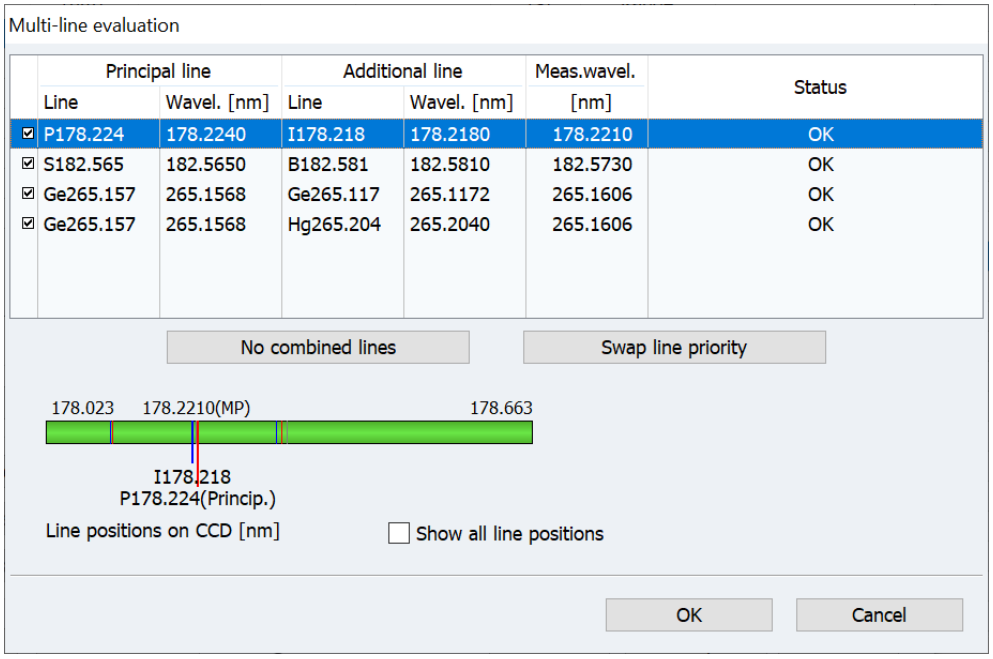
- To edit a line in the own list, select the line with a mouse click in the list of the window **Edit lines**.
  Enter the new line data and then click on **[Modify]**.
- You can remove a selected entry in the list by clicking on **[Delete]**.

3.2.1.2 Measuring lines simultaneously

When combining lines a search is performed in the current measuring program for lines that can be recorded together with the same monochromator configuration by the detector and thus also be measured simultaneously.

- In the **Methode/Linien** window click on **[Mult-line eavoluation].**

  The window of the same name appears.



Mult-line eavoluation window with line combinations

The **Multi-line evaluation** window lists the line combinations that can be recorded simultaneously by the detector. A diagram shows the position of the lines on the detector for the selected list row.

Table properties

Table column	Contents
Checkbox	If enabled the respective line combination is measured simultaneously in the method.
Principal line	**Line** Line name of the measuring line **Wavel. [nm]** Wavelengths in nm of the measuring line
Additional line	**Line** Line name of the additional line to be analyzed **Wavel. [nm]** Wavelength in nm of the additional line to be analyzed
Meas. wavel. [nm]	Measurement wavelength
Status	Remarks
[No combined lines]	Delete all selections of line combinations. No lines in the method are measured together.
[Swap line priority]	Swaps the main line and additional line in a line combination.

For a line combination a main line and the additional line are automatically determined. The additional lines accept all method parameters determining the measurement from the main line. This applies to the analysis time and plasma parameters. With **[Swap line priority]** this assignment can be reversed.

3.2.1.3 Defining own line favorites

You can add favorite analysis lines to a favorite list with notes on the preferred application. The information on the analysis lines is saved with all line-relevant method parameters in this entry. The favorite list is available during each selection of element lines.

- Mark the line in the table of the fixed **Method | Lines** and click on ☆.
- In the window **Add to favorites** edit the line name.
- In the **Comment** field you can enter additional notes for the line.
- In the list **Tags** select one or several applications.
  You can supplement the key word list with your own entries. Predefines key words are highlighted in blue.

The line is then available in the window **Select Element/Line**.

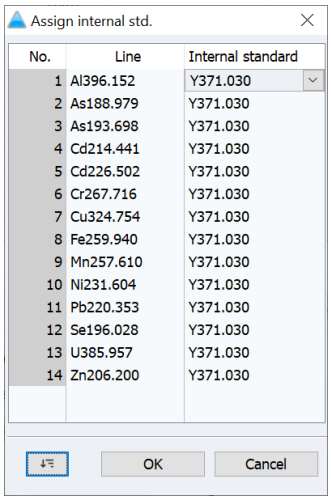
3.2.1.4 Assigning internal standards

Internal standards are mainly used to correct non-spectral interference caused e.g. by sample transport faults.

- Insert the analysis line that you want to use as internal standard into the line table and in the table column **Type** select the option **Int. std.**
- Click on **[Assign internal Std.]**.

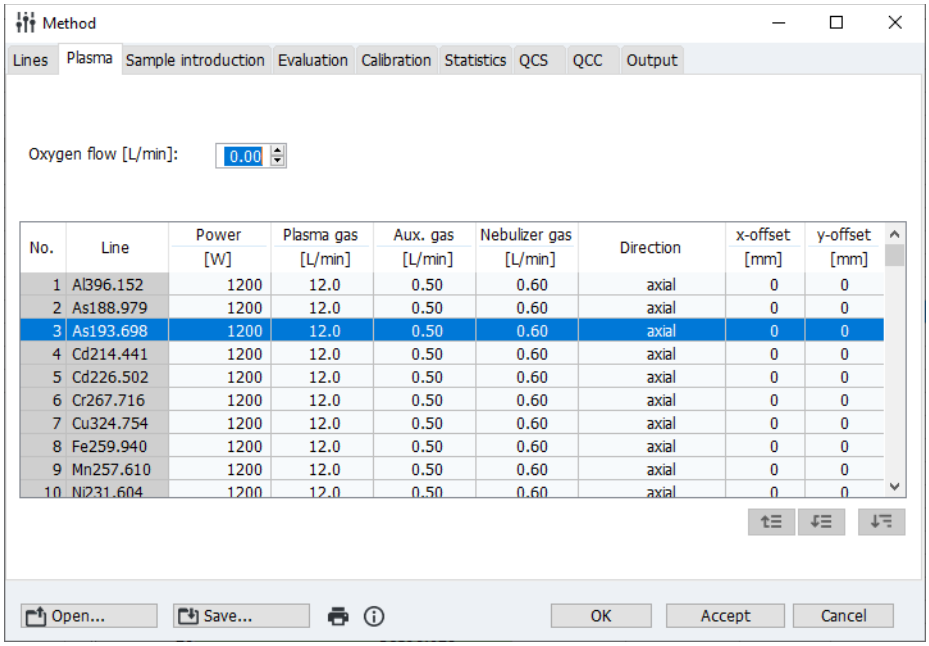
  The window **Assign internal std.** opens.
- You can now assign an internal standard to each analysis line in the table.

- With a click on you transfer the settings for an analysis line to all subsequent lines in the table.
- With [OK] the settings are transferred to the method.



Window Assign internal Std.

3.2.2 Configuring parameters for plasma and transfer optics – Plasma tab



Window Method / Plasma

Table parameters

Table column	Description
No.	Sequence of selected lines in the table.
Line	Name of the analysis line. The name is specified on the **Line** tab.

Power [W]	Change the effective plasma power Increasing the plasma power improves the stability of the plasma, e.g. with organic solvents or samples with a high salt content as measuring solution. At the same time a higher plasma power also requires a higher plasma gas flow to prevent melting or damage of the torch.
Plasma gas [L/min]	The plasma gas flows between the outer and inner quartz tube of the torch. It is put into the plasma state by the induction of the coil and simultaneously cools the outer tube of the torch. A higher plasma gas flow can improve the lifetime of the torch.
Aux. gas [L/min]	Auxiliary gas flow. The auxiliary gas flows between the inner quartz tube and the injector. It supports the development of the measuring channel.
Nebulizer gas [L/min]	Nebulizer gas flow. The nebulizer gas is introduced at the nebulizer. It nebulizes the sample and moves it through the nebulizer chamber and injector into the plasma.
Direction	Select the monitoring direction of the plasma. With the transfer option the emission radiation from the plasma can be coupled to the spectrometer from two directions. Dependent on the analyte line the optimum monitoring direction can be selected. **radial** — The plasma is monitored from the side at a specific height above the upper coil edge. **axial** — Monitoring is from the top along the longitudinal axis of the plasma. Both monitoring directions can also be weakened. This avoids an overflow of the detector for high intensities and increases the analytical range.
x-offset [mm] and **y-offset [mm]**	Correction of the transfer optics By shifting the optics along the measuring channel (for radial observation) and out of the center of the measuring channel (for radial and axial observation), areas of varying hotness can be scanned and the optimum emulsion temperature of the analysis line can be determined. The optimum for a line can be determined automatically in the **Plasma** window (→ "Adjustment and optimization of plasma" p. 107).

Note

During the first phase of the method development (selection of suitable lines) the preset plasma parameters are sufficient. These parameters can be changed after defining the analysis lines, the necessary background corrections and the determination of the linearity range to further optimize the method parameters.

Using oxygen

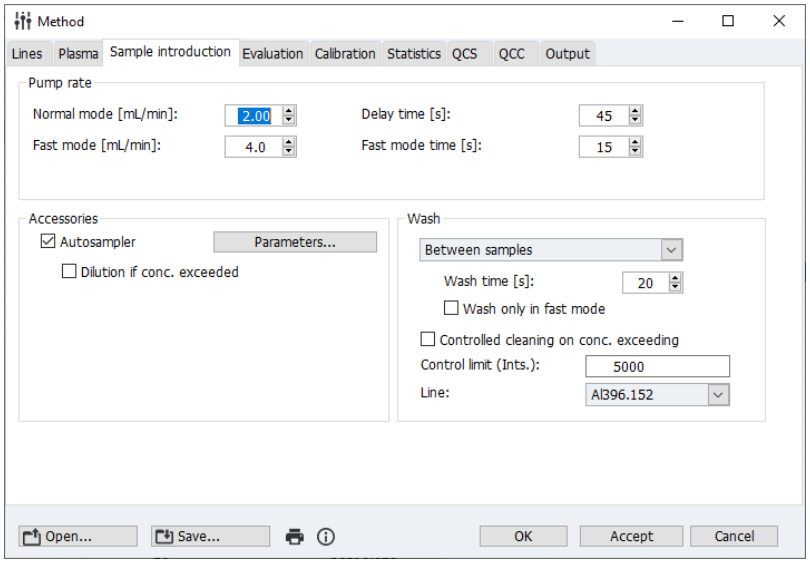
For special applications, e.g. organic matrices, oxygen can be used as additional gas.

▶ Set the gas flow in the field **Oxygen flow**.

3.2.3 Configurations for sample transport – Sample introduction tab

On the **Sample introduction** tab you make the following settings for the analysis device:

- Pump rate of the pump at the analysis device
- Use of the autosampler
- Wash option



Window Method / Sample introduction

Setting the pump rates

Option	Description
Normal mode [mL/min]	Normal pump speed with which the sample is transported during the analysis. This speed should ensure optimal nebulization of the sample and should be based on the recommended pumping rate of the nebulizer used.
Fast mode[mL/min]	Increased speed with which the wash solution is transported during the measuring pauses and with which the sample can be transported up to the nebulizer. Activating this speed optimizes the transport time. However, this speed must not be used during the measuring time, because the homogeneous nebulization of the sample will then no longer be ensured.
Delay time [s]	Time from the start of sample aspiration up to the actual measuring start. This time is required to wash the entire sample path up to and including the torch with the sample and ensure stable atomization. **Note:** The delay time also includes the time entered in the field **Fast mode time**.
Fast mode time [s]	Time with which the sample is transported with a high pump rate during the passing of the delay time.

Note

In the window **Method / Sample transport** set the pump rates of the hose pump of the ICP-OES device. The pump rate at the autosampler for the transport of the washing solution can be regulated with the rotary knob above the pump at the autosampler or at the Cetac samplers in the window **Autosampler / Technical parameters**.

Using the autosampler

If the autosampler is used for the analysis, enable the option **Autosampler**. Via **[Parameters]** you get to the autosampler settings.

Wash

During the completion of a sequence you can agree the washing steps after each sample measurement. The washing solution is then taken from the washing cup of the autosampler during automatic measurement with autosampler. During manual measurement a prompt is shown to provide the washing solution.

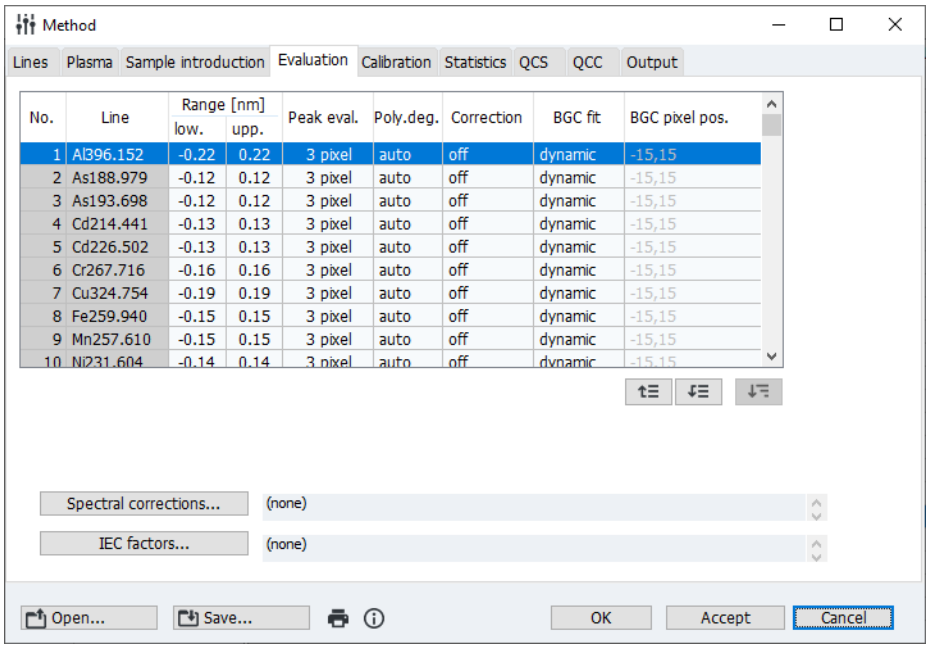
- In the group **Wash** select in the list field the option **Between samples** if you want to wash the sample path during the sequence.
- Set the wash duration in the input field **Wash time** in seconds.
- If the entire wash step is to be performed in fast mode only, activate the option **Wash only in in fast mode**. If the option is deactivated, the wash step is first performed in fast mode using the entered fast mode time and for the rest of the wash time in normal mode.
- Select the option **off** if there is to be no washing.

Controlled cleaning

If samples are analyzed which result in the calibration graph working range being exceeded by more than 10 %, the sample path and the torch can be washed in order to remove contamination from the previous measurement. During washing, the intensity is measured to check the cleaning result and washed until the control limit is reached. The automatic cleaning control is recommended after the measurement of highly concentrated samples.

Option	Description
Controlled cleaning on conc. exceeding	If activated, controlled cleaning is performed automatically when the concentration is exceeded.
Control Limit	The value to which the signal level must have returned during washing before the next samples are measured.
Line	Element line, which is used for control

3.2.4 Evaluating peaks – Evaluation tab



Window Method / Evaluation

Note

In the method development you determine the optimum settings for the background correction of the respective analysis line in the window **Edit spectra / Evaluation** and

then transfer it to the method (→ "Evaluating spectra and determining the background correction – Evaluation tab" p. 83).

Parameter of table Evaluation

Table column	Description	
no.	Sequence of selected lines in the table	
Line	Name of the analysis line The name is specified on the **Line** tab.	
Range	**low.**	lower limit of the wavelength range for spectral analysis relative to the measuring wavelength
	upp	upper limit of the wavelength range relative to the measuring wavelength
Poly.grd.	Selection of the polynomial degree of the regression graph for static background correction Polynomial degrees of 0, 1st, 2nd and 3rd order are available for selection or an automatic search for the polynomial degree (option **auto**).	
Peak eval.	Selection of the peak evaluation	
	1-19 pixels	Number of pixels used to analyze the intensity and ultimately for generating the measuring values. The intensities of the evaluation pixels are totaled. In this manner analysis inaccuracies can be reduced by fluctuations in the peak position. Recommended number of evaluation pixels: 3
	Height	Interpolation of the peak maximum
	User defined.	Free selection of the evaluation pixels, e.g. for the analysis of multiplets. Sample entry: 50,120 - 130 forms the total across the measuring values of the pixels 50 and 120 - 130.
Correction	Algorithm for spectral correction (see below)	
	off	Apply no spectral correction
	LSM	Spectral correction with Least Squares Model
	IEC	Spectral correction with Inter Element Correction
BGC fit	Adjust pixels to the background correction.	
	dynamic	The pixels for background correction are automatically found by the software.
	static	The pixels for background correction are specified by the user in the **BGC pixel pos..** column.
BGC pixel pos.	Position of the pixels relative to the measuring pixel for static adaptation of the background correction. Enter the pixel numbers for the background correction.	

Buttons

Button	Description
[Spectral corrections]	Specify a model for spectral correction for analysis line.
[IEC factors]	Specify a inter element correction for analysis line.

3.2.4.1 Spectral corrections with Least square models

With spectral corrections structured background emissions can be removed by way of calculations that were caused by the overlap of analysis lines with lines of the matrix elements. A precondition is that for the respective analysis line the possible interference spectra have been combined in a correction model (→ "Removing spectral interference – Spectral corrections " p. 85).

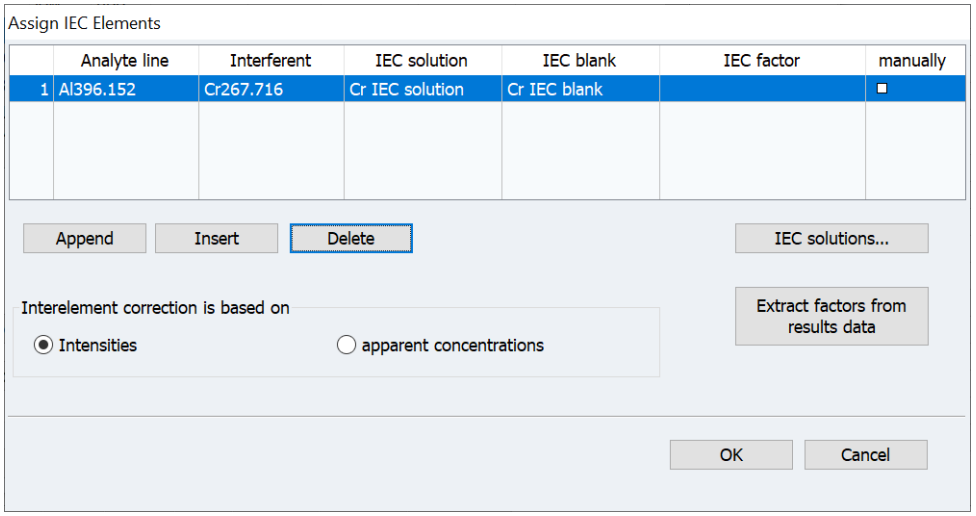
- ▶ In the **Method / Evaluation** window click on **[Spectral corrections]** and configure the suitable correction model separately for each line.
  - ✓ Lines to which a correction model has been assigned are identified with LSM in the **Corrections** column.

3.2.4.2 Interelement correction

With the interelement correction it is possible to correct direct line overlaps. A condition is an additional, interference-free wavelength of the interferent.

With a single element solution (IEC solution) the ratio of the two lines of the interferent (overlapped analysis line and interference-free line) is determined. The quotient (IEC factor) is used during subsequent sample measurements to subtract the apparent intensity or concentration of the interferent from the analyte line.

Window Assign IEC elements



Window Assign IEC Elements

Control element	Explanation
[IEC solutions]	Input of name, concentration, unit and sampler position of the IEC element solutions and blank values used for the interelement correction.
[Append]	Append a new row at the end of a list.
[Insert]	Inserts a new row at the selected place of the list.
[Delete]	Delete the marked row.

Interelement correction is based on	**Intensities**	Correction takes place by subtracting the intensities.
	apparent concentrations	Correction takes place by subtracting the apparent concentrations.
[Extract factors from result data]	Extract calculated IEC factors from a loaded result file.	

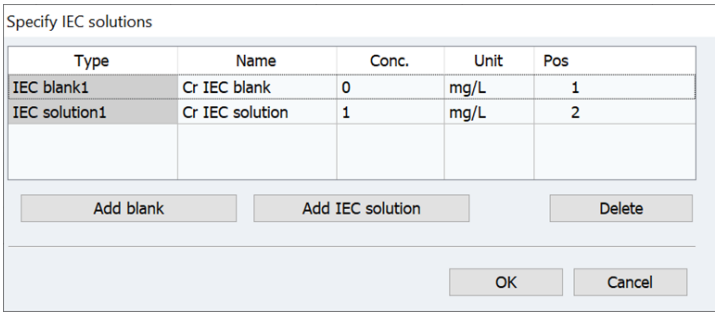
Table column	Description
Analyte line	Name of the analysis line with interference.
Interferent	Name of the interference line.
IEC solution	Name of the single element solution containing the interferent. IEC solutions are specified via the IEC solutions button.
IEC blank	Name of the blanc value solution subtracted from the intensity or concentration of the interferent. Blank value solutions are specified via the IEC solutions button.
IEC factor	IEC correction factor. Calculated factors are highlighted in gray.
manually	If enabled, an IEC factor can be entered manually. No measuring solutions are required.

Assign interelement correction

- In the **Method / Evaluation** window click on **[IEC factors].**

  The Assign IEC Elements window opens.

- First specify the IEC solutions. You need a blank value and an IEC solution (single element solution) for each interferent.
  - Click on **[IEC solutions]**.
  - In the window **Specify IEC solutions** add a blank value and an IEC solution for each interferent to the table by clicking on **[Add blank]** and **[Add IEC solution]**.
  - In the corresponding table cells enter a name for each solution.
  - For the IEC solutions enter the concentration of the interferent in the IEC solution into the **Concentration** column.
  - Confirm the entries with [**OK**].



Specify IEC solutions window with an IEC blank value and an IEC solution

- ▶ Back in the **Assign IEC Elements** window select the interfered line of the analyte in the table column **Analyte line**.
- ▶ In the **Interferent** column select the interference-free line of the interferent.
- ▶ In the columns **IEC solution** and **IEC blank** configure the corresponding single element solution and blank value solution.
- ▶ Select the type of **Interelement correction** either based on **Intensities** or **apparent concentrations**.
- ▶ Confirm the entries with **[OK]**.
  - ✓ Lines which have an interelement correction assigned are identified in the line table of the **Method / Evaluation** window with IEC in the **Correction** column.

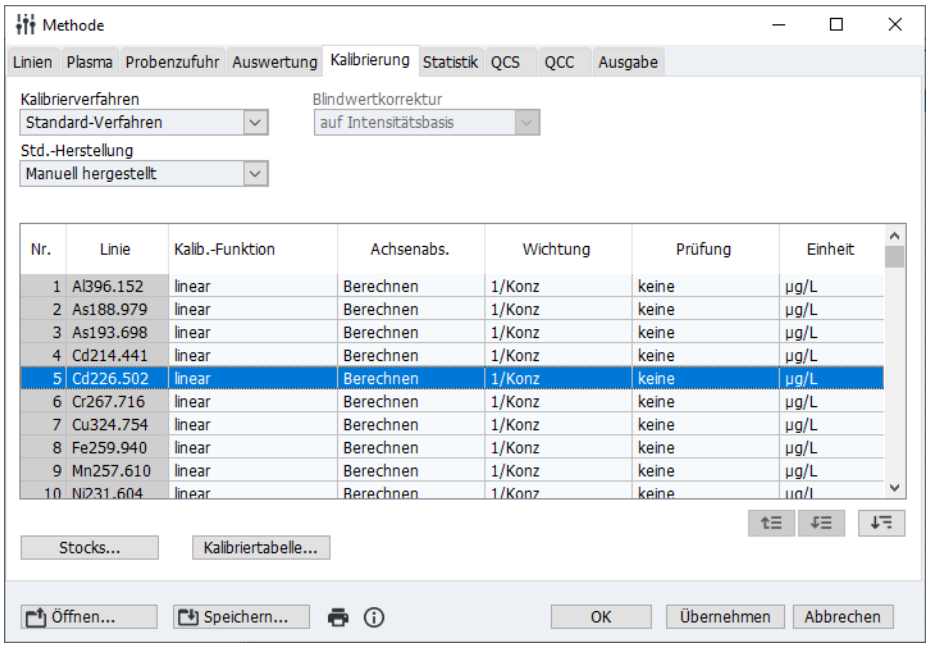
The measurement of the IEC solutions must be carried out in the sequence following the measurement of the calibration standards or calculation of the calibration.

Instead of determining the factors for the interelement correction by measuring the single element solution, known factors can be entered directly into the table.

- ▶ After entering **Analyte line** and **Interferent** enable the **manual** checkbox.
- ▶ In the **IEC factor** column enter the already calculated factor.

3.2.5 Entering calibration parameters – Calibration tab

In the window **Method / Calibration** the type of calibration and the blank value correction are selected. Generally multiple element standards are used for this purpose.



Window Method / Calib. with settings for a calibration with standard methods

Select Calibration Technique

In the list **Calibration mode** select the method:

Calibration techniques	Description
No calibration	The sample results are presented exclusively as intensity. Calibration is not necessary for these measurements.
Standard calibration	The calibration takes place with samples of known concentration in the analytes (standards). The samples of unknown concentration are measured against these calibration standards.
Method of additions	Different concentrations of a standard are added to the unknown sample and measured. The concentration of the analyte results from the comparison.
Method of additions calib.	The calibration curve, by means of which other concentrations can be determined, is set up by the method of standard addition. At the same time, the concentration of the first sample is found by this method.

Agreeing blank value corrections

Standard addition methods and addition calibrations require a blank value correction. In the list **Blank correction** select the method:

Correction	Description
Intensity corrected	In every standard addition procedure, the blank is measured too and the measured intensity value subtracted from all measured values before the regression line is calculated. This method was customary for a long time; with many real samples however, it leads to incorrect results.
Concentration corrected	First, a separate standard addition is carried out for the blank solution using the same concentration additions as for the sample. The resulting concentration is automatically subtracted from all other concentrations determined by standard addition.

Preparing standards

Option	Description
manually	The calibration standard solutions are prepared manually.
By diluter system	Only when using the Cetac ASX-560 autosampler with the dilution system set up The standards are prepared by diluting one or more stocks in the vortexer (mixing vessel) of the autosampler.

Line-specific calibration parameters

The line-specific parameters are set in the table:

Table column	Description
no.	Sequence of selected lines in the table.
Line	Name of the analysis line. The name is specified on the **Line** tab.

Calib. function	Only for calibration using the standard method **linear** - Linear progression of the calibration function $y = a + bx$	
	nonlin. ratio.	Non-linear progression of the calibration function described by a broken ratio function $y = \frac{a + bx}{1 + cx}$
	nonlin. quadr.	Non-linear progression of the calibration function described by a quadratic function $y = a + bx + cx^2$
	automatically -	For the calibration a linear and non-linear function each are calculated. The sums of the squared residuals are compared to each other (Mandel test). If the sum for the nonlinear function is significantly lower than that for the linear function, the nonlinear calibration curve will be selected. Otherwise, the linear calibration curve will be used. The non-linear function is selected in the window **Options/analysis process** (→ "Options for analysis sequence" p. 138). As default setting the broken ratio function has been provided.
	Note: Only linear graph progressions are permitted for the standard addition method and the addition calibration.	
Intercept	**Set zero**	The calibration graph exactly intercepts the measured zero point.
	calculate	The zero value is included in the calculation like any other calibration point.
Weighting	**non**	All calibration points are taken into account equally.
	1/conc	Calibration points obtained with lower concentrations are to be weighted more strongly.
	1/SD	Points with lower deviations within several repeat measurements of a standard are considered with a stronger weighting (condition: average statistics enabled).
	1/(SD*conc)	Combination of the calculation methods **1/conc** and **1/SD**.

Check	ASpect PQ provides automatic checking of determined calibration curves by means of a prognosis range that is calculated based on a manually selected statistical certainty. **none** – All measured and undeleted calibration points are used for calculating the graph. Calibration points are neither labeled nor eliminated. **Elim. outliers** – If calibration points are outside the calculated prognosis range, outliers will be eliminated by means of an F-test (a test to ascertain whether the exclusion of a point results in a significant improvement of the residual scattering): ■ An F-test is performed for the calibration point being farthest outside the prognosis range. If the exclusion of this point does not lead to a significant improvement of the residual scattering, the point will be included, and the calibration graph not further optimized. ■ If the exclusion of this point results in a significant improvement, the calibration point will be defined as outlier (marked in the table by "!", in the graph marked by red color) and the calibration recalculated without this point. ■ For the point which now lies furthest outside the forecast range (strongest deviation), another F-test is carried out – this procedure is repeated until all outliers are removed. ■ All calibration points lying outside the new prognosis range, that have not been eliminated as outliers are marked by "?" in the table and blue color in the graph.
Unit	Enter units for the concentration separately for each element.

transfers the value of the active cell to all subsequent cells in the table column. With the button **[Calibration Table]** the table for entering the standard concentration is opened.

3.2.5.1 Specifying stock standards

If you use the stock standards, you can enter the respective dilution factors for the individual standards instead of the concentrations. To this end you must specify the stock standards before completing the calibration table, and it is possible to use several stock standards with different elements and concentrations.

- In the window **Method / Calibration** use **[Stocks]** to open the window **Stock Standards**.
- With **[New]** or **[Insert]** add a new row in the stock list.
  Max. number of stock standards: 20
- For the option **From stock database** select the stock name in the list. The stock database is managed in the **Data** window (→ "Managing databases for stocks and QC samples" p. 131).
- Select the option **Enter manually,** if you do not use stock standards from the database.

Back in the window **Stock Standards** enter the data of the stock standard directly into the table:

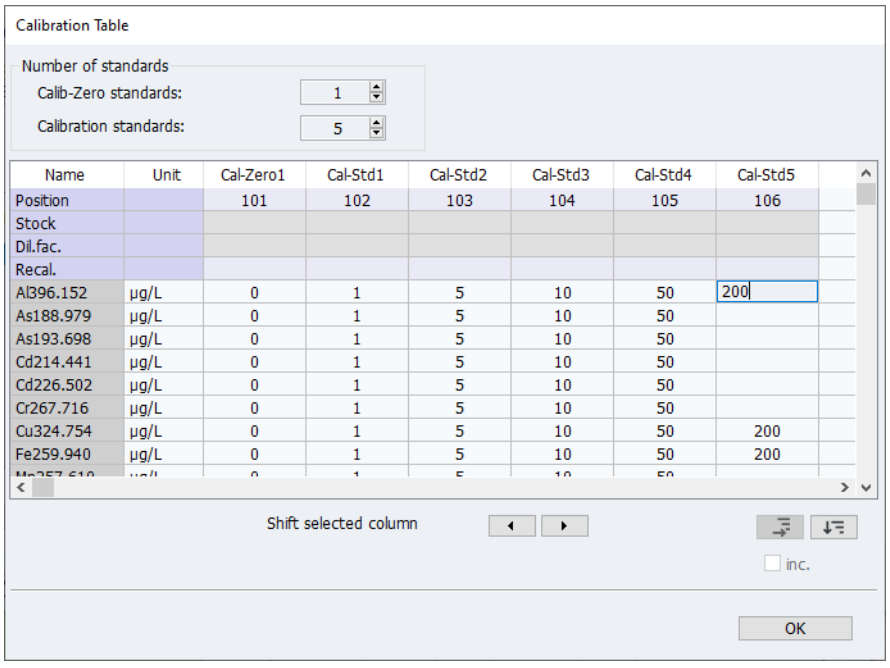
Table column	Description
Name	Name of the standard
Elements and concentrations	Elements and corresponding standard concentrations With **[Concentrations...]** open a list for entering the concentrations. Alternatively enter the value in the following input format directly into the row in the format *Element symbol-space-Concentration*, e.g. nickel with a concentration of 0.5 mg/L: Ni 0.5 Additional elements and their concentrations are simply added separated by a semicolon. An example of the input format is given under the stock list.
Unit	Concentration unit of the elements in the standard.

3.2.5.2 Entering the calibration table

Enter the standard data in the calibration table.

Note

When using stock standards for calibration, the dilution factor for producing the standards can be entered manually. Select the stock standards used with **[Stocks]** in the window **Method / Calibration**.



Window Calibration Table

- In the window **Method / Calib.** click on **[Calibration Table]**.
- First enter the number of standards into the input fields. Dependent on the calibration method chosen, various standards must be selected.

  For the standard method the number of **Calibration standards** and **Calib-Zero standards** must be entered. Several **Calib-Zero standards** entries are possible, e.g. if

the elements to be analyzed are dissolved in different solvents. In this case the concentration of the respective element line must be set to "0", the other columns remain blank.

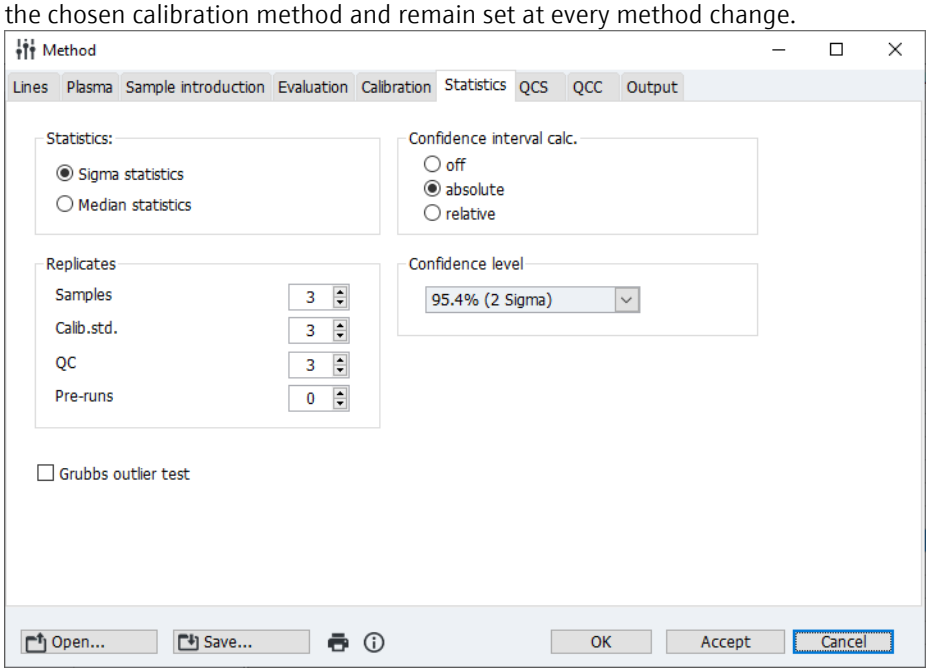
For **Method of additions** and **Method of additions calib.** the number of **Add standards** be entered in each case.

- For the preparation of standards by a connected dilution system, you must select the stock standard used in the **Stock** row and the dilution factor in the **Dil. fact** row for each calibration standard (→ "Specifying stock standards" p. 42").
  The following dilution factors can be selected: 1, 2, 5, 10, 15, 20, 25, 50, 75, 100, 200, 250, 500, 1000, 1500, 2000, 2500, 5000. The number of dilution factors is limited according to the range settings in the Sample Dispensing / Dilution window (→ "Dilution function" p. 115). For the dilution factors 1 ... 100 the dilution is carried out in one step, for higher values in two steps.
- If you prepare manually the calibration standards, you can also calculate the concentrations of the calibration standards by selecting a stock standard and entering a dilution factor.
  Alternatively, for each standard in the table, enter the concentration of each element for each analysis line.
- For manually prepared standards, you can define the position of the standards in the autosampler in the **Position** line.
  If the autosampler is not used, the entries in this line are not considered.
  For autosamplers with dilution function, the position of the stock is taken from the stock database.
  The assignment of the autosampler positions can be entered or changed in the sequence.
- For recalibrations specified as sequence actions or as a sequence of QC actions, at least one **Calib-Zero standard** and one **Calibration standard** or at least two **Calibration standards** must be selected in the **Recal**. line. If more than two recalibration standards are selected for a line, the lowest and the highest recalibration standard is used.

3.2.6 Specifying statistical analyses – Statistics tab

in the window **Method / Statistics** select the statistical methods to be applied to the calibration and the sample measurement. The settings selected here are independent of

the chosen calibration method and remain set at every method change.



Window Method / Statistics

Statistics type

Option	Description
Sigma statistics	Calculates mean value and standard deviation. Error statistics based on the arithmetic mean: The sample is measured repeatedly after the blank cycles. Based on the measurement results, the arithmetic mean, the standard deviation and the relative standard deviation are calculated.
Median statistics	Calculates median and range (R). Error statistics based on the median method: The sample is measured repeatedly after the blank cycles. The measured values are sorted by size. The displayed median is: ■ The value in the middle of the sorted list, if the number of measurement cycles is odd. ■ The mean value of the two measured values in the middle of the sorted list if the number of measurement cycles is even. As the smallest and largest individual measured values do not influence the measurement result, the median statistics are suitable for the elimination of outliers.

Number of replicates

Option	Description
Samples	Number of repeat measurements per sample
Calib. std.	Number of repeat measurements per calibration sample
QC	Number of repeat measurements per QC measurement (for QC types → "Specifying quality control samples for QC tabs - QCS tab" p. 46).

Pre-runs	Number of repeats of blank measurements Blank measurements are sample measurements preceding the statistics series and disregarded for the calculation of the measurement result.

Grubbs outlier test

This function is provided for mean value statistics with at least three repeat measurements per sample.

Status	Description
deactivated	Includes all values of the statistics series for the calculation of the mean value.
activated	Outliers are eliminated and are not used in the calculation of the statistics. The thus found mean values in the result table are marked by "!".

Confidence interval calc.

The calculation of the confidence interval is based on the chosen statistical certainty (see below). In the calculation of the confidence interval, the errors in sample measurement and particularly the calibration errors are included, so that a value will be presented even if the statistics function has been deactivated.

Adjustment	Description
of	Confidence interval is not calculated.
absolute	Displays the confidence level in absolute values (in concentration units)
relative	Displays the confidence interval in relative values (in percent of the concentration value)

Confidence level

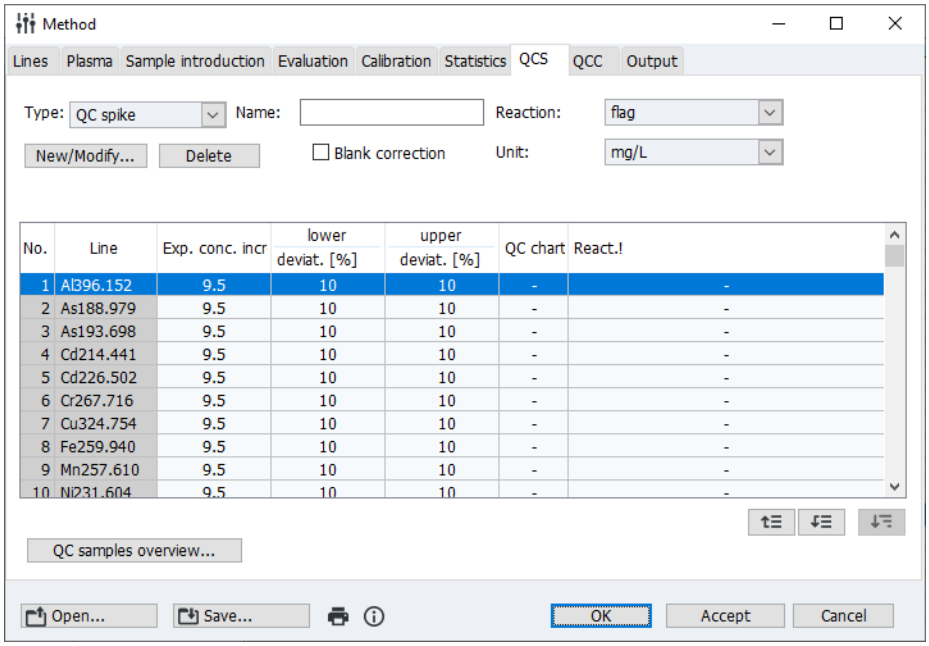
The **Confidence level** (selectable between 68.3 ... 99.9%) is used for the calculation of the confidence interval of the samples and the prognosis ranges of the calibration graphs.

3.2.7 Specifying quality control samples for QC tabs - QCS tab

Specify the QC samples for the quality control tabs in the window **Method / QCS**. The system of the QC control tabs serves to monitor quality over a long period of time. This involves the insertion of control measurements, with samples in predetermined positions during measurement. These control measurements should yield known results. It is either the absolute value (intensity or concentration) or the concentration difference from the previous sample which is known.

The results of the quality control measurements are automatically recorded on so-called QC charts (also called quality rule charts or control charts). The tabs are saved with the method and continued for any further measurement with the method.

You may define various quality control samples (QC samples). The details on the concentrations of these samples and their tolerances are entered in the window **Method / QCS**.



Window Method / QCS

Elements of the QCS tab

Elements	Description
Type	Select QC sample type whose parameters (error limits and procedures) to be displayed in the line list. You can select a QC sample from the list for viewing and editing.
Name	Name of the displayed QC sample
Reaction	Select the procedure to be taken if the results of the QC sample exceed or fall below the defined error limits.
[New/Modify]	To be activated for defining a new or modifying an existing QC sample.
[Delete]	Deletes the displayed QC sample.
Unit	List box for selecting the corresponding concentration unit.
[QC samples overview]	Opens a list with the line-specific parameters of all QC samples.
Table	The table displays the parameters of the QC sample selected in the Type list box.

Entering parameters of QC samples

- ▶ Create a new parameter record for a QC sample type with **[New/Modify]** or modify the one currently displayed.
  The window **Add/modify QC sample type** opens.
- ▶ From the **Type** list, select the sample type and, if you intend to define several QC samples of the same type, assign a number (e.g. QC-Std.2). The following sample types are selectable:

Option	Description
QC sample	Defines a sample as QC sample. The concentrations of the QC sample may either be loaded from the database or typed in directly. To recall a stored data record for the QC sample from the database, enable the option **from database** and select the corresponding QC sample from the adjacent list box (→ "Managing databases for stocks and QC samples", p. 131). Alternatively, you can enter the concentrations of the QC sample directly in the table in the **QCS** window. In this case, enable the option **enter manually**. Max. number of QC samples: 50
QC std.	Define a standard as QC sample. Any standard defined in the calibration table (**Clib.** tab) can be used as QC standard. The autosampler positions will be transferred from the **Calibration table**. The assigned number at the same time defines the used calibration standard e.g. "QC-Std.2" - the second calibration standard is used as a QC sample. Possible number of QC standards = number of standards in the calibration table (max. 65)
QC blank	Defines the blank as QC sample.
QC spike	Defines a spiked sample as QC sample. In recovery/addition, the measurement results of a defined concentration addition to one or several samples are checked. To this end, a QC stock sample is to be defined after any sample in the sample table (QC-Stock sample = sample + addition with a solution of known concentration). After the measurement the concentration difference (Conc1 of sample and QC stock sample) is compared to the "Expected concentration increase" specified here and the recovery rate calculated.

If no certified quality control samples are available, quality control may also be carried out by means of double determinations:

Option	Description
QC trend	The measured concentration value is stored when the quality control sample appears for the first time in the analytical procedure. When the QC sample appears the next time, the concentration difference is formed and evaluated. It is advisable to measure these QC samples at the beginning and at the end of a sample measurement series.
QC matrix	A sample to be analyzed is split before preparing the sample. Both portions are separately subjected to all steps of sample preparation. They are placed separately on the autosampler tray as QC Trend and QC Matrix. The difference between the determined concentrations is evaluated.

- In the list **Reaction** select the subsequent procedure if the error limit is exceeded:

  For **QC sample**, **QC std.** and **QC spike**

Option	Description
flag	The measured value is flagged in the sample table; the measurement program continues measuring the next sample.

recal. + continue	A recalibration is run. The QC sample is then measured again. If the QC sample is now within the permissible range, the measurement is continued with the next sample; otherwise, the measurement program will be stopped.
cal. + continue	A new calibration is run. The QC sample is then measured again. If the QC sample is now within the permissible range, the measurement is continued with the next sample; otherwise, the measurement program will be stopped.
recal. + return	A recalibration is run. The QC sample is then measured again. If the QC sample is outside the range, the measurement program will be stopped. If it is within the range, all samples measured after the last QC sample or the last (re)calibration, will be re-measured. If the QC sample is again outside the permissible error limits, the measurement program will be stopped.
cal. + return	A new calibration is run. The QC sample is then measured again. If the QC sample is outside the range, the measurement program will be stopped. If it is within the range, all samples measured after the last QC sample or the last (re)calibration, will be re-measured. If the QC sample is again outside the permissible error limits, the measurement program will be stopped.
next method	The current measurement program is canceled and the measurement program of the next method started if the sequence includes another method.
stop	The current measurement program will be stopped.

For **QC blank** it is possible to choose between the reactions **flag**, **next method** and **Stop** described above.

For QC Stock it is possible to choose between the reactions **Flag, Recal + continue, Cal. + continue, Next Method** and **Stop** described above.

For **QC trend** and **QC matrix** no reaction has been provided.

- For **QC trend** and **QC matrix** an optional blank correction has been provided. To this end enable the checkbox **Blank correction.**
- In the list of QC samples, define the line-specific parameters for every element line dependent on the QC sample type selected:

Option	Description
Line	Name of element line
Exp. conc.	For QC Sample and QC Std. Expected concentration in QC sample
Exp. conc. incr.	For QC stock Expected concentration increase from the sample to the spiked sample Enter the value corresponding to the stock volume and the concentration of the stock solution.
Ext. Ints.	For QC blank value Expected intensity in QC blank value
upper limit [%]	Lower range of error limit in percent.
lower limit [%]	Upper range of error limit in percent.
QC chart	If marked with "+", the result of the quality control for this line will be presented on the QC tab in the result list.

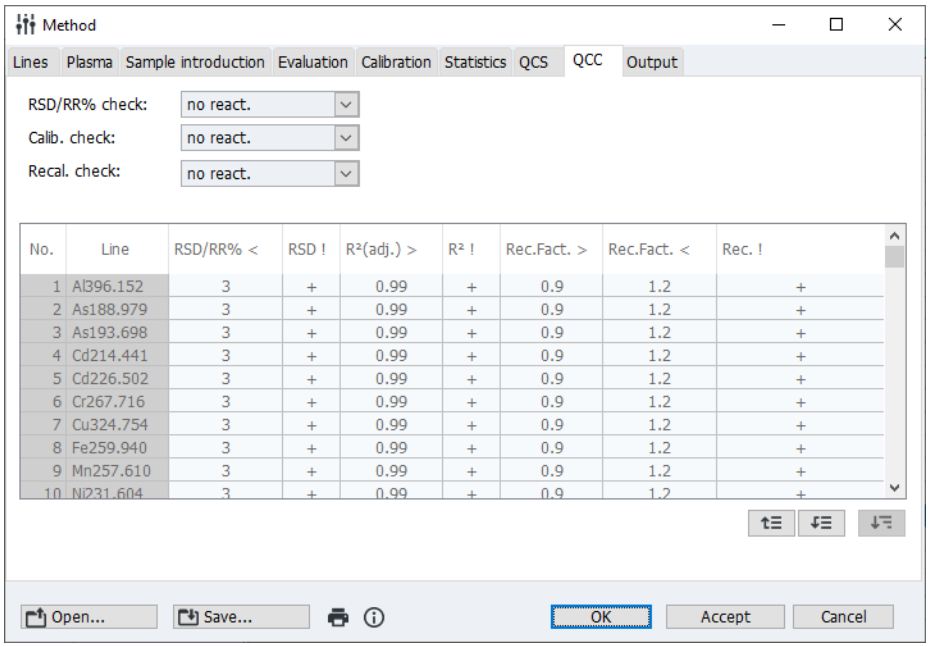
React.!	If the error range limits are exceeded, the procedure selected in the Reaction list will be applied. If several lines have been marked by "+", it will do that the error limits are exceeded for one of these lines to start the selected reaction (OR logic).
Unit	Unit of the expected concentration (only with selected QC Std. option)

3.2.8 Specifying quality control during the sequence - QCC tab

In the window **Method – QCC** specify the parameters for the quality control during a sequence:

- relative standard deviation (mean value statistics) or relative range (median statistics)
- calibration control and recalibration control
- procedure to be run if error limits are exceeded.

You may choose various control options with different reactions simultaneously.



Window Method / QCC

Types of quality control

Control type	Description
RSD/RR% check	Control of the relative standard deviation or relative range (→ "Specifying statistical analyses – Statistics tab", p. 44).
Calib. check	Control of the coefficient of determination of the calibration
Recal. check	Control of recalibration factor

Reactions if error limits are exceeded

Reaction	Description
no react.	The corresponding check is not performed.
flag	Marks the corresponding sample, calibration or recalibration in the sample table, if the error limits are exceeded.
repeat + continue	Only **RSD/RR% control** Repeats the measurement of the respective sample, if the serial precision limit was exceeded, before the next sample is measured.
cal. + continue	Only **Cal. check** and **Recal. check** Runs a new calibration if the error limits for the calibration or the recalibration factor were exceeded. Afterwards, measurement is continued with the next sample.
next method	Only **Cal. check** and **Recal. check** The current measurement program is canceled and the measurement program of the next method started if the sequence includes another method.
Stop	Only **Cal. check** and **Recal. check** Stops the measurement of the currently running method, if the error limits were exceeded.

Line-specific parameters of quality checks

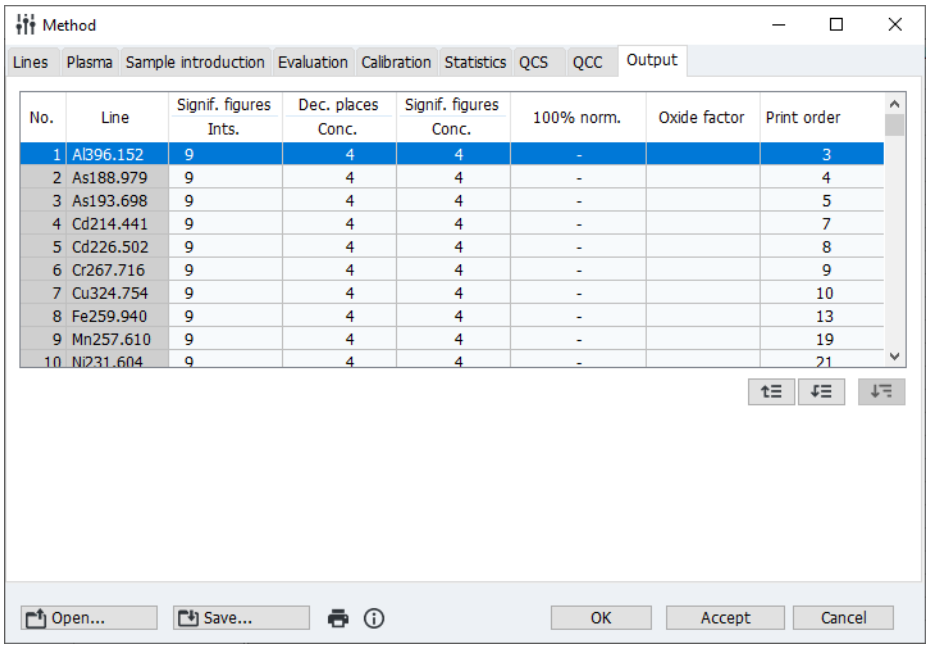
In the table, enter the line-specific parameters of the various quality checks. You may define for every analysis line, whether it shall be considered for the check. If one or several of the checked lines exceed the error limit, the reaction chosen above will be released.

Quality control	Parameter	Meaning
RSD/RR% check	**RSD/RR%<**	The system will respond with the defined procedure if the relative standard deviations or the relative ranges are larger than or equal to the specified value. RSD! — For lines marked with "+", the RSD% or RR% will be checked.
Calib. check	**R2(adj.)>**	The coefficient of determination R2(adj.) must be larger than or equal to the specified value. Otherwise, the system will respond as selected. R^2! — For lines marked with "+", R2(adjust) will be checked.
Recal. check	**Rec.Fact>** **Rec.Fact<**	Upper limit of recalibration factor Lower limit of recalibration factor. The selected response will be released, if the calibration factors are outside these specified limits. **Rec.!** — For lines marked with "+", the recalibration factor will be checked.

3.2.9 Specifying output formats for results - Output tab

On the **Output** tab specify the number of decimal places with which results are presented on the screen and on the printouts, additional output types, and the order of lines for an analysis of several elements on the printout.

In the table below, define the number of decimal places for the display and printout of intensity and concentration values, and the order in which the analysis lines shall appear on the printout.



Window Method / Output

Element	Description
Signif. figures. (Ints.)	Number of significant figures of intensity values.
Dec. places (conc.)	Number of decimal places of concentration values.
Signif. figures (conc.)	Number of significant figures of concentration values.
100% norm.	The concentration of the original sample (conc. 2) is converted to the percentage value in relation to the total concentration. The total concentration is the sum of the concentrations of the lines marked with "+".
Oxide factor	If an oxide is selected, the element concentration (conc. 2) is converted to the oxide concentration. The oxide factor is displayed in parentheses, e.g. Ti is converted to TiO_2 by multiplication with 1.6681.
Print Order	Order in which line data are displayed in the report.

4 Sequences

The sequence contains the information on the samples and actions within the measurement in their order of processing. It is based on a loaded method, which contains the information on the type of calibration, statistical analyses, quality control, etc. Some sample describing data such as sample name and position on the sample rack may also be entered directly. For permanent storage, however, the sample describing data must be saved as sample information file.

The meaning of buttons and symbols contained in the **Sequence** window, which are also used in other windows of this application, are described in Section "Frequently used control elements" p. 14.

4.1 Creating, saving and opening sequences

Like methods, sequences are saved to a common database. When saving and opening sequences, the database window is utilized (→ "Managing methods and sequences", p. 124).

Create new sequence

- To open the **Sequence** window, click on in the icon bar.

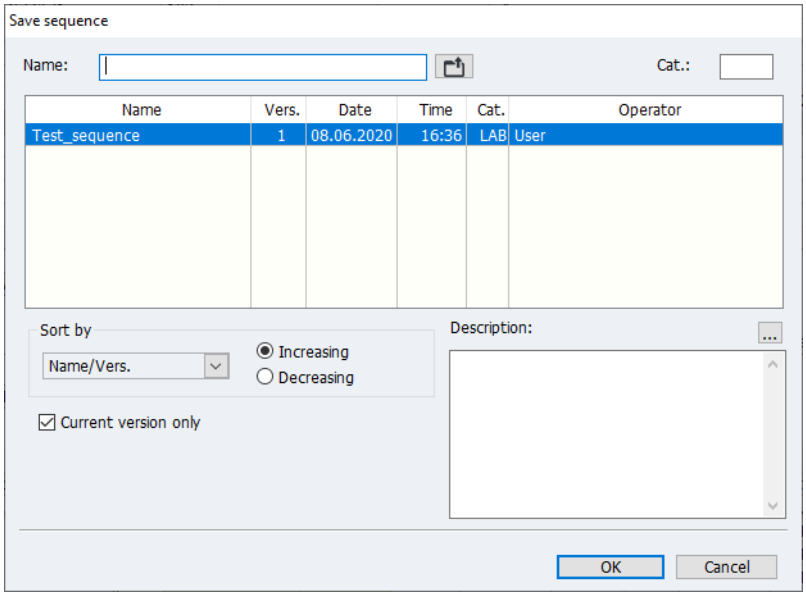
  Alternatively you can select the menu items **File | New Sequence** or **Method development | Sequence**.
- Make the settings in accordance with section "Combining sample and action sequences for the sequence" p. 56.
- Click on **[Accept]** to release the sequence for the subsequent measurement or save the sequence.

Save sequence

- In the **Sequence** window click on **[Save]**.

  Alternatively select the menu item **File | Save | Sequence**.
- In the **Name** field of the database window enter the sequence name.
- In the field **Cat.** (category), you may optionally enter an additional identifier of three characters to facilitate later sequence search in the database.
- In the **Description** field you can optionally enter information about the sequence.
- Save the sequence with **[OK]**.

On doing so, the sequence will be saved to the database. If you choose an existing sequence name, the existing method will not be overwritten, but a new version created in the database. To remove sequences from the database, you must explicitly delete them (→ "Managing methods and sequences" p. 124)!



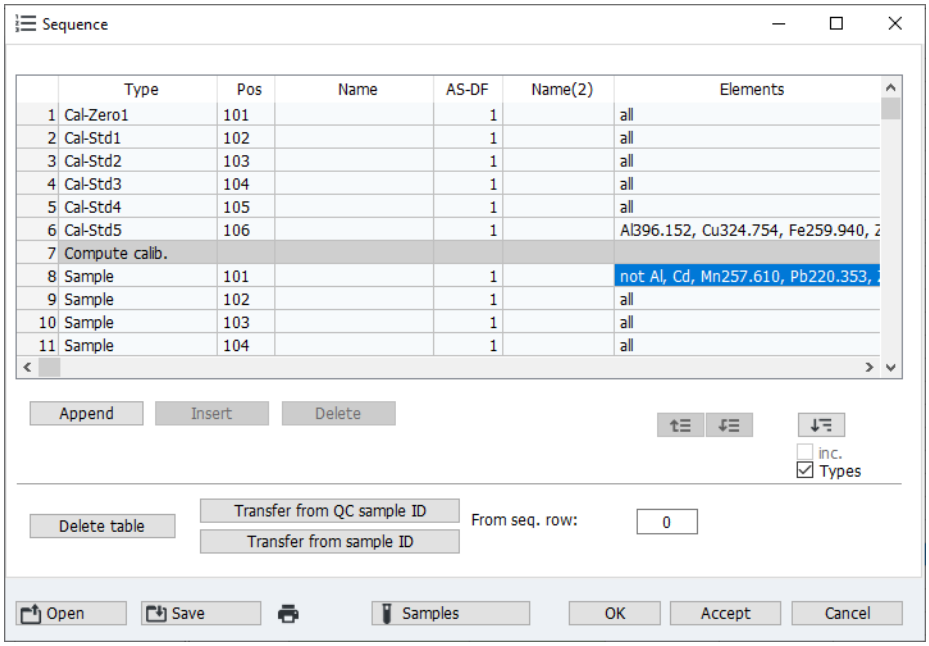
Database window for saving the sequence

Opening sequence

- Open the database window with one of the following alternatives:
  - in the toolbar click on the folder icon next to the field **Sequence**
  - select the menu item **File | Open Sequence** or
  - in the **Sequence** window click on **[Open]**.
- Choose the desired sequence from the list.
- In the field **Cat.** you can define that only sequences of the specified category are displayed.
  If you want to see sequences of all categories, delete the entry in the **Cat field.**
- Enable the **Current version only** checkbox if for sequences of the same name you want to see the sequence of the highest version number only.
- Open the selected sequence with [OK].

4.2 Dialog functions in the Sequence window

After clicking on the **Sequence** window opens.



Window Sequence

Table of sample and action sequences

The table shows the selected sample and action sequences in the order of processing. The following related information is displayed:

Table column	Explanation
Type	Sample type or analysis step.
Item	Sample position on autosampler tray (if used).
Name	Sample name This entry is optional. For calibration and QC samples this sample name is transferred from the method if a sample name was specified there. For analysis and QC samples the names can be transferred from the sample information file.
Name (2)	Additional designation for sample identification (optional).
Elements	Select the elements to be analyzed in a sample or for which special actions are performed (→ "Selecting elements/lines for a sample analysis/action" p. 59). ■ "none" Current selection is deleted. ■ "all" All elements/element lines defined in the method will be determined (default). ■ Element symbol Only the specified elements will be determined, e.g. "Cu, Pb". ■ Element line (symbol+wavelength) Only the specified element lines will be determined, e.g. "Mn 257.610, Ca 315.887". ■ "not" element symbol or element line The specified elements or element lines are not determined, e.g. "not Cu, Pb", "not Mn 257.610, Ca 315.887"

Buttons in the Sequence window

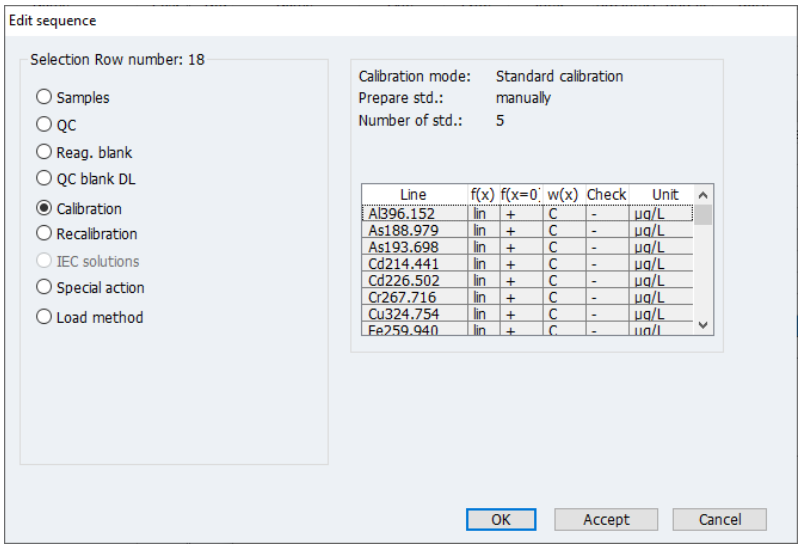
With the buttons you can add samples and actions to, or delete them from, the sequence list or accept existing sample information data.

Button	Explanation
[Append]	Add a new row at the end of the list and open the **Edit Sequence** window.
[Insert]	Inserts a new row above the selected place of the list.
[Delete]	Delete marked lines.
[Delete table]	Delete entire sequence list.
[Transfer from QC sample ID]	Transfer information about the QC sample name and position in the autosampler from the window **Sample ID / QC sample information**. The information from the QC sample ID table are entered in the sequence table. The first row with the new sample ID is be defined in the field **From seq. row**.
[Transfer from sample ID]	Transfer information about sample name, position in the autosampler and the elements to be analyzed from the window **Sample ID / Sample information**. The information from the sample ID table is entered in the sequence table. The first row with the new sample ID is be defined in the field **From seq. row**.
[Samples]	Open the window **Sample ID.**

Additional buttons and entry options have been described in section "Tables" p.15.

4.3 Combining sample and action sequences for the sequence

- Load or create a method.
- Open the window **Sequence** by clicking on .
- Click on **[Append]**. The window **Edit Sequence** opens.



Window Edit Sequence with calibration selection

- Consecutively select the options for the samples and actions and transfer these to the sequence list with **[Accept]**:

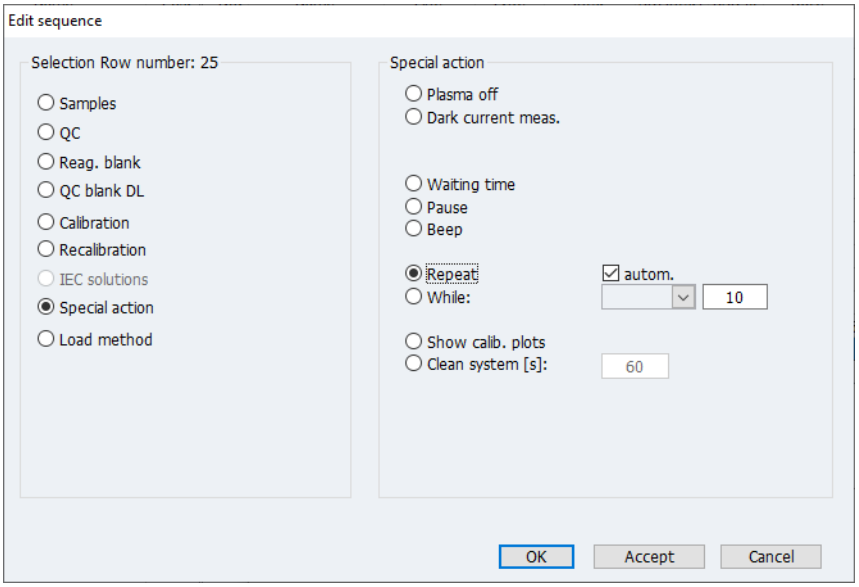
Sample/Action	Description
Samples	Measure the number of samples specified in **Number**.
QC	Measure a QC sample and evaluate it as specified in the Method. In the list select a QC sample specified in the method. The parameters of the QC sample are displayed in the opposite field.
Reag. blank	Measure the blank value.
QC blank DL	Measures a blank for the determination of the detection and quantitation limits according to the blank method.
Calibration	Measures the calibration samples and runs the calibration according to the options defined in the method.
Recalibration	Measures the calibration sample provided for recalibration and performs the recalibration.
IEC solution	Only for peak correction with IEC Measure the IEC solutions.
Special action	Perform actions which do not directly affect the measurement of samples. List of possible special actions (→ "Inserting special actions in the sequence" p. 58).
Load method	Load a saved method, e.g. to start another element analysis within a sequence. With [...] open the database window with the saved methods (→ "Load method" p. 25). Select one of the two saved methods.

- After selecting the last sample/actions of the sequence, accept them with **[OK]** and then return to the **Sequence** window.
- As default for the element to be analyzed the option **all** has been selected in the sequence table for each sample/action. By clicking on the table cell Elements of the respective sample/action, you can change this setting in the window (→ "Selecting elements/lines for a sample analysis/action" p. 59 below).
- When using the autosampler:
  Define the position (**Pos.**) of the samples in the autosampler. The positions of calibration and QC samples are automatically taken from the method. However you can change the positions here, the positions set in the sequence always have priority.

Note

The data of the samples to be analyzed should best be entered in the window **Sample** ID and subsequently transferred into the sequence list (→ "Specifying information data for samples and QC samples" p. 61).

4.3.1 Inserting special actions in the sequence



Window Edit sequence with special actions

The following special actions can also be inserted in the measurement process:

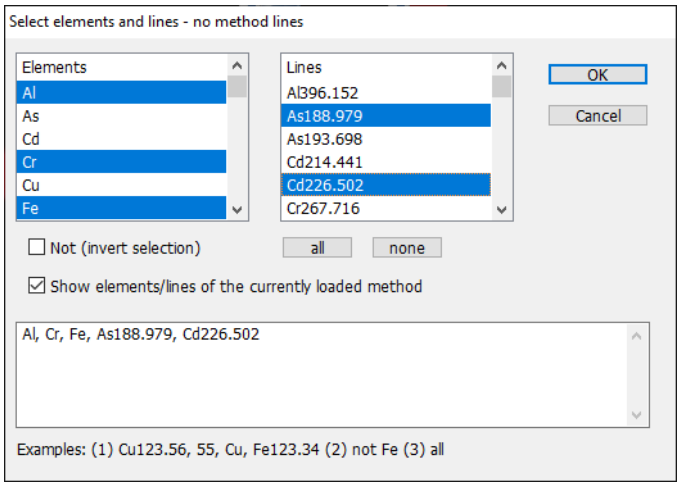
Action	Description
Plasma off	Extinguish the plasma.
Dark current meas.	Perform additional dark current measurement. With this measurement of the dark current the signal is determined with closed shutter. The dark current measurement is always performed automatically, even if it is not inserted in the sequence.
Waiting time	Wait for the time (in minutes) entered in the field and then continue the analysis. When using an autosampler, the cannula remains in the rinsing position and further rinsing fluid is aspirated.
Pause	Stop the analysis. The sequence can then be continued with or with the menu item **Routine | Continue**.
Beep	Allow the PC to generate a signal tone, e.g. in order to indicate the end of the calibration. (Requires a sound card and loudspeaker.)
Repeat / While	Define a loop (repetition) in the sequence. The part of the sequence enclosed by the starting point **Repeat** and the end point **While** is repeated until the cancellation criterion has been met. As a cancellation criterion a number of loop cycles or a time in minutes can be specified. For an online measurement (as part of remote maintenance) the option **autom.** must be enabled. This prevents the prompt for sample metering during the manual mode.
Show calib. plots	Display the calibration curve during the sequence in progress. In this case, the measurement is only continued after the calibration has been confirmed with **[OK]**.
Clean system [s]	Wash the sample path up to the torch with washing solution with normal pump speed. Enter the cleaning time in the field.

4.3.2 Selecting elements/lines for a sample analysis/action

In the sequence all elements for the analysis of samples or the execution of actions have been enabled by default. If you want to exclude elements for the analysis of a sample or an action, proceed as follows:

- In the **Sequence** window click on the table cell **Elements** of the corresponding sample or action. The window **Select elements and lines** opens.

  By default all elements/lines set in the method have been enabled. In the **Elements** list all elements are highlighted in blue.



Select element lines for the analysis/action

- To fully exclude an element, remove the selection by clicking on the corresponding element. To enable the element, click on the element again.
- If several lines have been set for an element in the method and you only want to use selected lines, select the desired line with a mouse click in the **Lines** list.
- With the buttons **[all]** and **[none]** you either enable all elements or exclude all elements for the analysis/action.
- Using the option **Not (invert selection)** all selected elements/lines are excluded from the analysis/action. Only the unselected elements/lines will be analyzed. The list of elements/lines is preceded by "**not**".

In the output field all selected elements/lines are listed. The elements/lines can be edited directly in the table cell after returning to the sequence window.

5 Sample information data

The sample information data (sample IDs) include the specific data for the current analysis samples and QC samples, such as sample name, position on the autosampler, weight, dilution or concentration unit. Sample names and positions can be transferred to the sequence table by mouse click. The sample information data are saved as a table in the *.csv format and can also be edited in a spreadsheet program, e.g. Excel. The reverse is also possible: externally created sample tables can be imported to ASpect PQ.

The window with the sample information data is opened with a click on  in the icon bar.

5.1 Creating, saving and opening sample information data

Create a new sample/ID record

- To open the **Sample ID** window, click on  in the icon bar.

  Alternatively open the **Sample ID** window with the menu commands **Method Development | Samples** or **File | New Sample Information File**.
- Make the settings in accordance with section "Specifying information data for samples and QC samples" p. 61.
- Save the record.

Saving sample IDs

- In the **Sample ID** window click on **[Save]**.

  Alternatively select the menu item **File | Save | Sample information**.

  The default window **Save as** appears.
- In the **File name** field, enter the desired name for the sample information.
- Save the sample information with **[Save]**.

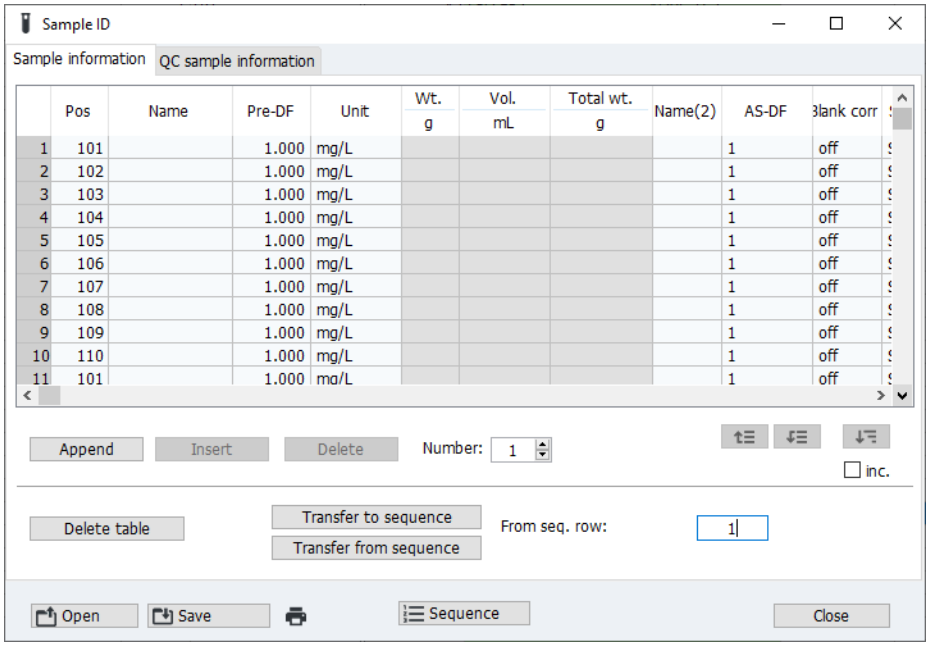
Open sample information data

- Open a sample information file with one of the following alternatives:
  - in the toolbar click on the folder icon next to the field **Samples**
  - select the menu item **File | Sample Information File** or
  - in the **Sample ID** window click on **[Open]**.

  The default window **Open** appears.
- Choose the desired file from the list and open the file with t with **[Open]**.

5.2 Specifying information data for samples and QC samples

After clicking on the Sample ID window opens.



Window Sample ID

The program pops up brief information windows on the meaning of the table columns when moving the mouse pointer over the table header. The meaning of buttons and symbols contained in the **Sample ID** window, which are also used in other windows of this application, are described in Section "Frequently used control elements" p. 14. The sample information data then need to be transferred to the sequence.

5.2.1 Sample information tab

The window Sample ID / **Sample information** includes a list of samples and their properties.

Table column	Description
Item	Position of sample on autosampler
Name	Sample name This entry is optional. Max. number of characters: 20
Pre-DF	Only for the unit type **liquid** and **solid** (→ "Specifying units of measurements" p. 131) The pre-dilution factor of the sample designates the factor with which the original sample has been diluted before it has been placed into the autosampler, or when working without autosampler, has been supplied to the plasma. The factor is required for the calculation of the concentration of the original sample (Conc. 2).
Unit	Concentration unit of sample.

Wt. [g]	Weighed portion in grams (only for the unit type **solid**) Mass of the original sample which was dissolved in sample preparation. The entry of the weight is required for the calculation of the concentration of the original sample (Conc.2).
Vol. [ml]	Total volume or filling volume (only for the unit type **solid**)
Total wt. [g]	Total weighed portion (only for the unit type **liquid grav.**).
Name (2)	Additional sample name. This entry is optional. Max. number of characters: 20
Blanc corr.	Blank value correction (only for sample type **Sample**) **off** No blank correction performed. **on** For the calculation of the concentration of the original sample, the blank value measured last in the sequence will be subtracted.
Sample type	Selection between **Sample** and **Reag. blank**
Elements	Elements or lines to be analyzed in the sample After clicking on the table cell the window **Select elements and lines** opens in which these settings are made (→ "Selecting elements/lines for a sample analysis/action" p. 59).

Control buttons	Description
[Append]	Insert number of new rows at the end of the list.
[Insert]	Insert number of new rows before the selected place in the list.
[Delete]	Delete the marked row.
Number:	Input field for the entry of the number of rows to be inserted.
[Delete table]	Deletes the complete table of sample information.
[Transfer to sequence]	Transfer sample names, positions in the autosampler and elements to be analyzed to the sequence list. The first row of the sequence list from which the sample data should be transferred must be defined in the input field **From seq. row**.
[Transfer from sequence]	Transfer sample names, positions in the autosampler and elements to be analyzed from the sequence list to the sample ID table. The first row of the sequence list from which the sample data should be transferred must be defined in the input field **From seq. row**.

5.2.2 QC sample information tab

Analog to the **Sample information** tab, this tab contains the QC samples.

The columns correspond to the columns of the **Sample information** tab. In addition, the column **Type** includes the information about the QC type. The column unit is omitted because the unit has been defined in the method. A blank correction for QC samples is defined in the method and displayed in the **Blank corr.** column for information (on/off).

With the button **[Transfer to sequence]** the data can be transferred to the sequence list.

5.2.3 Specifying sample information

- Click on to open the **Sample ID** window.
- in the **Number** field enter the number of samples to be analyzed. Then click on **[Append]** to insert the corresponding number of rows into the list.
- In the table, enter the required information for every sample.
  - If the entries in the column are identical, you can use to copy the entry of the selected cell to all subsequent cells in the column.
  - If you enable the checkbox **inc**. (for increment), the value is increased by 1 at a time when transferring the information to the next cell. In this way, you can easily e.g. assign successive places on the tray of the autosampler or number a sample name consecutively.
  - Text can be copied to the Windows clipboard and re-inserted via the menu items **Edit | Copy** and **Edit | Insert** or the key combinations [Ctrl+C] and [Ctrl+V]. You can also marc the text and use the right mouse button to open the context menu for copy and paste.
- Once you have entered all the information, specify in the **From sequence row** field from which row onwards you want to transfer sample information to the sequence. Transfer the information with **[Transfer to sequence]**.

6 Performing analyses / calculating results

6.1 Overview of the menu commands and buttons for starting the analyses in the main window

Sequences are executed with the icons in the toolbar or vie the ROUTINE menu.

Icon	Menu item	Function
	**Routine | Start Sequence**	Starts an analysis process.
	**Routine | Run Selected Sequence Row**	Repeats the selected rows in the sequence. Several rows can be marked using the mouse in combination with the Ctrl- and/or Shift-Key.
	**Routine | Stop**	Stop the analysis process.
	**Routine | Continue**	Continues a stopped sequence.

6.2 Igniting plasma/extinguishing plasma

Igniting plasma

Ignite the plasma as follows:

- ▶ Switch on the ICP-OES device at the power switch.
- ▶ Switch on the PC at the power switch and boot the operating system.
- ▶ Open the gas supply ensuring a preliminary pressure of 6 bar.
- ▶ Switch on the exhaust unit.
- ▶ Switch on the recirculating chiller at the mains switch.
- ▶ Check if the torch is in starting position. The injector tip must be situated approx. 1 to 2 mm below the bottom edge of the induction coil.
- ▶ Close the door of the plasma chamber.
- ▶ Check the pump hoses. Replace hoses that are no longer flexible or showing signs of heavy abrasion.
- ▶ Clamp the pump hoses between two stoppers in the pump on the ICP-OES device.
- ▶ Place the hose guides over the hoses and attach them with the clamping levers. Make sure that the clamping levers snap into place!

  Note!
  Note the pump flow direction. This pump rotates anti-clockwise.

- ▶ Ensure that enough washing solution is in the bottle for the analysis.

  Note:
  The washing solution should have the same acid content as the samples and standards. Use a 2% nitric acid solution unless specified differently.

- ▶ Check the filling level of the waste bottle and empty the bottle if there is too little volume left for the analysis.
- ▶ Dip the sample suction hose into the wash solution in case of manual operation without automatic sampler. No air must follow during the plasma ignition process.

  For automatic operation switch on the automatic sampler at the power switch and fasten the pump hose for the washing solution. Wait for the initialization phase of the autosampler.
- ▶ Start the ASpect PQ program (→ "Starting ASpect PQ" p. 7).
- ▶ If the system was out of service for a longer period (more than a week) or the nebulizer chamber was dismantled, purge the chamber and the torch using nebulizer gas to evacuate any air from the system:
  - Use to open the **Plasma / Control** window.
  - Click on **[Purge spray chamber]**.
- ▶ Ignite the plasma:

  With open the window **PLASMA / Control** and click on **[Ignite plasma]**.
- ▶ This is followed by an initial phase in which the torch is purged with argon and the safety circuits of the ICP-OES device are tested. This takes about 1 min. If everything is OK, the plasma is ignited. Verify that the plasma is forming correctly, i.e. the plasma forms a cone shape, extends beyond the induction coil and tapers upwards.

  If a ring plasma forms (plasma is formed only inside the induction coil) or a rattling sound is heard, you hit the red Plasma-OFF button on the left side of the device. Verify before the next ignition attempt that the sample tube is immersed in the washing solution and that the gas supply and the recirculating chiller are working properly. Also check that the torch is correctly assembled and that the sealing rings are in proper condition.

  ✓ The plasma ignites and the hose pump and the cooling of the detector start. The ICP-OES device is ready for measuring after a brief warm-up period.

Note

Prior to plasma ignition internal safety circuits will check gas flow, cooling and exhaustion and verify that the plasma torch is in working position (clamped in height adjustment) and the sample chamber door is closed. If a fault is detected in one of the components the plasma will fail to ignite.

Extinguishing the plasma

Extinguish the plasma and switch off the ICP-OES device as follows:

- ▶ After the analysis is completed you let washing solution flow for approx. 3 min and then water for 1 min through the system. Allow the device to run dry after this. In case you need to replace any hoses, they will be drained of acid!
  - ▶ Extinguish the plasma in the ASpect ICP software by clicking on in the tool bar. Alternatively use to open the **Plasma w**indow and click on **[Plasma off]**.
- ▶ Exit the ASpect PQ program with **File | Exit**.
- ▶ Confirm the query on turning off the purge gas for the detector with **[Yes]** if you want to turn off the purge gas. If work is only interrupted for a short period (up to 30 min) do not turn off the purge gas. This will save time during the ignition process until the spectrometer is sufficiently purged.

- ▶ Wait for the message to appear that the device and the cooling can be switched off.
- ▶ Switch off the ICP-OES device and the autosampler, if applicable, from their respective device switches.
- ▶ Release the pump hoses at the ICP-OES device.
  - – Release the pressure levers to ensure that the hose guide no longer exerts pressure on the hoses.
  - – Pull the hose stoppers on the right-hand side of the pump from their lock.
- ▶ In case the automatic sampler is used, release the pump hose in the same way as with the hose pump on the ICP-OES device.
- ▶ Close the gas supply after switching off the devices.
- ▶ Switch off the recirculating chiller at the mains switch.
- ▶ Switch off the exhaust unit.
- ▶ Shut down Windows and switch off the PC.
  - ✓ The analyzer is now turned off.

Note

Wait for the ICP-OES device to cool down before switching it off!

After extinguishing the plasma you should wait for at least 30 s before you switch off the device with the power switch.

6.3 Start analysis

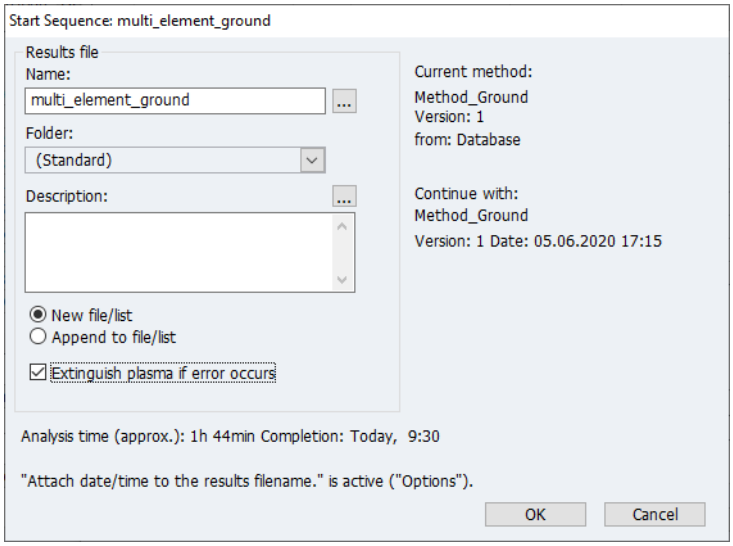
After you have selected the method, the sequence and possibly the sample information data, the system has all information necessary to start the analysis process.

The device must have been prepared for the measurement:

- The plasma has been ignited and burns for the run-in time required for the method.
- When using the autosampler: The samples are ready on the autosampler.

Saving result data during the analysis process

The results of the analysis are saved to a database in the default folder or a user-defined subfolder directly during the measurement. They may be saved optionally to a new database or added to an existing database. However, it is not possible to overwrite a result database by selection of the same name.



Window Sequence measurement start

The target for result storage will be requested automatically at the start of a measurement routine. The related window **Start sequence** opens with the following options for the results file:

Option	Description	
Name	Enter the desired name for the result database.	
	New file/list	If enabled, a new file name must be entered. The program checks if the file name exists already. Existing files cannot be overwritten.
	Append to file/list	New results will be appended to the existing results file. With **[...]** open the selection dialog. Choose an existing results file from the displayed list.
Folder	Choose the save path for the results file.	
Description	Here, you may enter an additional comment that is saved along with the analysis results. The entry is optional.	
Extinguish plasma if error occurs	Extinguishes the plasma if the measurement is canceled with an error message.	

The file contains the results of measurement and evaluation and the sample ID information. In addition the method parameters are saved in the result database.

The results database is saved with the extensions ".tps" (method parameters, intensities and concentrations) and ".spk" (spectral raw data).

Start the measurement

- ▶ Start the measurement routine by clicking on or with the menu item **Routine | Start Sequence**.
- ▶ In the **Start Sequence** window, select a file name for the results file.

  You may optionally save the result to a new file or append it to an existing file. Overwriting of an existing file is not possible.

After you selected the file name, the measurement routine will start according to the settings selected in the method and the sequence.

If you use an autosampler, the measurement runs automatically.

- In the case of manual sample feed without autosampler, follow the prompts for the provision of samples displayed on the screen.

Displays during the analysis process

While the measurement is running, the results are displayed in real time in the main window. In the sequence list of the main window, the measurement progress is logged. The rows with the successive actions are marked by the following symbols in the table column:

Icon	Meaning
-	Not measured/executed yet.
○	Just being measured.
+	Already measured/executed.

Displaying the results window

In addition the windows **Signal plot**, **Spectrum plot**, **Bar graph**, **Report window** and **Sample conc. in calibration curve** can optionally be shown with the current result. These display windows are selected in the window **Options / Analysis sequence** (→ "Options for analysis sequence" p. 138). The results windows can be shown and hidden during the analysis.

- To open the windows use the menu command **View | Open Results Window** or the function key F7.
- To hide the windows use the menu command **View | Close Results Window** or the function key F8.
- With  the window can also be opened during the analysis.

Buttons in the icon bar

Larger buttons are shown in the icon bar during the measurement:

Button	Description
 **Results windows**	Opens the window **Results display** where the individual results windows can be enabled independent of the entries in the window **Options / Analysis sequence**. Enable the options of the results windows and release the windows by clicking on **[Results display]**.
 **Show method**	Turns the method window on. A method can only be read, it cannot be changed.
 **Sequence samples**	Turns the sequence window on. The sequence can be expanded during the running analysis. The sequence window includes the link **[Samples]** to open the window **Sample ID** for amending the sample ID data.

6.4 Interrupting/continuing the analysis sequence

Analysis processes can be interrupted and continued later.

- With the menu item **Routine | Stop** or  the analysis sequence is stopped immediately.
- With **Routine | Continue** or  an interrupted routine is continued.

  The window **Continue sequence** opens where the action status prior to the interruption is output.

  To change the method enable the option **Continue with modified method**. This results in a new method entry in the results file and another version of the method is saved.

The measurement can be continued as follows:

Option	Description
Continue	Repeat current sample, current line and current statistical run.
First statistical run	Continue with current sample, current line and the first statistical run.
First element	Continue with current sample, first line and first statistical run.
From table row	Continue with the table row displayed in the text box.

6.5 Repeat the actions of the sequence

Single actions in a sequence can be repeated.

- In the main window on the **Sequence** or **Sequence/Results** tab select the row(s) with the action to be repeated.
  Multiple selections are made by clicking on the respective rows with the Ctrl or Shift keys held down.
- Start the measurement routine by clicking on  or with the menu item **Routine | Run Selected Sequence Row**.
- In the **Start Sequence** window, select a file name to which the result of the repeated measurement shall be saved.

  You may optionally save the result to a new file or append it to an existing file. Overwriting of existing results by selection of the same file name is not possible.

  Next the repetition of the selected action will start.

Note

If changes have been made to the method in the past, the changed method will be used when repeating a sequence or single lines and saved as a new version with the results.

6.6 Reprocessing analysis results

The reprocessing of analysis results is used for changes in the analysis conditions, e.g. change of the calibration function or method changes, to take effect in the analysis. A

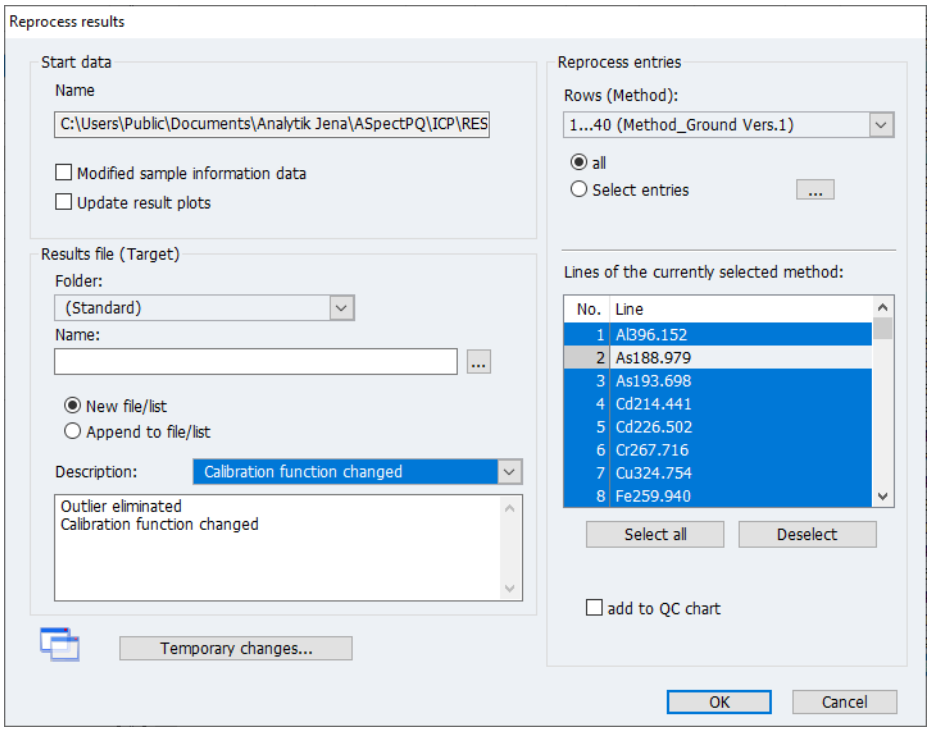
change of sample information data, e.g. sample name, dilution factors, also requires a reprocessing to take account of it in the output of the analysis results.

The reprocessed data can optionally be appended to the current result file or saved to a new file. Manipulation of the original data is ruled out. If in a result file the reprocessing is repeated several times with different parameters, each reprocessing refers to the original data of the result file.

Note

With every reprocessing a new method version is saved.

Input options in the window Reprocess results



Window Reprocess result

Option / Field	Description	
Start data	Selection of the input data	
	Name	Display of the name of the results file whose data are reprocessed
	Modified sample information data	To be enabled if data in the sample information file, e.g. the dilution factor, have been changed
	Update results plots	The results windows, e.g. **Spectr plot,** are updated as for the measurement. **Note:** This slows down the reprocessing.

Results file (Target)	Select the location for saving the reprocessed results data.	
	New file/list	Save results data in a new file For the results file select under **Folder** and **Name** the storage location for the calculated data. The remarks entered under **Description** are saved with the results data.
	Append to file/list	The reprocessed data are appended to the existing results file.
Reprocess entries	Select the rows for reprocessing.	
	all	Reprocess all entries in the results list.
	Selected entries	Only reprocess selected sequence rows. In ••• and in the window **Select entries** select all sequence rows to be reprocessed.
	Lines of the currently selected method	In the list select all lines to be reprocessed. **[Select all]** selects all lines. **[Cancel selection]** removes all selections in the line list.
[Temporary changes]	Save temporary changes for reprocessing (wavelength offsets, deletion markers) (file ending ".rep"). The data are subsequently loaded automatically with the corresponding results file (of the same name).	
Add to QC chart	When active, QC sample type results are entered into QC charts during reprocessing (→ "Specifying quality control samples for QC tabs - QCS tab", p. 46).	

Reprocessing data

- ▶ Changes are made in the method parameters or in the window **Sample ID**.
- ▶ Click on or select the menu item **Routine | Reprocess**.

  The **Reprocess results** window opens.
- ▶ Specify the input data (Name, modified sample ID and update results plot), the storage location and the name of the target file.
  Note: If you are recalculating because of changes in the sample information, enable the option **Modified sample information data**. Otherwise, these changes will not be considered.
- ▶ Select the rows/lines for reprocessing.
- ▶ Start reprocessing with **[OK]**. If no target file has been specified, the query "Reprocess data without saving to a permanent file?" appears.

Replacing a calibration standard

An existing calibration standard can be replaced by one that has been measured later. To do this, proceed as follows:

- ▶ In the main window select the row of the calibration standard to be replaced on the tab **Sequence** or **Sequence/Results**.
- ▶ Start the measurement of the sequence row with a click on .
- ▶ In the window **Start sequence** define for the result to be appended to the already existing file.

  Next, the measurement of the calibration standard starts.

- In the results list right click on the standard you want to replace. In the context menu select the item **Sample single values**.
- In the window **Individual sample values** enable the checkbox **Replace with entry number** and enter the row number of the standard to be replaced into the input field.
- Start reprocessing as described above.

  The data are reprocessed for the selected rows.

6.7 Evaluating measurements parallel to running analyses (offline mode)

While measurements are running, it is impossible to evaluate results in the same program instance. However, it is possible to start a second program instance of the application in offline mode, while measurements are running in the first instance. In this mode, there is no communication with the device. However, all other functions such as developing methods or loading and analyzing results can be used parallel to the running measurements of the first program instance.

- Start ASpect PQ in the second program instance using the menu item **File | Start Offline Program Instance**.
- Open the results file of the currently running measurement with the menu item **File | Open Results**.

  The results measured so far are loaded to the results window.
- Additional results from the running measurement are loaded by clicking on  in the toolbar or with the menu item **View | Update Result List**.

The results can be edited further.

Note

In reprocessing, the recalculated results are saved to a new database. It is not possible to access the original results file.

6.8 Displaying results and analysis progress in the main window

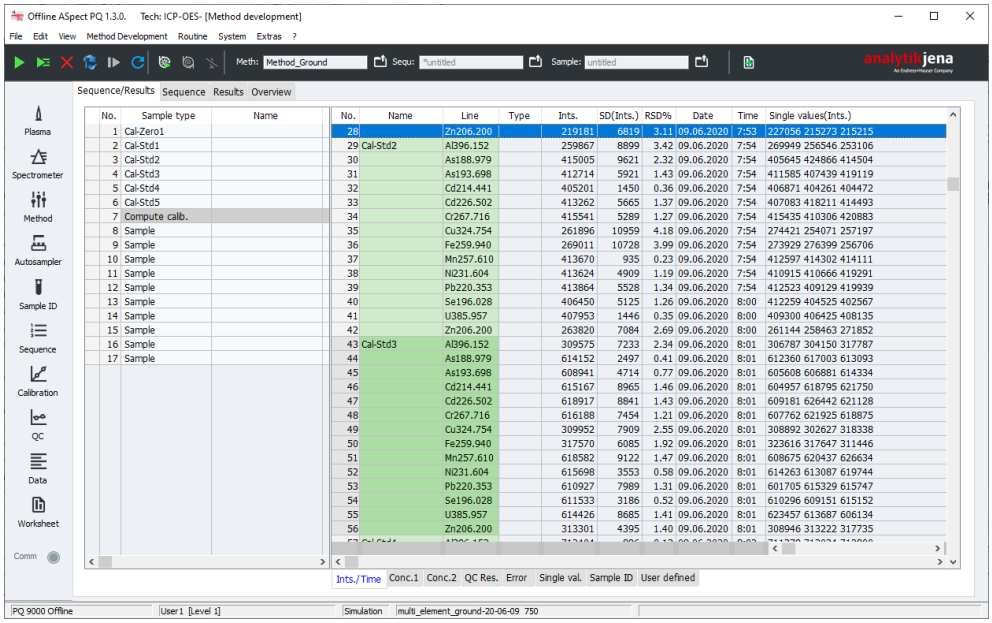
The measurement results and the sequence are extensively displayed in the main window in the background of the workplace.

The presentation on different tabs in the main window provides a good overview of measurement results and statistical analyses.

The following tabs are selectable:

- **Sequence/Results** (content of the **Sequence** and **Results** tabs on one tab)
- **Sequence** (display of the current sequence)
- **Results** (display of the measurement results)
- **Overview** (summary of measurement results)
- **User-defined** (user-defined results display)

The status bar of the result window shows the file name of the current results file.



Main window of Aspect PQ with results

6.8.1 Sequence/Results tab

The **Sequence/Results** tab contains the data of the two tabs **Sequence** and **Result** (→ "Sequence tab" p. 73 and "Results tab" p. 73).

6.8.2 Sequence tab

On the **Sequence** tab, the active sequence is listed.

On this tab, you can follow the progress of the running analysis. The various samples and special functions are marked in the first column of the table as follows:

Icon	Meaning
-	Not measured/executed yet.
○	Just being measured.
+	Already measured/executed.

Note

After the measurement, you can remeasure a selected sample. To this end, the sample row in the sequence must have been selected and then pressed in the toolbar.

6.8.3 Results tab

The **Results** tab lists all measurement results and statistical analyses. The values are split up in further tables for a clear presentation. The index tabs for these tables are arranged at the bottom edge of the window.

The values are sorted by the order of sample measurement. For every sample, the analyzed elements are listed.

Table Ints./Time

The table contains the intensities and the statistical analyses according to the selected method options (**Method / QCC** window).

Column	Description
no.	Number in analysis sequence.
Name	Name of the sample, standard or QC sample/standard
Line	Element line
Type	Internal standard or analyte
Ints.	Mean value of the measured individual intensities of the sample
SD(Ints.)	Standard deviation (mean value statistics)
RSD%	Relative standard deviation (mean value statistics)
Date/Time	Time of the measurement
Single values	Individual values of the intensity measurements

Table Conc.1

The **Conc.1** table shows the analyzed concentrations of the sample as supplied to the ICP-OES device. The unit used is the calibration unit set in the method.

Column	Description
no.	Number in analysis sequence.
Name	Name of the sample, standard or QC sample/standard
Line	Element line
Type	Internal standard or analyte
Unit	Concentration unit
Conc.1	Analyte concentration in the sample / analyte concentration in the standard
SD1	Standard deviation of the conc. 1 (mean value statistics)
RSD%	Relative standard deviation of the conc. 1 (mean value statistics)
R	Range of the conc. 1 (median statistics)
R%	Relative range of the conc. 1 (median statistics)
CS	Confidence interval
DF	Predilution factor of the sample Factor by which the original sample was diluted before being placed on the autosampler or supplied to the plasma, if no autosampler is used.
Rem.	Remarks (→ "Overview of markings used in the display of values" pg. 155).
Ints.	Mean value of the measured individual intensities of the repeat measurements
SD(Ints.)	Standard deviation of the intensity (mean value statistics)
Date/Time	Date and time of measurement
Single values (Ints.)	Individual values of the intensities of the repeat measurements

Table Conc.2

The **Conc.2** table shows the concentrations of the original sample. In calculating Conc.2, the sample information data are considered:

- Pre-dilution
- Weighed portion of solids

- Conversion factors for other units

Column	Description
no.	Number in analysis sequence.
Name	Name of the sample, standard or QC sample/standard
Line	Element line
Type	Internal standard or analyte
Unit	Concentration unit
Conc.2	Concentration of original sample taking sample information data into account
100% norm.	Conc. 2 normalized to the percentage of the total concentration.
SD2	Standard deviation of the conc. 2 (mean value statistics)
RSD%	Relative standard deviation of the conc. 2 (mean value statistics)
Cf	Confidence interval of Conc. 2
Ints.	Mean value from the determined individual intensities
SD(Ints.)	Standard deviation of the intensity (mean value statistics)
R(Ints.)	Range of the intensity (median statistics)
Date/Time	Date and time of measurement
single values (Ints.)	Individual values of the intensity measurements

Table QC Res

In the table **QC Res.** the results of the QC samples are output:

- Target value and actual value of the concentration
- Recovery rates (all types except blank value)
- Reactions to any deviations (all types except blank value).

Column	Description	
no.	Number in analysis sequence.	
Name	Name of the sample, standard or QC sample/standard	
Line	Element line	
Type	Internal standard or analyte	
QC (for calibration functions)	**R2(adj.)** or **R (**→ "Options for analysis sequence" pg. 138) **Slope** **BEC**	**B**ackground equivalent concentration
QC (for QC samples, not for QC blank)	**Conc.1** **Rated value** **Recovery rate**	With QC samples and QC Std., the recovery rate of concentration is determined. With QC-Stock, QC-Trend and QC-Matrix, the recovery rate of the concentration increase caused by additions is determined.

QC (for blank detection limit)	**SD**	Standard deviation of blank measurements
	CRDL	Detection limit
	LOQ	Limit of quantitation
Rem.	Remarks (→ "Overview of markings used in the display of values" pg. 155).	
Ints.	Mean value of the measured individual intensities of the repeat measurements	
SD(Inst.)	Standard deviation of the intensity (mean value statistics)	
Date/Time	Date and time of measurement	
Single values (Ints.)	Individual values of the intensity measurements	

Table Error

If errors occur during the measurements, the corresponding measurements are marked in red in all tables. In the **Error** table, the respective measuring error including error number is documented in writing.

Table Single val.

The table **Single val.** contains the measured individual values of the intensity and the corresponding background intensity.

Table Sample ID

The table Sample ID contains the sample information data (→ "Specifying information data for samples and QC samples" p. 61).

Column	Description
no.	Number in analysis sequence.
Name	Name of the sample, standard or QC sample/standard
Line	Element line
Item	Position of sample on autosampler
Pre-DF	Pre-dilution factor Factor by which the original sample was diluted before being placed on the autosampler or supplied to the spectrometer, if no autosampler is used. The factor is required for the calculation of the concentration of the original sample.
Wt.[g]	Weighed portion in gram The mass of the original sample in gram which was dissolved in sample preparation (in mg). The mass is required for the calculation of the concentration of the original sample (**Conc.2**).
Vol.[ml]	Volume of solvent used to dilute the weighed sample portion (in mL). This value is required for the calculation of the concentration of the original sample (**Conc.2**).
Total wt. [g]	Total weighed portion (sample and diluent) (only for the unit type **liquid gravimetric**).
Name (2)	Additional sample name from the sample information table
AS-DF	Dilution factor of the autosampler

Blank corr.	Blank value correction	
	off	No blank value correction took place.
	on	For the calculation of the concentration of the original sample, the blank measured last in the sequence will be subtracted.

Table User-defined

On the tab **User defined** you can directly select the parameters for the results output and their order in the table.

- Click on the button **[Select columns]** in the bottom right corner of the table.
- In the window **Select columns** select the desired parameters by mouse click.
- To change the order in the display, select the parameter whose position you want to change and move it with the keys and in the list.
- After returning to the main window the results are displayed. You can change the width of the table columns by moving the mouse pointer to the table line in the table header (the pointer changes to a double arrow) and moving the table column with the mouse button held down to the desired width.

Note:

The column width is saved in this view. For the other tables in the main window changes of the column width are reset after exiting.

6.8.4 Overview tab

On the **Overview** tab, the analysis results are summarized. You may choose among various presentation options:

Value	Description
Conc. 1	Concentration 1
Conc. 1 (RSD%)	Concentration 1 (relative standard deviation)
Conc. 2	Concentration 2
Conc. 2 (RSD%)	Concentration 2 (relative standard deviation)
Ints.	Intensity
Ints. (RSD%)	Intensity (relative standard deviation)
Ints.(R%)	Lintensity (relative range)
LOD	Limit of Detection
LOQ	Limit of Quantitation
Recovery(Nominal val.)	Recovery rate (target value)
R^2(adjust)/Recal. factor	Coefficient of determination / recalibration factor
100 % norm.	Conc. 2 normalized to the percentage of the total concentration

By activation of the respective check boxes, the following sample types can be displayed:

- Samples

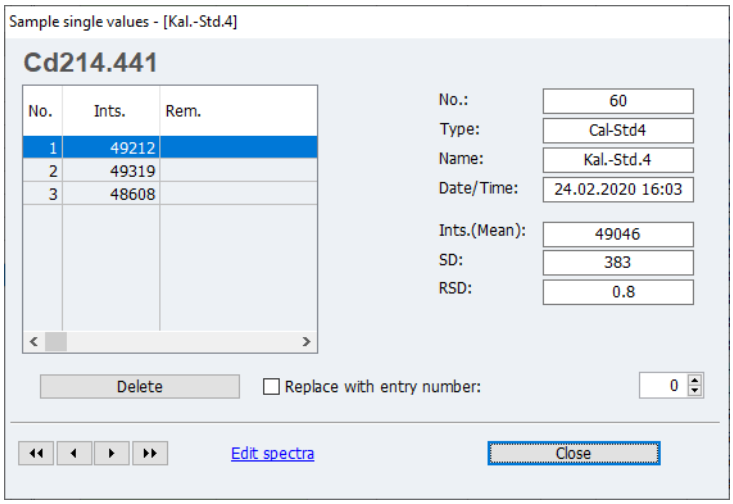
- QC samples
- Cal.-Std.
- Others

With  open the **Print Overview** window from where you can start the printout of the data displayed in the current overview (→ "Print functions in ASpect PQ" p. 117).

6.9 Displaying and editing individual sample values (window Sample single values)

▶ Right click on the row in the results table and select the item **Sample single values** from the context menu.

Alternatively select the sample row and select the menu command **View | Sample single values**.



Window Sample single values

Single value display (table)

The individual sample values are shown in the table.

Table column	Description	
no.	Number of single value within the sample measurement	
Ints.	Intensity of the single value	
Conc. 1	Concentration of the analyte in the analyzed sample.	
Rem.	**none**	The single value enters the calculation of the mean sample value.
	#MAN.	The value was manually excluded from the calculation of the sample value
	#KOR.	The value was automatically excluded from the calculation of the sample value due to the Grubbs outlier test.

Sample data

Field	Description
No.	Number of measurement in the result table
Type	sample type (sample, standard or QC sample type)
Name	Sample name
Date/Time	Date and time of the measurement selected in the table
Ints.(Mean)	Intensity averaged for all single values
SD	Standard deviation (mean value statistics). This parameter is displayed independently of the statistical method chosen for the measurement (mean/median).
RSD	Relative standard deviation (mean value statistics) This parameter is displayed independently of the statistical method chosen for the measurement (mean/median).

Additional buttons and options in the window Single sample values

Option / buttons	Description
[Delete] / [React.]	Exclude the single value from average calculation or reinclude it.
[Edit spectra]	Display measured wavelength-dependent line spectra (→ "Displaying and editing intensity spectra" p. 79).
replace with entry number	Only for calibration standards The current sample is to be replaced during reprocessing by a sample at position **no**. in the results table.
◂◂ ◂ ▸ ▸▸	Change between the lines of individual samples and from one sample to the next in the results table.

Excluding single sample values

If desired, you may manually exclude a single value from the calculation of the sample average.

- ▶ To this end, mark the single value to be excluded in the table.
- ▶ Click on **[Delete]** to exclude the value from the calculation of the sample average for result reprocessing.
- ▶ To reinclude the previously excluded single value in average calculation, click on **[React]**.

Note

By activating the Grubbs outlier test option, outliers among single values can be detected and eliminated automatically during the analysis.

6.10 Displaying and editing intensity spectra (window Edit spectra)

The display of intensity spectra is used for the following tasks:

- Calculate the main peak of an analysis line and save it in the line file
- Calculate the background correction with consideration of the sample matrix and transfer it to the method
- Create spectral corrections

- Identify lines adjacent to the analysis line

The intensity spectra can be displayed and edited for each measurement in the results window.

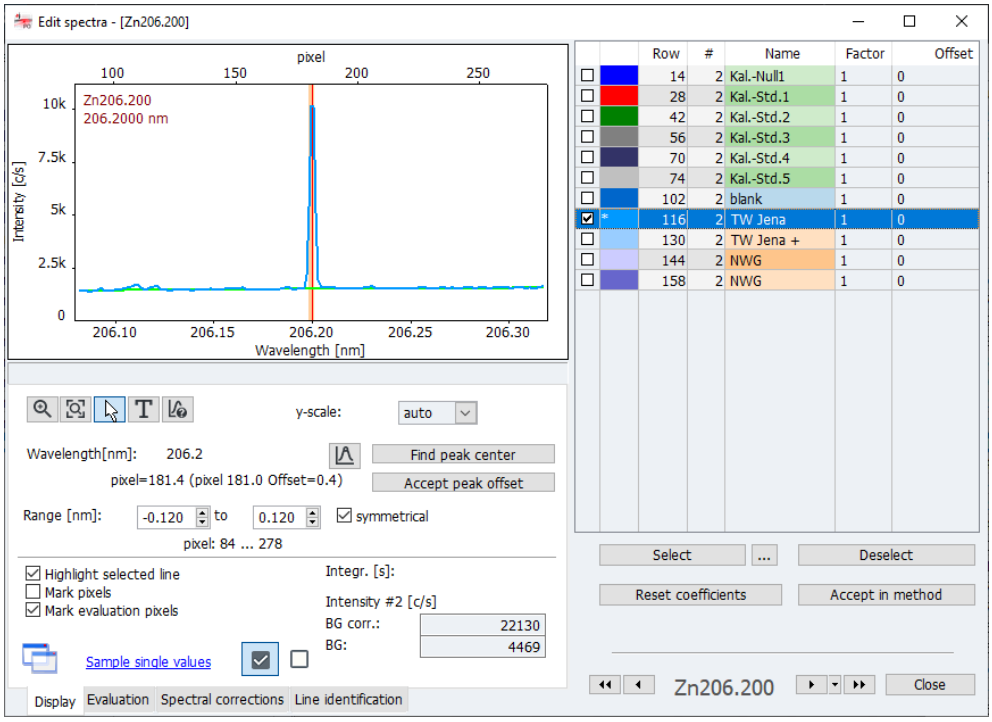
▶ Open the **Edit spectra** window by double click on the corresponding sample row in the results table.

Alternatively, right-click on the row of interest of the result table. In the resulting context menu click on **Edit spectra** or select the sample row and select the menu command **View | Edit spectra**.

In the **Edit spectra** window all measured samples with all single values are listed for one analysis line at a time. It is possible to change between the individual analysis lines.

The left side of the **Edit Spectra** window contains the graph of the intensity spectrum of the selected sample(s) and four tabs for the analysis and editing of the spectrum. On the right side the individual sample values to be displayed are selected from the overview.

6.10.1 Display spectra – Display tab



Window Edit spectra / Display

Selecting spectra / Sample table

The table on the right side lists all individual sample values of the analysis line.

▶ Enable the checkboxes of the single values you want to display in the graph.

The spectra of the individual sample values are shown as overlaid. The color of the field at the front in the table is assigned to the individual spectra.

▶ The single sample selected by the mouse (blue bar in the table) is highlighted in bold in the graph if the option **Highlight selected line** in the bottom left corner of the window has been enabled.

You can filter the display of the samples/repeat measurements in the sample list and the selection for the graphical display of spectra (enable the checkbox in the sample list) using the buttons under the table:

- Next to **[Select]** click on •••.
- In the **Selection** window make the following settings:

Option	Description
all	Select all rows of the result list in the main window for the graphical display (enable the checkbox for the graphical display).
from/to	Select all rows of the result list in the main window for the graphical display (enable the checkbox for the graphical display).
Replicate	Only select the spectra in the result list between set rows from/to.
show selected replicate(s) only	Select individual sample values of a sample: **all** Select all individual sample values of a sample. Reference number, e.g. "2." Only select the selected individual value of a sample.

- By clicking on **[Select]** you make the spectra selection with the parameters set above.
- **[Deselect]** disables all checkboxes.

Entry of factor and offset

- For every spectrum a factor and/or an offset can be entered into the table. A spectrum manipulated in this manner is spread out/compressed and moved along the x-axis.
- By clicking on **[Reset coefficients]** the factor and offset are reset again, and the spectrum returned to its original state.

Display of the line spectra

The selected spectra are displayed on the left. The intensity is plotted in [c/s] against the wavelength in [nm]. At the top margin of the graph the pixel allocation is shown. The spectrometer is adjusted to map the main peak to the measuring pixel, e.g. 180. The main peak offset must be corrected for each analysis line, see below.

The buttons for the spectral view have the following functions:

Option/button	Description
	Enable graphic zoom. After clicking select the spectral section to be enlarged with the left mouse button held down.
	After zooming restore the original coordinates.
	Enable the selection mode in signal or spectral plots. The measuring points are selected with the left mouse button. The values of the selected measuring point are displayed in the output field below the buttons.
	Enable text mode. Keeping the left mouse button pressed you can draw a frame and enter text that shall be added to the graph. A double click on the existing text opens the window for editing or deleting the text. Holding the Ctrl-key and the right mouse button depressed, you can shift existing text across the graph.

	Enable line identification mode. By clicking or dragging with the mouse element lines at the selected wavelength position are searched for in a line database. The found line is displayed below the graph (→ "Finding lines – Line identification " p. 87).
y-scale	Select the scaling of the graph: **auto.** Automatic scaling: The spectrum is displayed with optimum ordinate expansion. **value** Manual scaling. The upper ordinate limit must be selected in the list.
Wavelength	Display the wavelength of the analysis line.
	Set the main peak manually.
[find peak center]	Automatically search for the peak and correct the offset.
[Accept peak offset]	Save the peak offset in the line library. The offset will be used from this time onwards for each measurement of this element line.
Range [nm]	Select the wavelength range below and above the analysis line. This wavelength range is available for spectral analysis, e.g. background correction. If the checkbox **symmetrical** has been enabled, the wavelength range below and above the wavelength is identical. The corresponding pixel range is displayed below the input fields. The settings for the wavelength range of the selected line are transferred to the current measuring method by clicking on **[Transfer to Method]**. This area is used for dynamic background adjustment (or automatic background correction) for calculation. The data are also changed in the **Method** window on the **Evaluation** tab.
[Highlight selected line]	The single spectrum selected in the right overview is highlighted with a bold line in the graph.
[Mark pixel]	Pixels are identified with a circle in the spectral graph.
[Mark evaluation pixel]	The central analysis pixel at the main peak is highlighted with a red line. If several pixels are used for the analysis, their range is highlighted in light red.
Display of intensities	**BG corr.** background corrected intensity **BG** intensity of the background
Sample single values	Link to the window **Sample single values**
	If the icon has been highlighted in this manner, the line is used in the method (→ "Selection of analysis lines – Lines tab" pg. 26. You can select suitable lines in this manner during the method development in the window **Edit spectra**.
	Do not use line in the method.

Automatically setting the main peak

During the method development you need to correct device-related peak offsets and offsets caused by line interference, e.g. duplicates.

▶ Click on **[Find peak center].** The automatic determination of the main peak is well suited for determining most peaks.

Alternatively, enable and select the main peak manually in the spectrum.

▶ Optionally you can recalculate the results to evaluate the new peak position.

– Change to the results window and click on .

– Proceed as described in "Reprocessing analysis results" p. 69.

▶ Save the peak offset found with **[Accept peak offset]** to the line/wavelength file of the device.

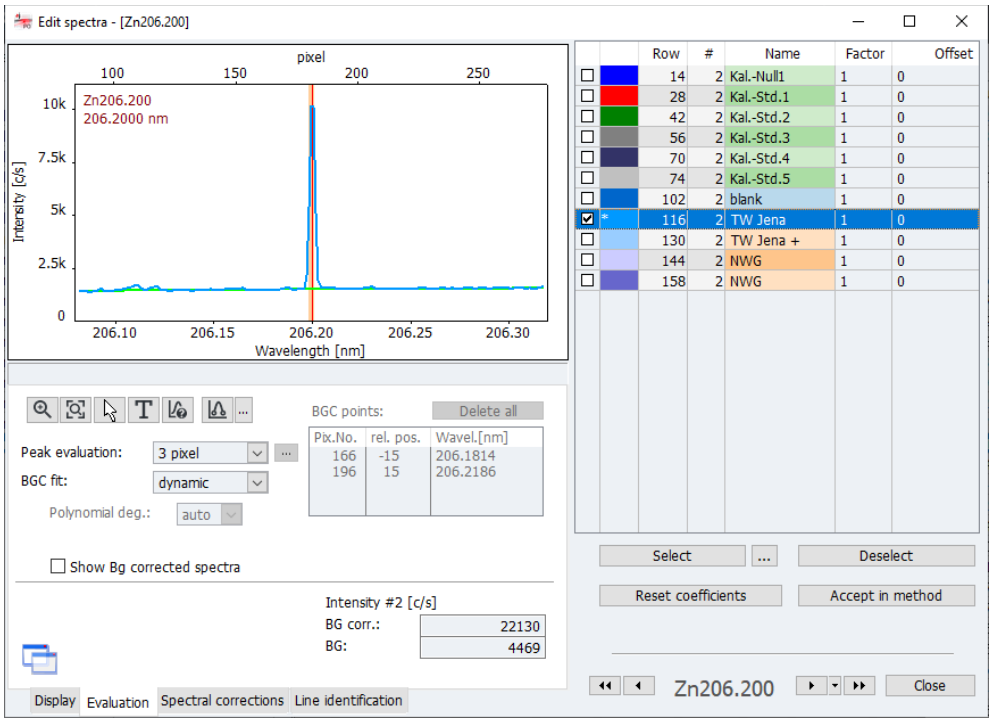
✓ The data are now available for every subsequent analysis of the analysis line.

6.10.2 Evaluating spectra and determining the background correction – Evaluation tab

Continuous background emissions causing intensity fluctuations across a wide spectral range around an analysis line can be compensated using background correction. Pixels (background corrections points) are selected on both sides of the analysis line, a regression is calculated through the points and the regression graph used for background correction.

In the static method for selecting the background correction points the points are set manually and the polynomial degree of the regression graph determined independently. In the dynamic method the regression graph is automatically calculated using the ABC algorithm (ABD = automatic baseline correction).

A discontinuous background interference, e.g. by line overlap with a matrix element, can be minimized with the aid of correction spectra (→ "Removing spectral interference – Spectral corrections " p. 85).



Window Edit spectra / Evaluation

Element overview for peak evaluation and background correction

The buttons for the spectral view, some value outputs and the selection of the single sample values have been described in section "Display spectra – Display tab" p. 80".

Option/ button	Description	
Peak evaluation	Set the number of pixels for the peak evaluation.	
	1	The measuring signal is only determined at the pixel at which the main peak is located (→ "Display spectra – Display " p. 80).
	value > 1	Number of pixels across which the measuring signal is determined. The individual signals of the pixels are totaled. The result is therefore greater than the maximum peak. The pixel with the main peak is located in the center of the range.
	Height	The peak height is used for evaluation.
	User defined	The evaluation range is defined by the user. This is the preferred option for the evaluation of duplicates. After clicking on ••• enable all pixels in the list that are used for the evaluation.
BGC fit	Select the type of background correction:	
	dynamic	The background correction is automatically calculated using a mathematical algorithm. No other settings are required for this option.
	static	The background correction points are set manually via mouse click in the spectrum. For the correction function the polynomial degree must additionally be selected.
	For static adaptation set or delete the background correction points. A cross is shown when moving the mouse over the spectral graph. Clicking on the arrow opens the function list:	
	Set background correction points	Set the correction points to the desired wavelength on the spectrum with a mouse click. If you move over a range with the mouse button held down, select the entire range.
	Delete background correction points	Clicking on an already selected point deletes the respective background correction point. Ranges can be deleted by dragging the mouse.
	Delete all background correction points	Deletes all selected points.
BGC points: [Delete all]	Delete all manually set background correction points.	
Table	Displays the manually set background correction points.	
Polynomial deg.	Select the polynomial degree for the regression of the background correction graph. For the option **auto** the regression is selected automatically.	

Mark BGC points	Select background correction points with a square.
Show BG corrected spectra	Show background-corrected spectra. The adapted background (green line) is subtracted from the sample spectrum. The background then corresponds to the zero line.

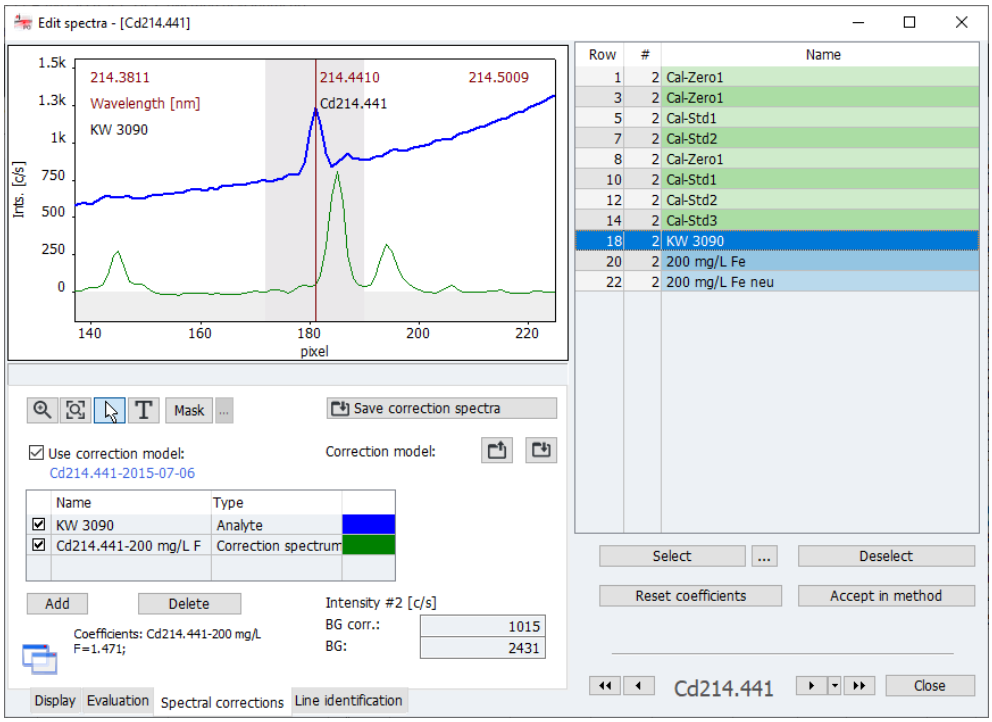
Transferring data to the method

Transfer the settings for the peak evaluation and for background correction for the selected line to the current measurement method by clicking on **[Accept in Method]**. The data are modified on the **Evaluation** tab in the method window.

6.10.3 Removing spectral interference – Spectral corrections tab

During the routine it is attempted to select lines for the analysis that are without interference and/or feature an easily corrected. Where this is not possible, the discontinuous interference caused e.g. by line overlap with one or several matrix elements can be removed with the aid of correction spectra. The correction spectra for each matrix are combined in a model and can then be linked to the line in the method.

The functions for saving the individual correction spectra and combining the correction model are available in the window **Edit spectra / Spectral correction**.



Window Edit spectra / Spectral corrections

Option/button	Description
[Save correction spectra]	Save spectra of the pure components of a matrix as correction spectra.
Use correction model	If enabled the correction model is applied to the analytes.

Correction model		Save the current correction model.
		Load an existing correction model.

The line table lists the analyte and the correction spectra used in the model. By enabling the checkboxes the individual spectra are displayed in the diagram. With [Add] further spectra are added to the correction model. With [Delete] the spectrum selected with the mouse is deleted from the model.

Note:
All correction spectra in the line table are used for the calculation irrespective of whether the checkbox for their display has been enabled or not. If a correction spectrum is to be excluded, it must be deleted.

6.10.3.1 Creating a correction model for spectral corrections

To create and use a correction model for an analysis line, you must carry out the following steps:

1. Identify possible interferences
2. Create and save the correction spectra
3. Create a correction model.
4. Transfer the parameters of the analysis line with correction model into the method.

1st step: Identifying possible interferences

- ▶ Create a method with the analysis line.
- ▶ Measure the analyte in the matrix and load the spectrum in the window **Edit spectra** (double click on the sample row in the main window).
- ▶ In the window **Edit spectra /Line identification** identify the possible interference lines.

2nd step: Measuring the correction spectra

- ▶ Add to the sequence the measurement of the interfering matrix components that cause spectral superposition and measure these components in single element solutions.

  Note:
  The concentrations of the matrix components do not need to match those in the samples but must be at least high enough for the spectra to have clear intensity values. For a correct spectra correction only measure one component at a time as a pure substance.
- ▶ Load a spectrum of a matrix component to the window **Edit spectra / Spectral correction.**
- ▶ Click on [**Save correction data]**.

  The database window for saving the correction spectra opens.
- ▶ Issue a name and finish the process with **[Save]**.
- ▶ Save the spectra of the other matrix components in the same manner.

3th step: Creating a correction model

- Load the spectrum of the analyte in the matrix again.
- Enable the checkbox **Use correction model**.
- With **[Add]** open the selection of the already saved correction spectra.
- Select a correction spectrum in the list and click on **[Load]**.
- Add all correction spectra in this manner.
- Check in the spectral view whether the resulting sample spectrum is not free of overlaps.
- Using the **[Mask]** button you can mask ranges with the mouse button held down that are not to be included in the calculation of the correction model.
  By default the range of the analysis line (± 9 pixels) is masked. It might be necessary to mask additional ranges if no pure substances were available for recording and these contaminations might be present in varying proportions.
- To save the correction model click on  and issue a name for the model. Finish the process with **[Save]**.

4th step: Transferring the analysis line with correction model to the method

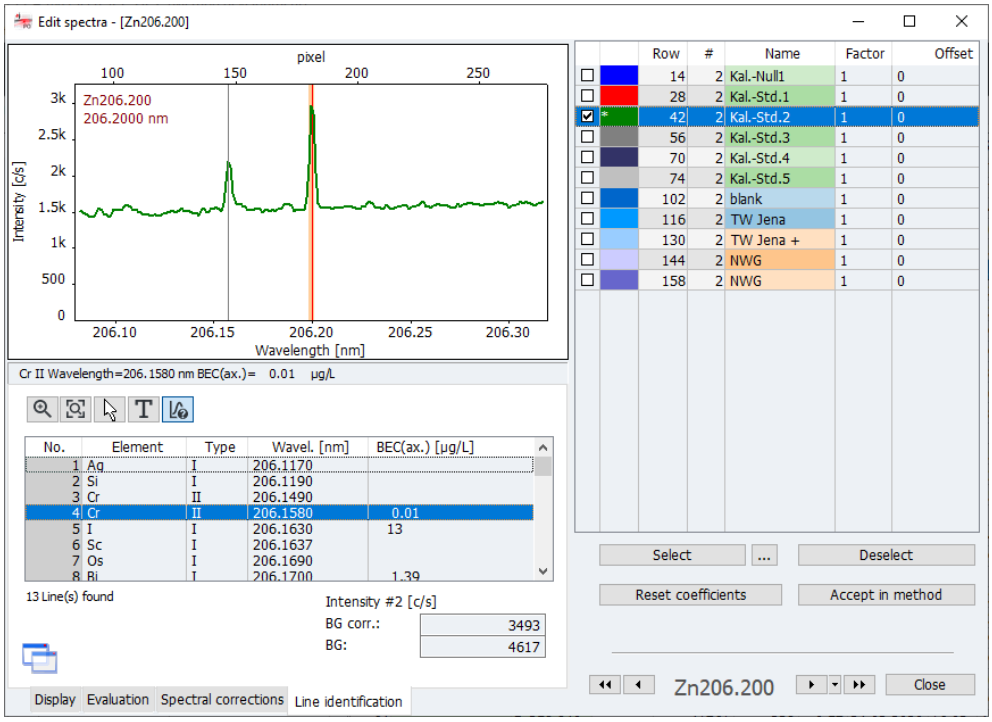
- Transfer the parameters or the analysis line with the correction model to the current method with **[Accept in method]**.
  - ✓ In the **Method / Evaluation** window the analysis line is identified with **LSM** (Least Square Model) in the **Correction** column.

After saving the method the future measurements will be performed with this method with the created correction model. Already completed measurements can be reprocesses with the new method version making a new measurement unnecessary.

The spectral correction models and correction spectra are saved with the results data. If the results data are transferred to a different computer on which the correction models have not been saved, the models are imported after a query.

6.10.4 Finding lines – Line identification tab

Lines in the measured spectra are identified based on the line database.



Window Edit spectra / Line identification

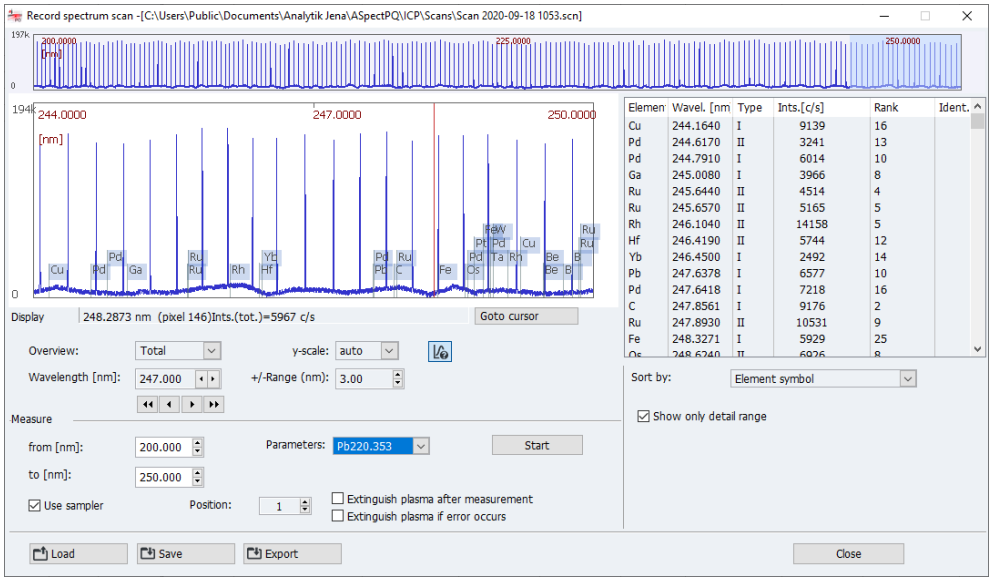
In the table below the spectrum all lines identified in the spectral section are displayed.

- ▶ Enable the button .
- ▶ Click on the peaks of interest in the spectrum.

  The nearest line is displayed below the spectrum and highlighted in the table.
- ▶ Inversely, you can also select a line in the table which is then displayed in the spectrum.

6.11 Recording an overview spectrum

With the menu item **Method development | Overview Scan** you can record an overview spectrum in a specified wavelength range.



Window Record spectrum scan

- ▶ Select the menu item **Method development | Record overview spectrum**.
- ▶ Enter the desired wavelength range (**from / to**) in the **Measure** area.
- ▶ If you have activated a method, you can select the parameters of a line of the method for the spectrum scan. If no method is loaded, preset parameters are used.
- ▶ Prepare the sample. If you want to work with an autosampler, activate the option **Use autosampler** and select the position of the sample on the autosampler.
- ▶ Start the scan with **[Start]**.
  - ✓ When the scan is finished, the overview spectrum is displayed in the upper part of the window.
- ▶ If you click on a section in the overview spectrum, a detail area with the selected line is displayed in the graphic. You set the width of the detail area in the +/-range list.
- ▶ The lines found are displayed in the table on the right side. You can limit the display to the shown spectral range with the option **Show only detail range**.

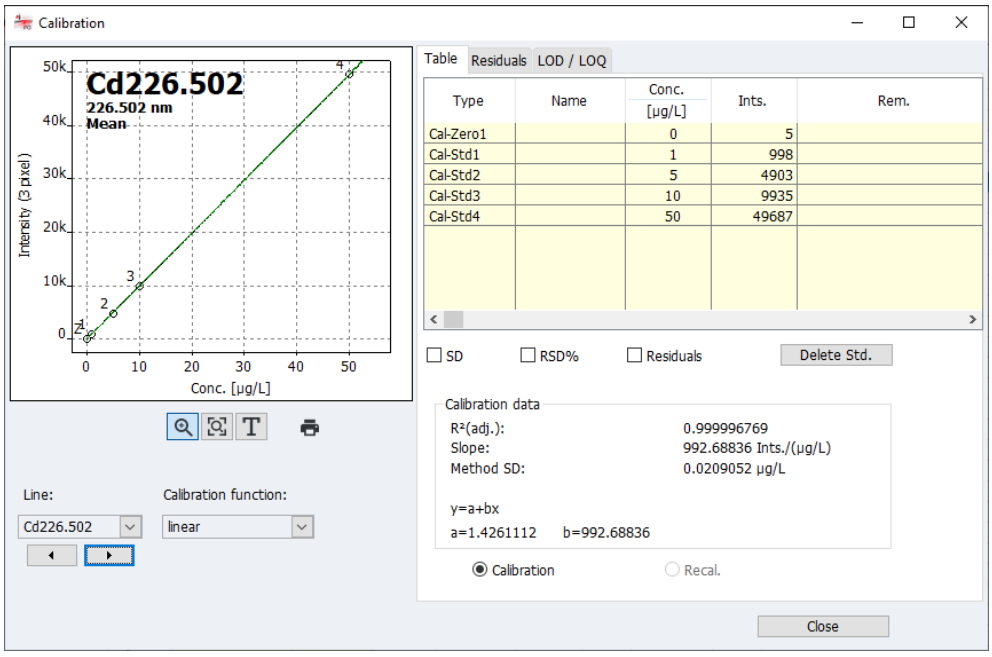
7 Calibration

The calibration is carried out during the measurement according to the options selected in the sequence. The calibration graphs and functions can be displayed and edited after the measurement.

▶ Open the window **Calibration** by clicking on .

Alternatively, double click on one of the sequence rows "Compute calib." or select the menu item **Method development | Calibration**.

The **Calibration** window shows the calibration graph calculated considering the graph parameters.



Window Calibration

The window contains for each of the analysis lines defined in the sequence

- Graphical representation of the calibration graph
- Calibration table
- Parameters
- Residuals
- Limits of detection (LOD) and limits of quantitation (LOQ).

Selecting a line

In the list field **Line** select the analysis line for the Calibration view. With the arrow keys under the list change between the displays of the individual lines.

Selecting a calibration function

In the list **Calibration function** select between the possible regression calculations of the calibration graph:

Calibration option	Description
linear	Linear progression of the calibration function $y = a + bx$
nonlin. ratio.	Non-linear progression of the calibration function described by a broken ratio function $y = \frac{a + bx}{1 + cx}$
nonlin. quadr.	Non-linear progression of the calibration function described by a quadratic function $y = a + bx + cx^2$
automatically	For the calibration a linear and non-linear function each are calculated. This is followed by a Mandel test in which the sums of the squared residues are compared. If the sum for the nonlinear function is significantly lower than that for the linear function, the nonlinear calibration curve will be selected. Otherwise, the linear calibration curve will be used. The non-linear function is selected in the window **Options/Analysis sequence** (→ "Options for analysis sequence" p. 138). As default setting the broken ratio function has been provided.

7.1 Graphic presentation of calibration curve

In the graph, the measuring points, the calculated calibration curve, and the residuals are displayed. The numbers at the measuring points correspond to those used on the **Table** tab. The calibration zero point has been identified with Z (Zero).

Color marking

Measuring points have been marked in the following manner:

Color	Meaning
Black	Normal measuring point
Light gray	Deleted/outlier (not included in calculation)
Blue	Suspected outlier (included in calculation)

The graphs have also been marked in color

Graph color	Meaning
Black	Calibration curve within the valid calibration range
Blue	Calibration curve outside the valid calibration range
Green	Lower and upper limit of the prognosis range within the valid calibration range
Light gray	Lower and upper limit of the prognosis range outside the valid calibration range

Note on the prognosis or confidence range

The position of the prognosis range depends on the selected statistical certainty. It is a measure of the "quality" of the calibration, from which also the statistical certainty of the measurement of the analytical samples depends in the end. Besides, the prognosis range serves to identify suspected outliers among the calibration points. The statistical certainty is selected in the window **Method / Statistics** (→ "Specifying statistical analyses – Statistics tab" pg. 44). For the selection between prognosis and confidence band see "Options for analysis sequence" p. 138.

Enlarge the calibration graph

After clicking on a graphical area can be enlarged with the mouse button held down. reverses the enlargement again.

Insert remark

A text field for a remark can be inserted in the graph.

- Click on .
- With the left mouse button held down drag the frame for the text field onto the graph.
- In the open input window select the font under **[Font]**.
- Enter the text and click on [OK].
  - ✓ The text is displayed on the graph.

Print calibration graph

The calibration graphs and the calibration data are output to the printer after clicking on . Additional information on printing has been provided in section "Printing results data" p.117.

7.2 Displaying the calibration results

7.2.1 Calibration - Table tab

On the **Table** tab, the value pairs of the standards (entered concentration / measured value) are output.

If the standards were measured repeatedly and a statistical analysis option set in the method, you can additionally output the standard deviation (SD) and the relative standard deviation (RSD%) or the range (R) and the relative range (R%) by enabling the corresponding checkboxes.

To exclude individual calibration standards from the calculation, select the standard in the table by a mouse click and then click on **[Delete std.]**.

The measurement is only marked as deleted and can be reactivated at any time.

Under the measured value table the calibration data are displayed provided they can be meaningfully calculated:

Parameters	Meaning
R^2 (adj.)	Coefficient of determination
Slope	Slope of calibration curve
Method SD	Standard deviation of the method

BEC	The BEC value (background equivalent concentration) is the concentration of the analyte producing an intensity equivalent to the background. A lower value corresponds to a higher sensitivity.

7.2.2 Calibration - Residuals tab

The graph on the **Residuals** tab shows the deviations of the calibration points from the calculated calibration graph and the limits of the prognosis range.

7.2.3 Calibration - LOD/LOQ tab

The limits of detection and the limits of quantitation of the ICP-OES device can be determined based on the current calibration results.

In this area, values of the blank method and the calibration graph method will only be displayed if the device has been calibrated already.

Parameters	Meaning
Limit of detection	The weight (concentration) of the element to be analyzed that can be detected with a defined statistical certainty.
Limit of quantitation	The minimum weight (concentration) of the element to be analyzed that can be determined with a defined confidence level.
SD Blank (DL)	only for blank value method Measured standard deviation of the blank value (DL sample)

With **[Calculate]** start the calculation of the limits of detection and quantitation.

Calibration graph method

The calculation of the limits of detection and determination according to the calibration graph method necessitates a linear calibration graph. The calibration should be carried out in the lower concentration range. Calibration parameters that are essential for the result of computation include the following:

- Number and position of calibration points
- Number of repeat measurements per standard
- Quality of regression
- Slope of calibration curve
- Relative statistical certainty (probability level)

The values obtained from the calibration graph method can be considered useful only if the calibration was run in the lower concentration range.

Blank method

The standard deviation of the blank is determined within the sample measurement. For this purpose, the measurement of the blank value (QC Blank DL) is included in the sequence (→ "Combining sample and action sequences for the sequence" S. 56).

For the blank method the following calculation rule is used:

▶ The blank is to be measured 11 x.

- From the obtained values, the absolute standard deviation SD of the blank is determined.
- The following formulas apply to the limits of detection and determination:

  Limit of detection (LOD) LOD = 3 * SD / (slope of the calibration graph)

  Limit of quantitation (LOQ) LOQ = 9 * SD / (slope of the calibration graph)

7.3 Modifying the calibration graph

You can modify an existing calibration graph by following procedures:

- Changing the used calibration function
- Disabling/Enabling standards
- Replacing a measured standard

- To change the calibration function select a new model in the list field **Calibration function**.
- To exclude a standard from the calculation, select it on the **Table** tab and then click on **[Delete Std.]** . The measurement is only marked as deleted and can be reactivated at any time.
- The modified calibration parameters will be applied to the results when you open the menu item **Routine | Reprocess** or click on  in the toolbar (→ "Reprocessing analysis results" p. 69).
- A standard can also be measured again and the results be reprocessed (→ "Reprocessing analysis results" p. 69).

8 Quality control

The Quality Control function serves to monitor the measurement results of a method over a longer period of time. For this purpose, specific QC samples of different types are chosen for a method and included in the sequence.

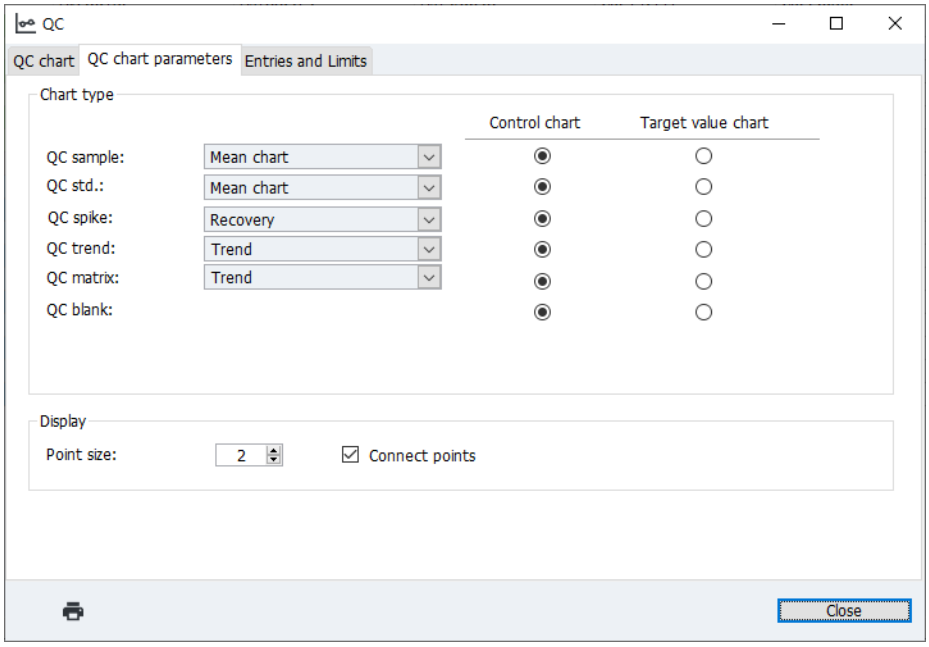
The evaluations are presented on quality control charts (QC charts) and saved along with the method. The QC charts are available after every loading of the method and will be updated at the next measurement start.

The type of QC samples and their parameters are defined in the window **Method / QCS** (→ "Specifying quality control samples for QC tabs - QCS tab" p. 46) and in the sequence the integration of the QC sample (→ "Combining sample and action sequences for the sequence", p. 56).

You can view the QC charts of the loaded (active) method in the **QC** window. There, you can also define the parameters and the configuration of the QC charts.

- ▶ Open the **QC** window by clicking on in the icon bar or select the menu item **Method Development | QC**.

8.1 Parameters of QC charts



Window QC / QC chart parameters

Chart types

QC sample type	Type of QC evaluation
QC sample	**Mean chart**
QC std.	**Mean chart (norm.)** - Not for target value chart.
	Recovery
QC spike	**Recovery**

QC trend **QC matrix**	**Trend** **Ranges** - Not for target value chart. **Precisions** - Not for target value chart.
Blank	No selection provided. The intensity of the blank is displayed.

The target parameters and control (**CL,CU**) and warning (**WL, WU**) limits for chart type **Control chart** are calculated from the mean and the variation of the values from preparation period. Taget parameters and exclusion limits for chart type **Target value chart** are determined from the target value and the limits specified for QC sample types (→ "Specifying quality control samples for QC tabs - QCS tab", p. 46).

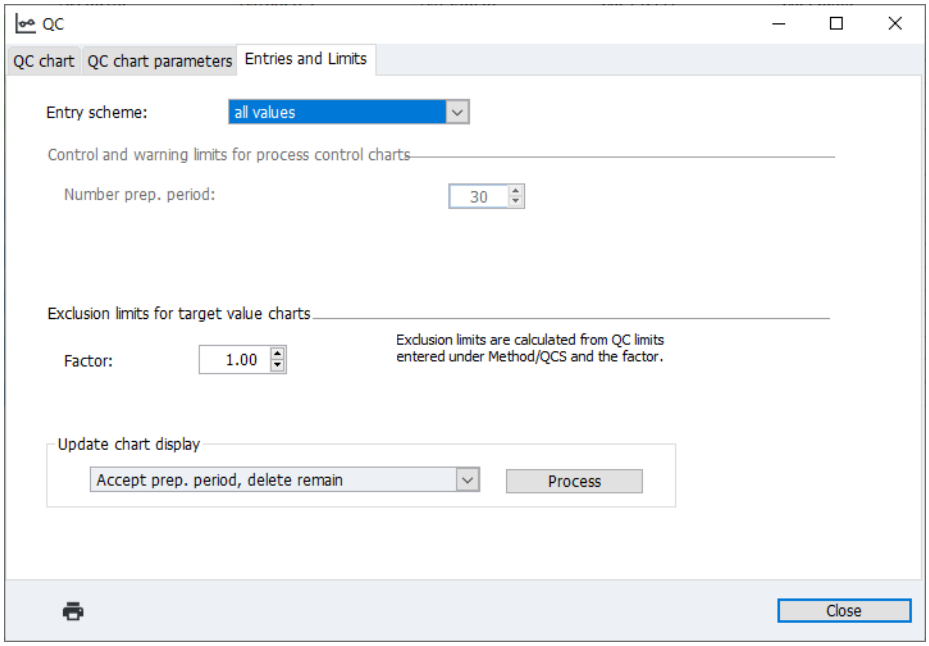
Display

In this field, you can choose the point size used for the graph, and if the points shall relate to each other by a line.

Option	Description
Point size	The individual points are displayed as circles. Choose a higher point size for larger circles.
Connect points	Connects the points on the graph with each other by a line.

8.2 Entries and limits of QC charts

The content of the QC charts is defined in the window **QC / Entries and limits** and can be customized to the requirements of your laboratory regarding the frequency of the entries.



Window QC / Entries and Limits

Option	Description	
Enter schema	**All values**	Enter every performed QC control.
	1 value/day	Enter only the last QC control of the day.
	2 value/day	Enter only the first and the last QC control of the day.
	Note A "day" corresponds to one day according to the PC clock, i.e. in the course of a day, any previous entry on the QC chart will be overwritten by a new QC value; however, when a new day begins, a new entry will be generated.	
Number prep. period	**Control chart** only: The preparation period is a number of QC chart entries that are used for the calculation of control (C) and error (E) limits. The preparation period always contains the older chart entries. If set to 0 (no prep. period), all entered QC data will be included in the calculation of control and error limits.	
Excluson limits for target value charts	**Target value chart** only: The exclusionlimits are calculated from the limits specified for the QC control samples multiplied by Factor (default is 1)(→ "Specifying quality control samples for QC tabs - QCS tab", p. 46).	

Replacing charts

Define the procedure for (almost) full charts. To this end select one of the options from the list:

Option	Description
Accept prep. period, delete remain	**Control chart only:** Accepts the preparation period of the old chart for application to the new chart and deletes remaining values.
Last values -> new prep. period	**Control chart only:** The values of the old chart measured last represent the preparation period of the new chart; all other values will be deleted from the chart. New measured values will be evaluated based on the newly created preparation period.
Delete all, new prep. period	All values will be deleted. **Control chart only:** New measured values will first fill the preparation period.

By clicking on **[Process]** you replace the QC charts in accordance with the option selected above.

8.3 Displaying QC charts

The QC charts are displayed in the **QC / QC charts** window. Separate charts are generated each for every QC sample type defined in the method and for every element line specified there.

Options/views

Options/views	Description
Type	Here, choose the QC sample type to be displayed.
Line	Here, choose the element line to be displayed.

Displayed values	Number of displayed values and the date of the first and the last value displayed.
Entries	Total number of entries on the current QC chart and the date of the first and the last value.
x(max)	Select the number of entries to be displayed on the graph.
y Scale	ENTRIES – Maximum of y axis is scaled according to the highest value CONTROL LIMITS - Maximum of y axis is scaled to the range of the control limits or exclusion limits
	Prints the QC graph inclusive of alphanumerical data and measured values.

Graph area

Color/marking	Meaning
Yellow field	**Control chart only:** Preparation period
Light gray horizontal line	**Control chart only:** Mean value calculated from preparation period Target value chart only: Target value
Red horizontal lines	**Control chart only:** Upper and lower control limit (C) calculated from preparation period (3 Sigma) Target value chart only: Upper and lower exclusion limits (EU, EL) depending on the limits of the QC sample.
Green horizontal lines	**Control chart only:** Calculated warning limits (W; 2 Sigma).
Small circles	Measuring points (black: active entry; gray: inactive entry)

If you click on a measured value on the graph, a window is opened with the following information on this measured value.

Option	Description
Number	Number of the measured value in the QC series
Value	Measured value (converted according to the presentation type of the QC chart)
Date / Time	Date and time of measurement
User	user logged in at the time of measurement
Version	Version of the method used
Delete Entry / Activate Entry	Flag a data point as deleted / re-activate a deleted data point
Add comment	Add a comment to the data point, e.g. reason for deletion

9 Controlling and monitoring spectrometer and accessories

9.1 Spectrometer

The **Spectrometer** window serves to test the spectrometer functions and set the spectrometer parameters.

The following data can be adjusted or viewed, and the following actions performed:

- Device data
- Display of the readout parameters of the detector
- Start measurements for device optimization

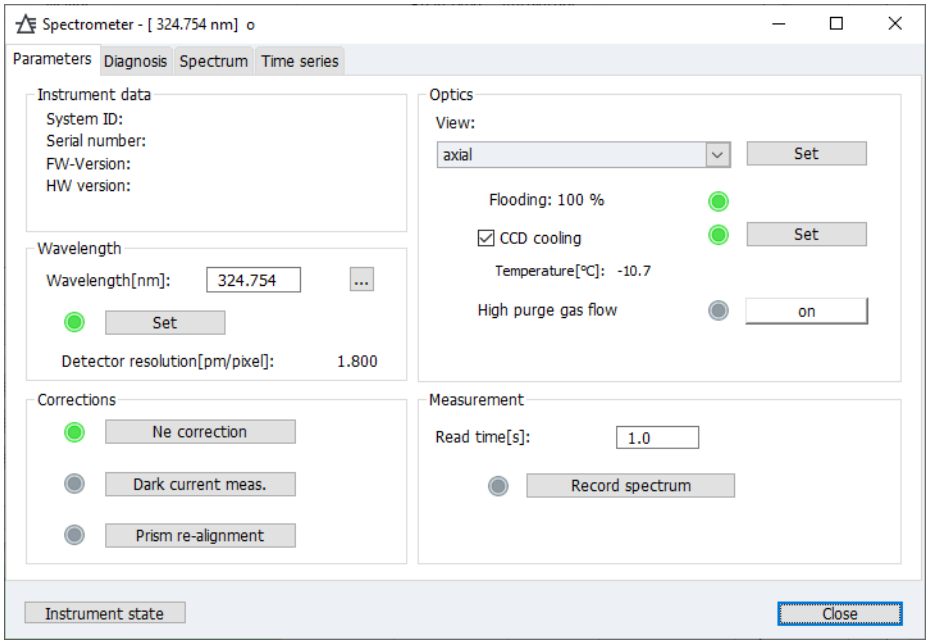
▶ Open the **Spectrometer** window by clicking on in the icon bar or select the menu item **Method Development | Spectrometer**.

With **[Instrument state]** a graph is displayed for the output of the messages of the safety sensors of the ICP-OES device. In case of problems with the plasma, you can view the error messages of the sensors here.

9.1.1 Configuring spectrometer parameters and testing functions

The **Spectrometer / Parameters** window includes the following functions:

- Check basic device functions
- Start automatic corrections to the optical system
- Start a test measurement at a selected wavelength



Window Spectrometer / Parameters

Elements of the window Spectrometer / Parameters

Parameters	Description
Instrument data	In the device data group various service and version numbers are displayed that are required for the device service.
Wavelength	In the field **Wavelength** the selected wavelength is displayed. A wavelength can be adjusted after clicking on ••• in the window **Select Element / Line** (→ "Inserting analysis lines into the line table" p. 28). With **[Set]** the spectrometer is moved to the selected wavelength.
[Ne correction]	Perform a wavelength calibration of the detector.
[Dark current measurement]	Start the measurement of the dark signal of the detector.
[Prism re-alignment]	Optimize the mapping of the dispersion order on the detector by prism adjustment (adjustment for energy maximum).
View	Select the monitoring direction of the plasma in the list field (**axial** – from above, **radial** – from the side).
CCD cooling	If the checkbox is enabled, the cooling of the CCD detector can be started with **[Set]**. If the checkbox is disabled, the cooling is stopped. The CCD cooling is started automatically with the ignition of the plasma. A manual control is only necessary in exceptional cases, e.g. after an error message during automatic start. In the field **Temperature** the current temperature of the CCD detector is displayed.
High purge gas flow	Purge the spectrometer with high gas flow.
Measurement	To start a measurement at the selected wavelength the total measurement period must be entered under **Read time**. **[Record spectrum]** starts the measurement. For the measurement the main settings for the plasma are used. The sample must be supplied manually. The autosampler is not used.

Measuring spectral peak at a selected analysis line

Start a test measurement at a selected analysis line in the window **Spectrometer / Parameter**.

- ▶ Ignite the plasma.
- ▶ In the area **Wavelength** use ••• to open the window **Select Element/Line** and set the desired line.

  Alternatively, enter the value directly into the input field **Wavelength**.
- ▶ Move the spectrometer with **[Set]** to the desired wavelength.

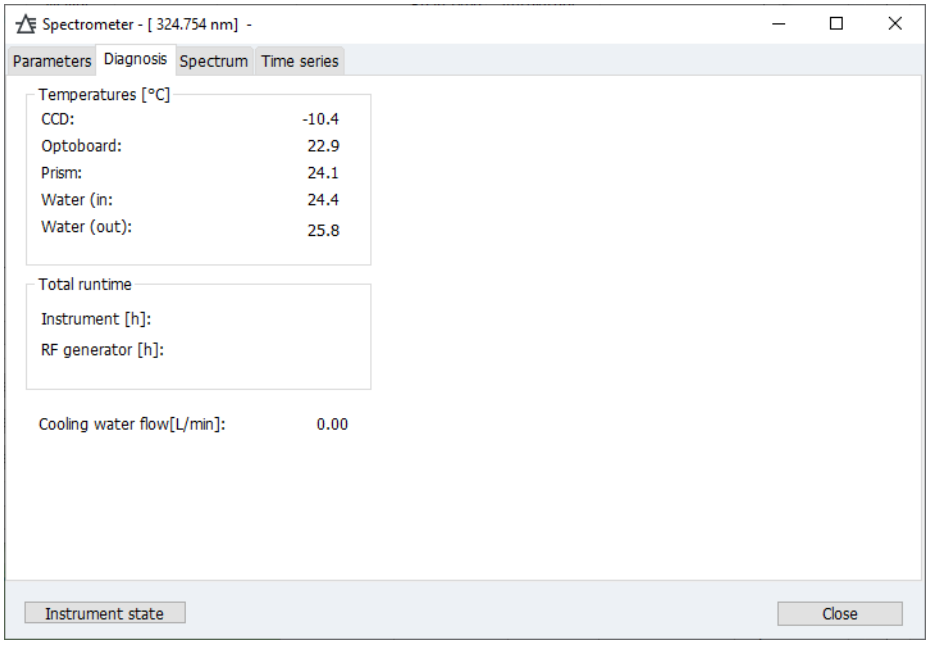
  If the setting has completed with success, the marker next to the setting turns green.
- ▶ Start a dark current measurement with **[Dark measurement]**.
- ▶ For the following measurement select the view direction **axial** or **radial**.
- ▶ Set the **Read time** [s].
- ▶ Provide the sample and immerse the intake tube in the sample.
- ▶ Wait for the time period to achieve stable nebulization of the sample.

▶ Start the measurement with **[Record spectrum]**.

✓ The measurement takes place and the measurement results are displayed in the window **Edit spectra** (→ "Displaying and editing intensity spectra (window Edit spectra)" p. 79).

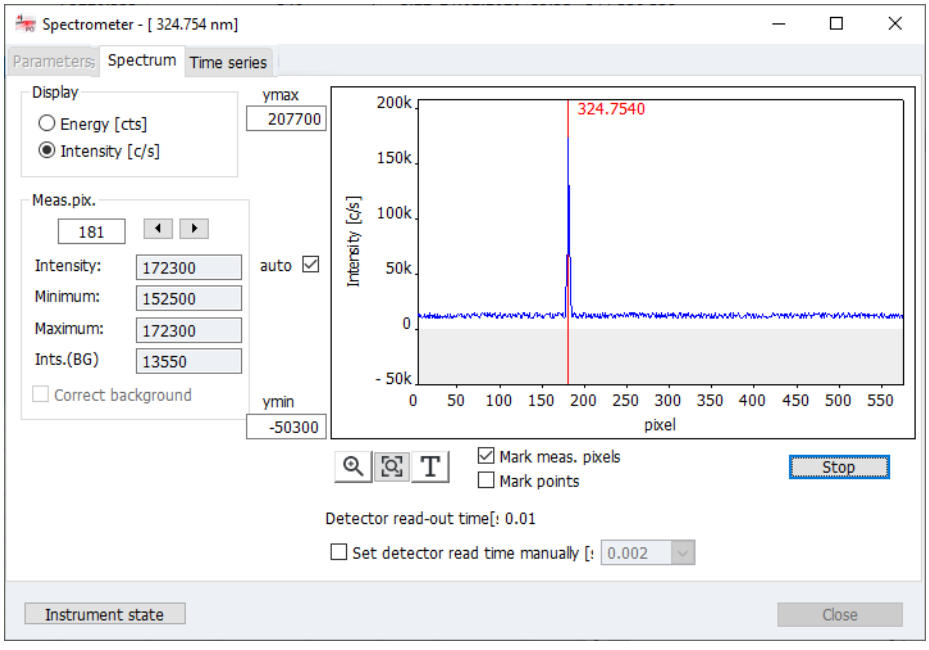
9.1.2 Diagnosis of the specktrometer

Service-relevant parameters are displayed in the **Spectrometer / Diagnosis** window.



Window Spectrometer / Diagnosis

9.1.3 Performing continuous peak measurements



Window Spectrometer / Spectrum

In the window **Spectrometer / Spectrum** start a continuous measurement at a specified wavelength.

The continuous measurements are used for device optimization during service.

Graphical display and digital analysis

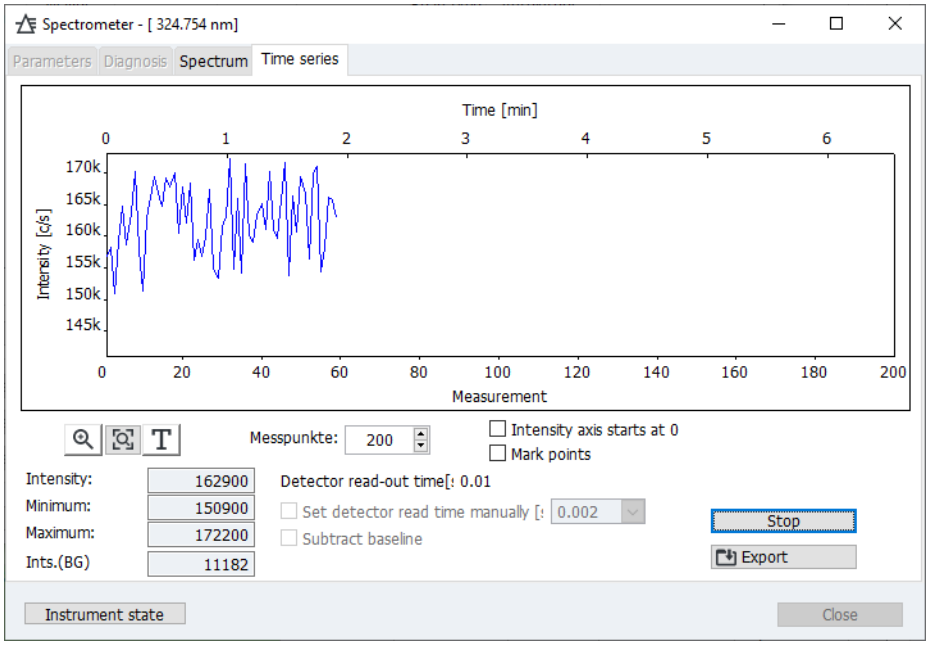
Option	Description	
Display	Options for displaying the spectrum.	
	Energy	Display of the energy spectrum, measuring unit: cts (counts) To obtain measurement results with as little noise as possible the integration times for the detector are chosen for the energy maximum to be at approx. 30000 cts.
	Intensity	Presentation of the energy per time unit, measuring unit: cts/s (counts per second) Based on the intensity different peaks can be compared irrespective of the integration time.
Meas.pixel	Selection of the pixel whose value is continuously displayed in **Intensity**. In the fields **Maximum** and **Minimum** the corresponding results of the continuous measurement are displayed.	
Mark. meas. pixels	Highlight the set MEASURING PIXEL in the graph by a vertical red line.	
Mark points	Highlight the measuring values for each pixel in the graph by a point.	
Set detector read-out time manually	Select the read-out time for the CCD detector from the list field. Longer read-out times result in higher energy values. The default for the read-out time of the CCD detector is 0.01 s.	
Graph scaling	Enter values for the start and end points of the ordinates directly into the input fields at the axes. Alternatively, you can select the range to be displayed after enabling zoom mode by holding down the left mouse button (→ "Frequently used control elements" p. 14). Reverse scaling by enabling the option **auto** or clicking on .	

- ▶ In the window **Spectrometer / Parameters** set the wavelength.
- ▶ Change to the **Spectrum** tab.
- ▶ Start the continuous measurement with **[Start]**.

The measuring values are recorded with the set parameters and repeated continuously until **[Stop]** is pressed.

9.1.4 Recording signal progression

In the window **Spectrometer / Time series** record the signal progression of the intensity for the wavelength currently set in the spectrometer across a selected number of measuring points.



Window Spectrometer / Time series

In addition to the graphical display the digital values of the current intensity and the maximum and minimum values of the intensity are output.

You can set the following parameters for recording the signal progression:

Option	Description
Scaling	After enabling zoom mode select the range to be displayed by holding down the left mouse button (→ "Frequently used control elements" p. 14). Reverse scaling by clicking on .
Intensity axis starts at 0	Do not set the scaling of the y-axis automatically, but let it start at "0".
Meas. points	Select the number of measuring points from the list.
Mark points	The measuring points are highlighted with a point in the graph.
Set detector read time manually	Select the read-out time of the CCD detector from the list field.

9.2 Plasma

The **Plasma** window includes the following functions:

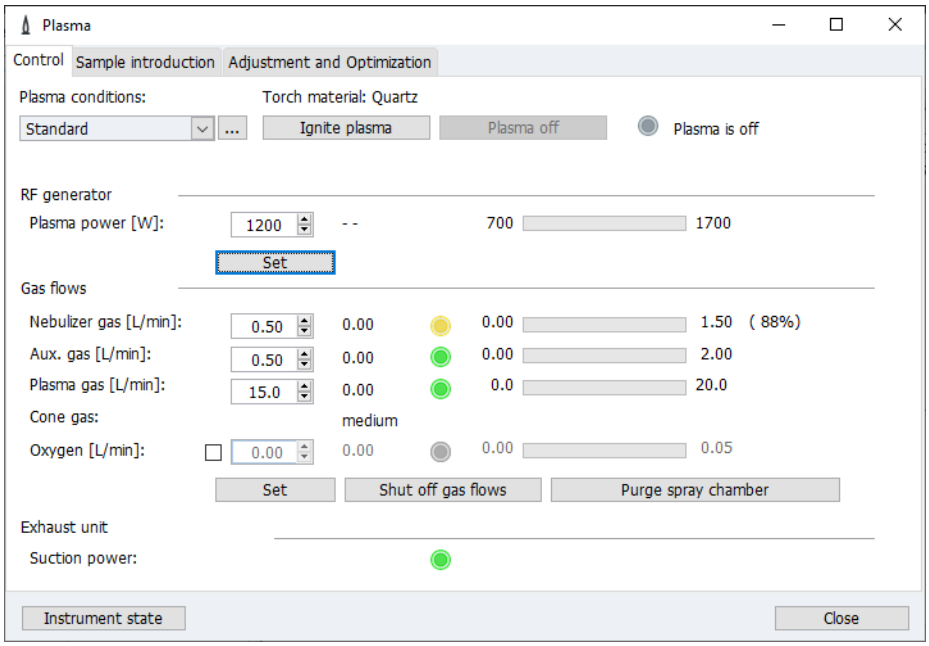
- Ignite plasma/extinguish plasma
- Control of the HF generator
- Setting the gas flows
- Control of the analyzer pump
- Adjustment of the transfer optics
- Optimization of nebulizer gas flow und plasma power

▶ Open the **Plasma** window by clicking on or select the menu item **Method Development | Plasma**.

With **[Instrument state]** a graph is displayed for the output of the messages of the safety sensors of the ICP-OES device. In case of problems with the plasma, you can view the error messages of the sensors here.

9.2.1 Igniting the plasma, setup plasma conditions

In the window **Plasma / Control** you ignite/extinguish the plasma and adjust the gas flows in the device.



Window Plasma / Control

Functions in the window Plasma / Control

Option	Description
Plasma conditions	Select plasma conditions (plasma power and gas flows) (see below).
[Ignite plasma]/[Plasma off]	Ignite the plasma and extinguish it when the ICP-OES device is ready (→ "Igniting plasma/extinguishing plasma" p. 64).
RF generator	Set the effective plasma power. The plasma power defines the plasma temperature. The generator current is controlled via the firmware to achieve the effective plasma power.

Gas flows	Switch on and adjust the gas flows.	
	Nebulizer gas	Nebulizes the sample and transports the sample aerosol to the plasma; it is connected to the nebulizer The percentage value in the row of the nebulizer gas provides information as to how permeable/clean the nebulizer is (see below)
	Aux. gas	Pushes the plasma away from the injector; flows between the inner tube and the injector
	Plasma gas	Flows along the outer tube; used to generate the plasma
	Cone gas	The cone gas removes the "cold" plasma tail to eliminate interference due to recombination in the plasma in the axial direction of observation. At the same time the cone gas supports the cooling of the cone.
	Oxygen	Oxygen can be added to the atomizer gas as an additional gas for selected applications. The oxygen flow must be activated with the check box in front of the gas setting before it can be changed.
[Shut off gas flows]	Close all gas valves.	
[Purge spray chamber]	The nebulizer gas is activated for 1 minute to purge air from the spray chamber. This facilitates the ignition of the plasma after interrupted operations.	
Suction power	A safety circuit checks that the power of the connected extraction is sufficient for the operation of the ICP-OES device.; If this is the case the indicator lamp is illuminated in green.	

With the **[Set]** buttons you configure the modified parameters (plasma power and gas flows) at the ICP-OES device.

Evaluating the nebulizer function

The nebulizer must be cleaned if sample particles or high concentrations of salt in the samples have clogged it up. An indicator that the nebulizer has clogged up is increased nebulizer gas pressure.

Compare the current percentage (pressure) of the **Nebulizer gas** parameter with the value achieved after installation of the new or cleaned nebulizer.

Clean the nebulizer as described in the operating instructions of the ICP-OES device if the percentage has increased significantly (by more than half the original value), at the latest however if 75% are reached.

Selecting the plasma conditions

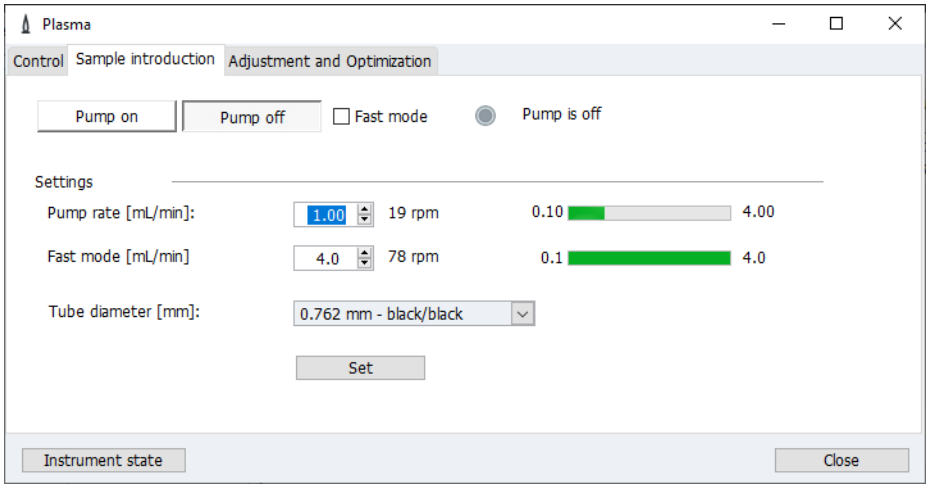
The list **Plasma conditions** contains saved plasma parameters for different sample matrices and, if a method has been loaded, the line-specific parameters of the method.

By clicking on ••• a context menu opens with functions for managing the parameters selected in the list:

Function	Description
Save current plasma conditions	Save configured plasma conditions (plasma power and gas flows) and add them to the list.
Delete entry	Delete the selected entry. The defaults **Standard**, **Kerosene** and **Hydride** cannot be deleted.
Configure plasma conditions	Configure the plasma parameters of the selected entry at the ICP-OES device.
Copy to method line	Available if a method line has been selected in the list. Transfers the plasma conditions to the method parameters of the selected line.
Copy to all method lines	Available if a method line has been selected in the list. Transfers the plasma conditions to the method parameters of all lines.
Set as method defaults	Save current plasma conditions as default values for newly inserted method lines (does not apply to line favorites).

9.2.2 Checking the sample introduction or the pump

In the window **Method / Sample introduction** check the function of the hose pump at the ICP-OES device.



Window Plasma / Sample introduction

Functions in the window Plasma / Sample introduction

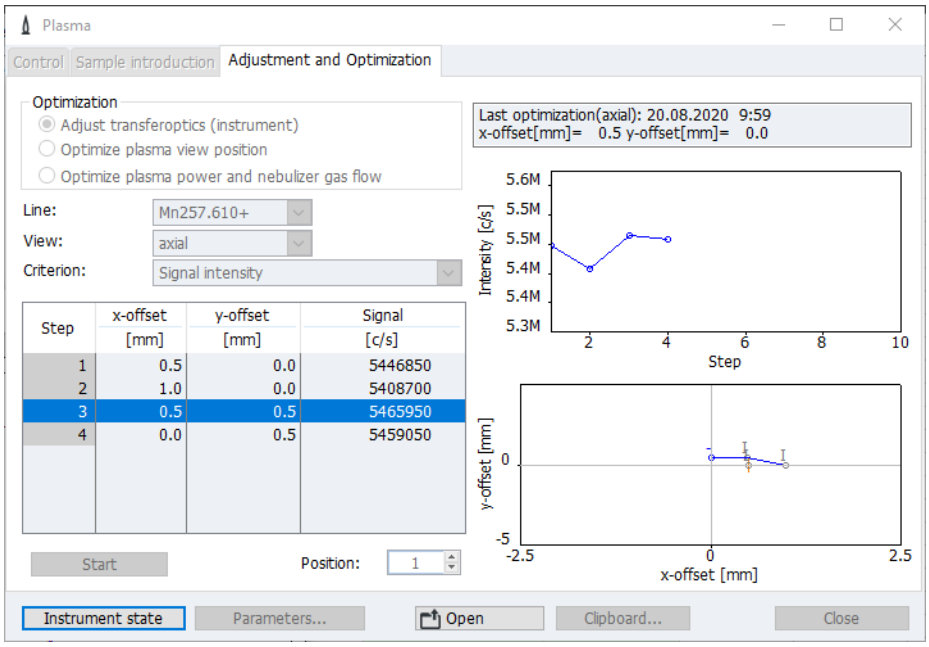
Function / parameters	Description
[Pump on]/ [Pump off]	Switch the pump on and off. In the original state after activation of the ICP-OES device the pump is switched on.
Fast mode	Move the pump manually into fast mode. The function can be used to manually wash the sample transport system. After successful washing the checkbox must be disabled.
Pump is running ...	The current pump speed is displayed with the unit RPM (rotations per minute).
Pump rate [ml/min]	Set the pump rate for the sample transport during the measurement.

Fast mode [ml/min]	Set the pump rate for fast mode. With the fast mode the transport time during a change of sample or the transport time of the washing solution to the nebulizer is optimized.
Tube diameter	Select the hose used from the list. The transported sample volume (pump rate) is calculated from the information of pump speed and hose diameter. The hoses have been coded with colored stoppers. Select the stopper combination of the hose used from the list.
[Set]	Apply the settings.

9.2.3 Adjustment and optimization of plasma

In the window **Plasma / Adjustment and optimization** made the following adjustments:

- Alignment of the transfer optics with the optical axes of the spectrometer
- Calculation of the offset values of the transfer optics for an analysis line from the method
- Optimization of plasma power and nebulizer gas flow



Window Plasma / Adjustment and optimization

Two different methods are available for adjustments and optimization and can be selected under **[Parameters]**:

Method	Description
Grid search	The range is scanned based on a grid. From the number of measuring points the one with the highest intensity is determined. The adjustment is exact but takes long due to the determination of many measuring points.

Simplex optimization	The energy maximum is determined iteratively. From a start measuring point the measuring point with the highest value in the vicinity is determined. Starting from this measuring point, the measuring point with the highest energy is determined again. The process continues until the energy maximum has been found. This method is faster than the grid search but less certain. In the various hot zones of the plasma several energy maxima may occur and thus an incorrect energy maximum be found if the start point was unfavorable. For the simplex method a **Stopping criterion** must be defined as percentage value. If 3 consecutive values do not differ by more than this percentage value, the adjustment is ended. If **Start with optimized values** is enabled, the optimized parameters of the last adjustment/optimization are used as start-up values for the current optimization.

For the **adjustment of the transfer optics (device)** the **Signal intensity** is used as criterion.

The criterion for the optimization is configured automatically dependent on the wavelength of the analysis line, but can be modified manually:

Criterion	Wavelength range of the analysis lines
Signal intensity	< 200 nm
Signal/background	200 to 350 nm
Signal/square root of background	> 350 nm

Adjusting the transfer optics on optical axes (plasma centers)

The adjustment of the transfer optics on optical axes takes place using Mn solution. Prepare Mn solution for the adjustments with the following concentration:

Monitoring direction	Mn solution
axial	1 mg/L
radial	10 mg/L

- ▶ Enable the option **Adjust transfer optics (instrument)**.

  The Mn analysis line is automatically set in the **Line** list.
- ▶ Select the adjustment method under **[Set]** (see above).
- ▶ Select the monitoring direction:

Option	Description
axial	Monitoring from above
radial	Monitoring from the side
attenuated axial	Monitoring of the attenuated energy from above
attenuated radial	Monitoring of the attenuated energy from the side
closed	Monitoring with the shutter closed (for service purposes)

- ▶ Immerse the intake hose into the sample. When using an autosampler, set the position on the sample rack.
- ▶ Click on **[Start]**.

  The adjustment of the transfer optics runs automatically. At the end of the adjustment the new data are displayed.
- ▶ Accept the new adjustment values by clicking on [OK].

Optimizing the monitoring position for an analysis line of the active method

The plasma has zones of different heat. During this optimization the monitoring point in the plasma is detected where the analyte has the greatest signal intensity. The values are saved in the method as **Offset**.

- ▶ In the **Linie** list select the analysis line from the method.
  The information about the monitoring direction is automatically transferred from the method and the criterion is set for the optimization (see above).
- ▶ Enable the option **Optimize plasma view position**.
- ▶ Select the adjustment method under **[Parameters]** (see above).
- ▶ Immerse the aspiration hose into the sample. When using an autosampler, set the position on the sample rack.
- ▶ Click on **[Start]**.

  The adjustment of the monitoring position runs automatically. At the end the optimized offset values are displayed.
- ▶ Transfer the new offset values to the method by clicking on **[OK]**.

Optimizing the plasma conditions for a sample

After having defined the monitoring position of the analytes in a sample you can optimize the plasma conditions (plasma power and nebulizer gas flow).

- ▶ Enable the option **Optimize plasma power and nebulizer gas flow**.
- ▶ In the **Line** list select the analysis line from the method.

  The information about the existing plasma conditions is automatically transferred from the method and the criterion is set for the optimization (see above).
- ▶ Select the adjustment method under **[Set]** (see above).
- ▶ Immerse the intake hose into the sample. When using an autosampler, set the position on the sample rack.
- ▶ Click on **[Start]**.

  The optimization of the plasma power and nebulizer gas flow runs automatically. At the end the optimized values are displayed.
- ▶ Transfer the new values to the method by clicking on [OK].

9.3 Autosampler

The autosampler is an optional accessory. The autosampler is detected during the initialization in the main settings window after starting the ASpect PQ program.

In the **Autosampler** window contains following functions:

- Display connected autosampler type
- Configure autosampler
- Adjusting the autosampler
- Rinse sample paths additionally
- Reinitialize autosampler
- Perform self test

The parameters directly relevant to the analysis (charging on the sample rack and wash steps) are to be specified in the method, the sequence and the sample identification data.

▶ Open the **Autosampler** window by clicking on or select the menu item **Method Development | Autosampler**.

Initializing the autosampler

The autosampler is generally initialized when the mains switch is activated. It may be necessary to re-initialize the autosampler, if it lost its orientation (e.g. because you pushed against it). Communication between the autosampler, the PQ 900 and the PC will be established.

▶ By clicking on **[Initialize]** you can re-initialize the autosampler when necessary without restarting the ASpect PQ program.

Note: When using the Cetac ASX-560 with dilution system, the **[Initialize]** button is hidden.

Detecting the autosampler

If the autosampler was only switched on after starting ASpect PQ, the use of the autosampler must be registered in the program. To do so click on **[Detect]** and then on **[Detect]**.

Washing the system

▶ In the window **Autosampler / Parameters** set the **Wash time**.
The default for the wash time is transferred from the current method.

▶ Click on **[Wash]**.

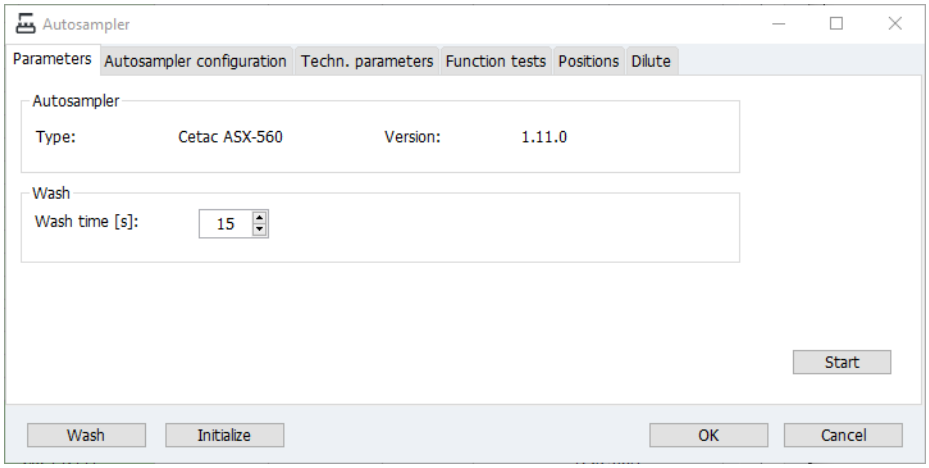
Alternatively select the menu item **Routine | Wash**.

✓ The system is washed with the pump in fast mode for the specified washed time.

9.3.1 Displaying the connected autosampler

In the window **Autosampler / Parameters** the following parameters are displayed or configured:

- Autosampler type
- Washing parameters



Window Autosampler / Parameters

Autosampler type

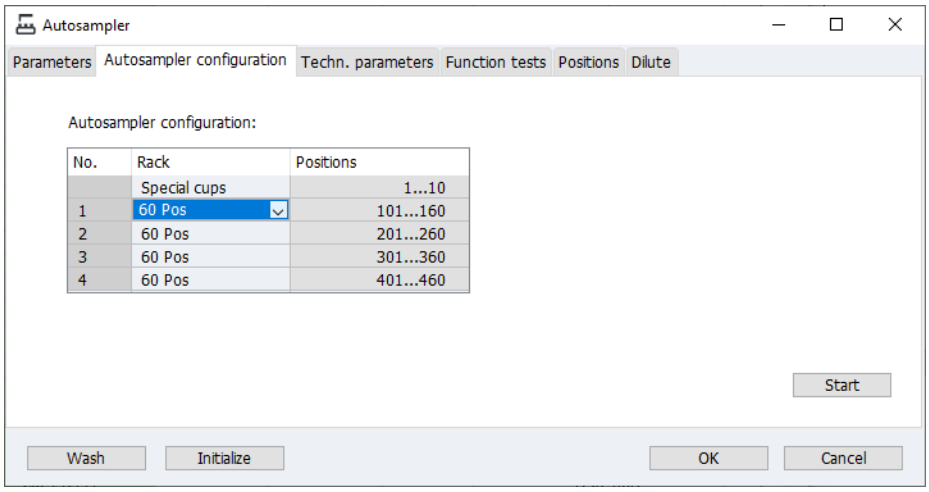
In the window **Autosampler / Parameters** the autosampler type detected during initialization and the firmware version of the autosampler are displayed.

Washing parameters

The parameters for washing the system from the sample cup through to torch are transferred from the current method (→ "Configurations for sample transport – Sample introduction " p. 33). Conversely, however, changes in the window **Autosampler / Parameters** do not affect the entries in the method. During system washing using the autosampler the wash solution is taken from the wash cup of the autosampler.

9.3.2 Configuring the autosampler

In the **Autosampler / Autosampler configuration** window configure the sample racks used in the sampler:



Window Autosampler / Autosampler configuration

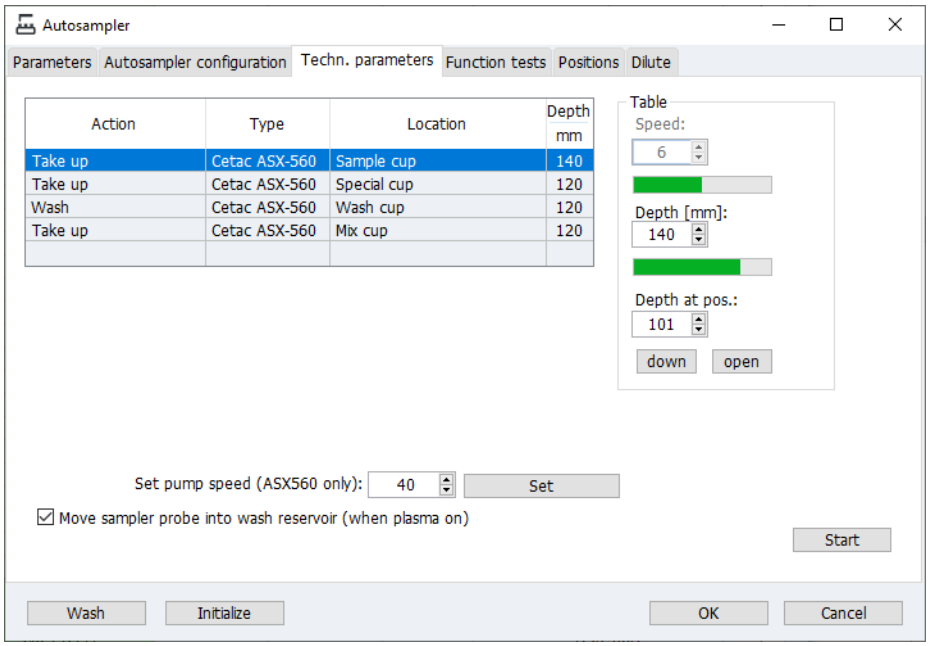
Depending on the autosampler used, different sample racks and racks with special samples can be positioned.

Select the sample trays from the table. For the variable sample racks three digit numbers have been provided as position numbers. The first digit defines the position of the sample tray on the autosampler, the two others the position on the sample rack. The

number 113 indicates e.g. position 13 on sample rack 1. The variable sample tray 1 is located on the autosampler before the wash cup, followed by sample racks 2 and 3.

9.3.3 Technical parameters of the autosampler

In the window **Autosampler / Techn. parameters** specify the immersion depth of the cannula into the various cups.



Window Autosampler / Techn. parameters

For the individual cup types the following actions are considered:

Cup	Action
Sample cup	Aspire samples through hose pump.
Special cup	Aspire special samples through hose pump.
Wash cup	Flush cannula and intake path.

Elements of the table

Option	Description	
Action	Available action options:	
	Take up	Pick the sample from the cup for transportation to the torch.
	Wash	Pick up wash solution.
Type	Connected autosampler type	
Location	Cup to which a given action refers	
Depth [mm]	The depth to which the cannula submerges in units of 1 mm	

Table subarea

Using the controls in the table area, you can change the parameters of the selected table row.

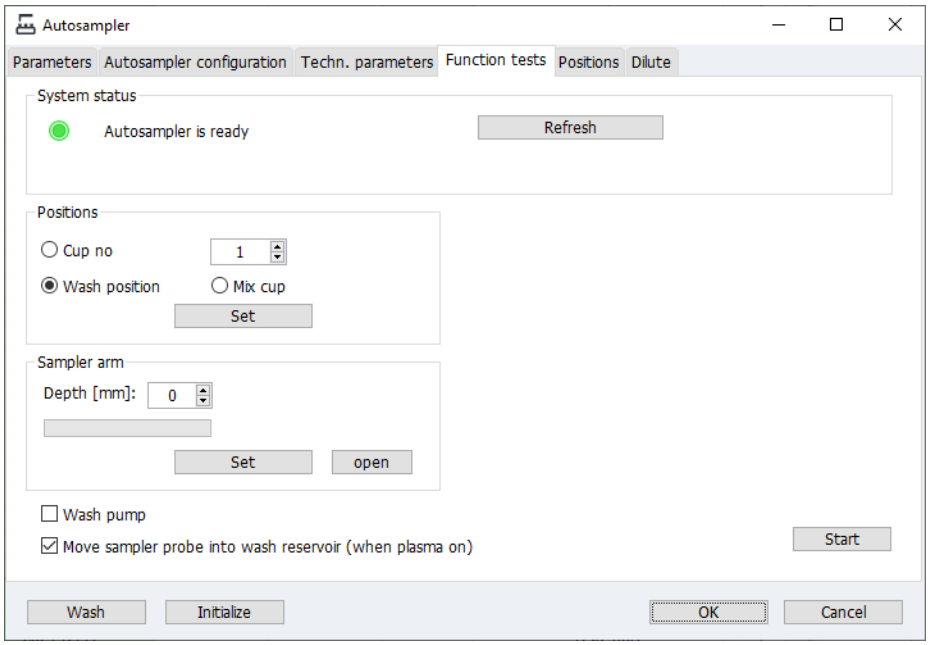
Option	Description
Speed	
Depth [mm]	Set immersion depth of the cannula The immersion depth is measured from the highest position of the sampler arm.
Depth at pos.	Position of special or sample cup at which the immersion depth is measured.
down	If activated, the sampler arm moves over the cup for which the positioning has to be adjusted. With sample and special cups, this is the position selected under **Depth at** pos. If not activated, the immersion depth and speed are changed without the sampler arm moving above the cup.

Further options

If the option **Move sampler probe into wash reservoir** is enabled, the cannula is automatically immersed into the wash reservoir after closing the window.

ASX-560 only: Set the speed of the wash pump (level 0...99). Button SAVE stores this value permanently in the autosampler.

9.3.4 Testing the autosampler functions



Window Autosampler / Function test

The following functions of the autosampler are tested:

Function	Description	
System status	Check for operational readiness. With **[Update]** the operational readiness is re-checked.	
Positions	After clicking on **[Set]** the autosampler moves to a selected position.	
	Cup no.	The autosampler moves to the position selected in the list.
	Wash position	The autosampler moves to the wash cup.

Sampler arm	Lower the autosampler arm to the **Depth** set in the list field.
Wash pump	Switch the wash pump on and off.

Move sampler cannula into wash reservoir

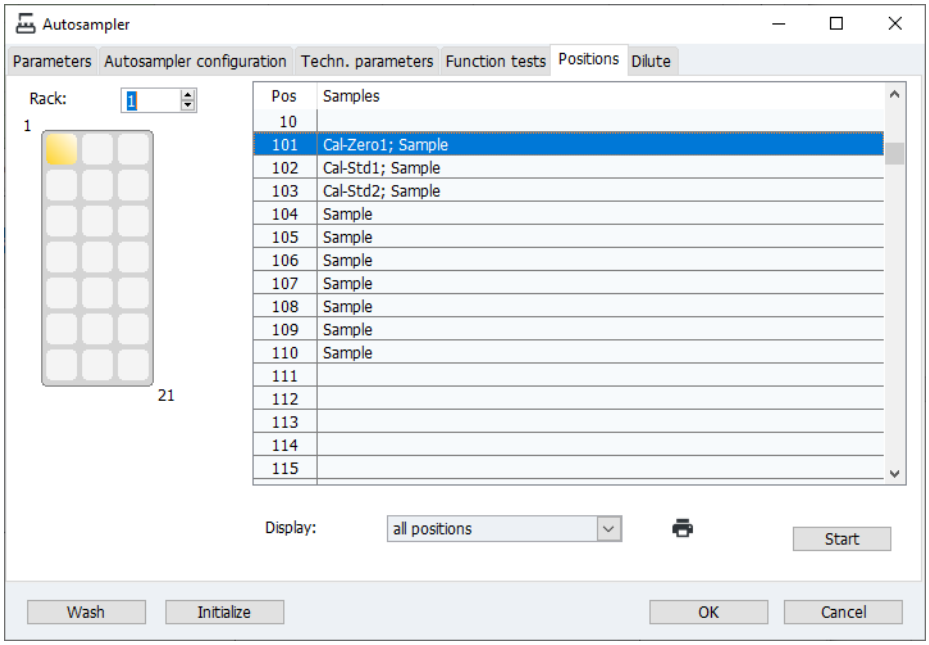
If the option **Move sampler probe into wash reservoir** is enabled, the cannula is automatically immersed into the wash reservoir after closing the **Autosampler** window.

9.3.5 Displaying the sample positions on the autosampler

The sample tray positions used in the current sequence are displayed in window **autosampler / Positions**.

For the display it is possible to choose between the options **all positions**, **only sample positions** and **only special positions**.

Next to the table a diagram of the sample trays with the currently selected sample positions is shown. The sample position can be selected both in the diagram and in the table.

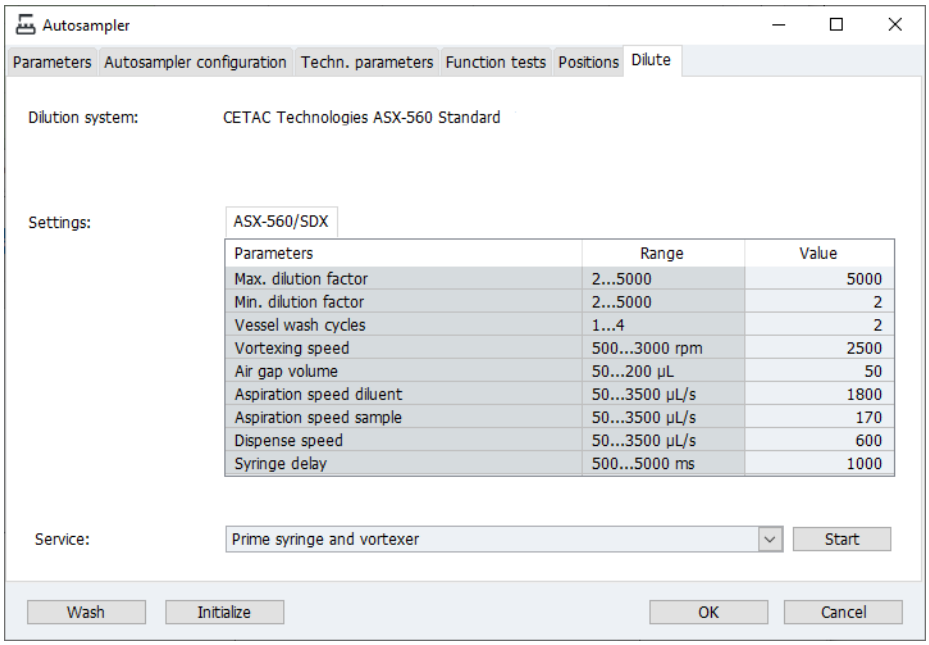


Window Autosampler / Positions

Note:
To display this window a method with at least one analysis line must have been loaded.

9.3.6 Dilution function

The parameters for sample dilution when using the autosampler ASX 560 with the Cetac SDX_{HPLD} are displayed in the **Autosampler / Dilute** window.



Autosampler / Dilution window

Settings

The parameters in the **Settings** section are preset settings that provide good results for sample dilution. You can vary the parameters within the setting ranges during a method optimization.

Service

In the Service list you can select service functions on the Cetac SDX Controller and execute them with [Start]:

Option	Function
Prime syringe pump and vortexer	Dilution fluid is pumped through the system with the syringe pump and dispensed into the vortexer. This removes air bubbles from the system and conditions the vortexer.
Move syringe to removal position	the syringe pump needs to be removed for maintenance, the syringe plunger must first be brought into the correct position using this function.
Initialize the ASXpress+ (after disassembly and cleaning)	If the ASXpress+ is installed: Initialize the ASXpress+ after installation or maintenance.

9.4 Recirculating chiller

In the cooling circuit a valve is switched in the ICP-OES device to open and close the circuit. The replacement of the cooling water is therefore supported by a wizard.

Note

Observe the notes on the maintenance of the recirculating chiller and the preparation of the cooling water in the operating instructions "ICP-OES device".

- ▶ Select the menu item **Extras | Maintenance**.
- ▶ In the Maintenance window click on **[Replace]**.
- ▶ Follow the instructions of the wizard.

10 Data Management

This section contains information about

- Print options
- Method and sequence management
- Management of results data
- Definition of units for concentrations and content
- Management of data for frequently used stock solutions and QC samples

10.1 Print functions in ASpect PQ

For the output of data ASpect PQ has a large number of output formats available. Along with issuing on the printer the data can be exported into Excel-, PDF-, HTML-; XML- or Text format or they can be saved as Bitmap or scalable graphs.

For the output of analysis results and the content of windows (e.g. the windows **Method** or **Sequence**) report templates are used. A set of report templates is installed by default. If required these sheets can be adapted individually with the report designer "Report-/Print module List & Label"

10.1.1 Printing results data

ASpect PQ offers a variety of options for printing measuring results:

- Print the complete record. The complete record of an analysis contains the method parameters, the calibration and analysis results with individual sample values (statistic runs). A report may be printed of the current results in the main window and the saved data.
- Print current results. In this printout only the data of the main window are printed. A selection can be made between a complete and compact printout.
- Print selected data of the **Overview** tab. For this printout you can select the analysis lines and results in a dialog window.

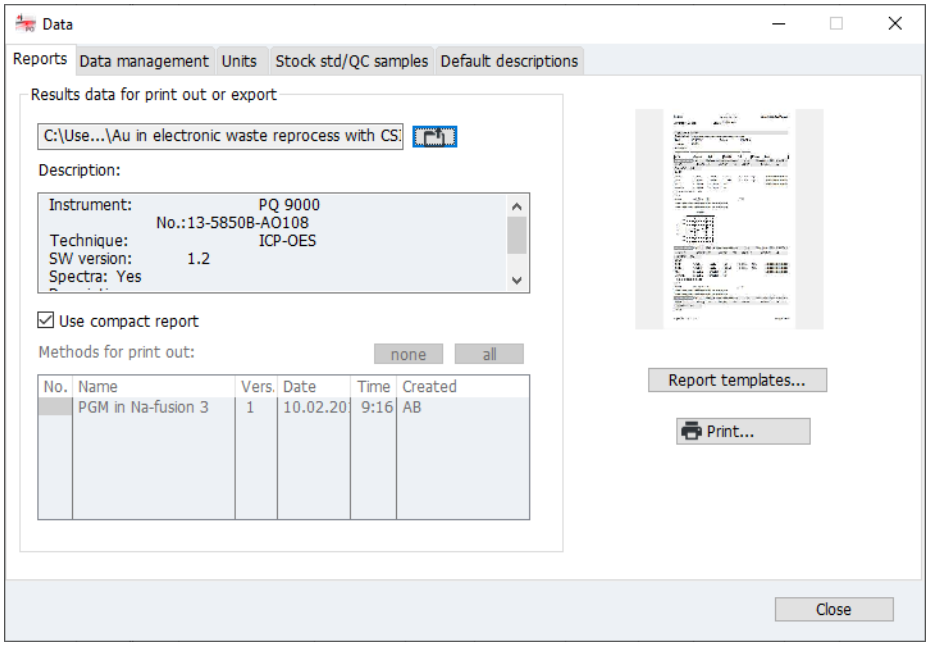
Print complete record

The complete record of an analysis contains the method parameters, the calibration and analysis results with individual sample values (statistic runs). The complete records can be printed of the results in the main window or the saved files.

▶ Open the **Data / Reports** window using icon  .

Alternatively, open the window using menu command **Extras | Data** or the menu command **File | Print | report**.

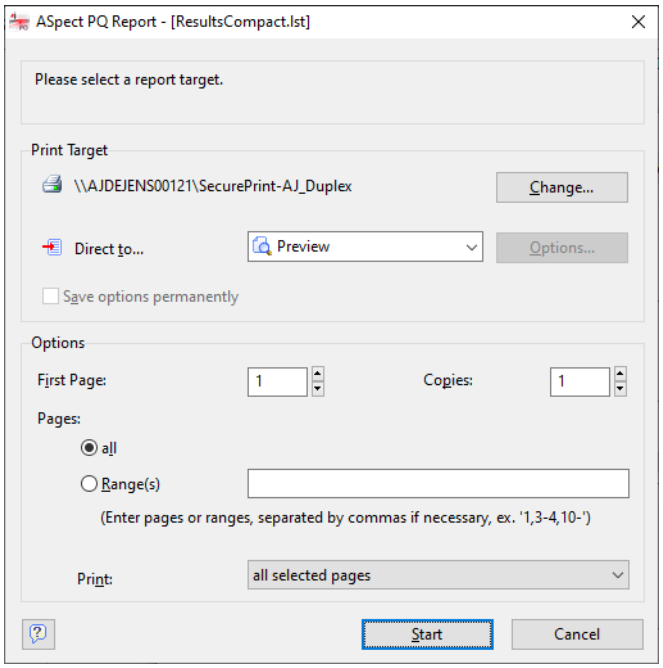
The name of the current file, file information (list **Description**) and all method versions used for creating the current results file are displayed.



Window Data / Reports with selection of the results data for printing

- If you want to print a saved file, use to open the default window **Open** and select the desired file.
- Select all method versions to be printed in the table.

  With the shift or Ctrl key held down click on the method versions you want to select. With the button **[all]** select all version, with **[none]** remove all selections.
- With **[Print]** open the window **ASpect PQ Report**.



Window ASpect PQ Report with the selection of the output format

- If necessary change the issue format in the list **Direct to** and set special parameters of the output format with **[Options]**.

- Enable the checkbox **Save options permanently** if you want to make the selected output medium the default for this print template.
- Start the printout with **[Start]**.

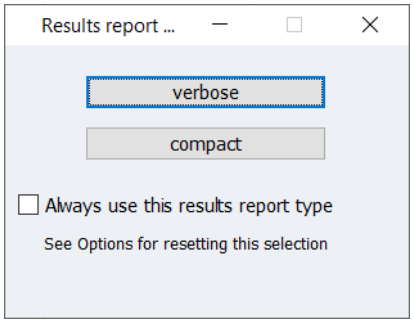
Note

For printing use the setting **Preview**. By clicking on **[Start]** the pages to be printed are first displayed in the print preview. This allows you to check that all desired data or whether unnecessary data are being output before they are sent to the printer.

Printing the main window result tabs

- Activate the Results tab in the main window, the content of which you wish to print.
- Start the printout with the menu command **file | Print | Active Window.**

  The window **ASpect PQ Report** opens.



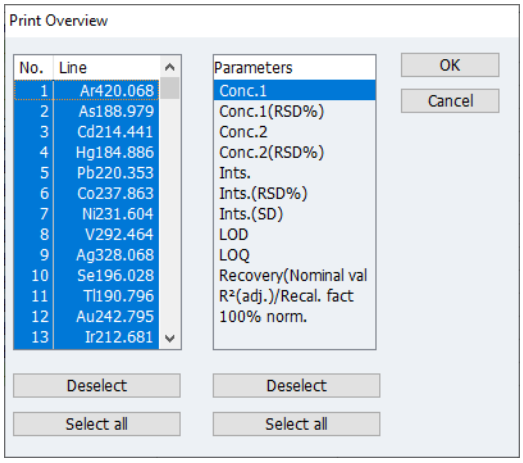
Window Results report format

- Click on **[verbose]** if you want to print the results with the signal diagrams. Select **[compact]** for printing the results in a compact overview.

  If you enable the checkbox **Always use this results report type** in the Results output window and then click on **[verbose]** or **[compact]**, this window no longer opens for the next result printout; the last result report type is automatically used. You can reset this configuration again in the window **Options / Display** (→ "View options" p. 135).
- Continue as described above for "Print complete record".

Print selected data

- Change to the **Overview** tab in the main window.
- Click on in the bottom area of this tab or select the menu item **File | Print | Active window**.
  - ✓ The **Print Overview** window opens.



Print Overview window with selection of the result printout

▶ Select all desired lines and parameters for printing and confirm the selection with [OK].

The window **ASpect PQ Report** opens.

▶ Continue as described above for "Print complete record".

10.1.2 Print further analysis parameters and settings

The following parameters and settings of the analysis can be printed from the respective window:

- Method
- Sequence
- Results data on the tab **Overview**
- Sample ID
- QC (Quality Control charts)
- Calibration
- Autosampler positions

▶ On the Aspect PQ workspace enable the window whose content you want to print.

▶ Start printing the parameters with a click on in the window.

Alternatively, open the menu command **File | Print | Activ Window**.

The window **ASpect PQ Report** opens.

▶ If necessary change the issue format in the list **Output to** and set special parameters of the output format with **[Options]**.

▶ Start the printout with **[Start]**.

10.1.3 Adapting report templates

Use report design mode

The report templates installed by default can be individually adapted. For a better overview report views can be edited with real values.

- ▶ Enable the menu item **File | Report design mode**.
- ▶ Open the window whose report template you want to change.
- ▶ If present, click on there. Alternatively, open the menu command **File | Print | Active Window**.
- ▶ Confirm the query about editing the report template with **[Yes]**. The report designer opens.
- ▶ Make the desired changes and save the changed report template.
- ▶ Link the report template to the corresponding print contents (see "Managing Report templates below).

Short introduction to the report designer

The individual components of the report template are called Objects. A table can thus consist of an object each for the header, the list values and a graph.

These objects again contain the information to be printed and carry the associated layout characteristics such as fonts, alignment, make up, colors etc.

The report designer makes various types of objects available, e.g. text objects, graphs, barcodes. These can be freely placed in the working area and the size can be changed. Depending on type an object can present different information or have different characteristics.

The desired objects are as a rule pulled onto the working area with the mouse and then provided with the relevant contents and layout characteristics. Alternatively you can pull a variable from the variables list onto the working area by "Drag & Drop". If there is still no object at the target position, one is automatically created and the variable is assigned to the object.

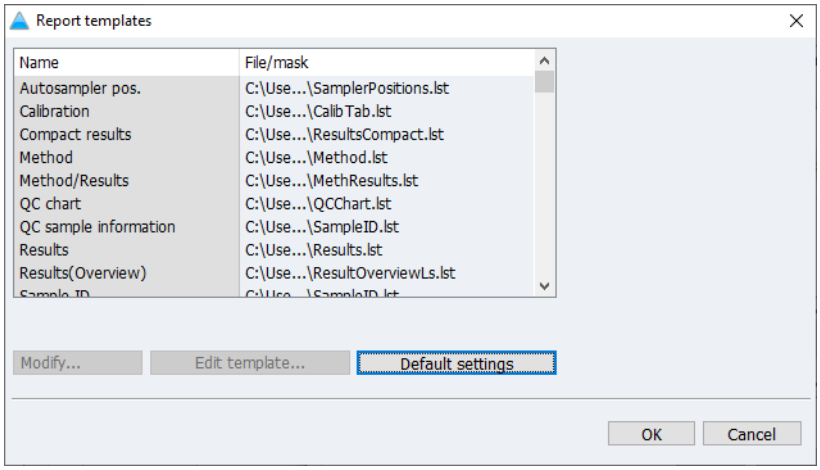
In order to process an existing object, it must first of all be selected. For this click on the object with the left mouse button. You will recognize a selected object by its highlighted frame. If you create a new object it is automatically selected and can be directly changed in terms of size and position. A dialog window is started via a double click with which further settings can be changed.

You will find further information on the operation and functions of the report designer in the manual "designer_deu.pdf" / "designer_eng.pdf" on the ASpect PQ CD.

The Report templates window

In the window **Report templates** the reports are edited and assigned to the windows of ASpect PQ. Several sheets can be assigned to one window by using a file mask, from which the desired report is selected at the start of printing.

- ▶ Using the icon open the window **Data / Reports**.
- ▶ Click on **[Report templates]**.



Window Report templates

For the following windows a report template must be available:

Name	Description
Results	Content of the **Results** tab in the main window
Compact results	Compact overview of the results
Results (Overview)	Content of the **Overview** tab in the main window
Calibration	Analysis calibration - **Calibration window**
Method	Method parameters - **Method window**
Method/Results	Summary report (→ "Printing results data" p. 117).
Autosampler pos.	Autosampler assignement - Window **Autosampler / Positions**
Sample ID	Sample information data – window **Sample ID – Sample information**
QC chart	Data of the QC charts - **QC window**
QC sample information	Information data of the QC samples - window **Sample ID – QC Sample information**
Sequence	Sequence – **Sequence window**

Change assignment

- In the window **Report templates** select the window whose report template is to be changed.
- With **[Modify]** open the dialog window to assign the files.
- If only one report template is to be assigned, enable the option **Use report template** (*.lst) and select the desired file by means of clicking on .
- If several reports are offered simultaneously at the start of printing, enable the option **Allow file selection**. Enter the mask name while using wildcards in the input field.
- Conform the settings with **[OK]**.

Edit report template

- In the window **Report templates** select the window whose report template is to be edited.
- With **[Edit templates]** open the window of the **Report designer**.

Restore defaults

▶ To restore the state after installation of the ASpect PQ program use **[Default settings]**.

Note

Import externally created format templates to the program in the window Data / Data management (→ "Importing report templates" p 129).

10.2 Data management in the window Data / Data management

ASpect PQ manages the arising data in different ways.

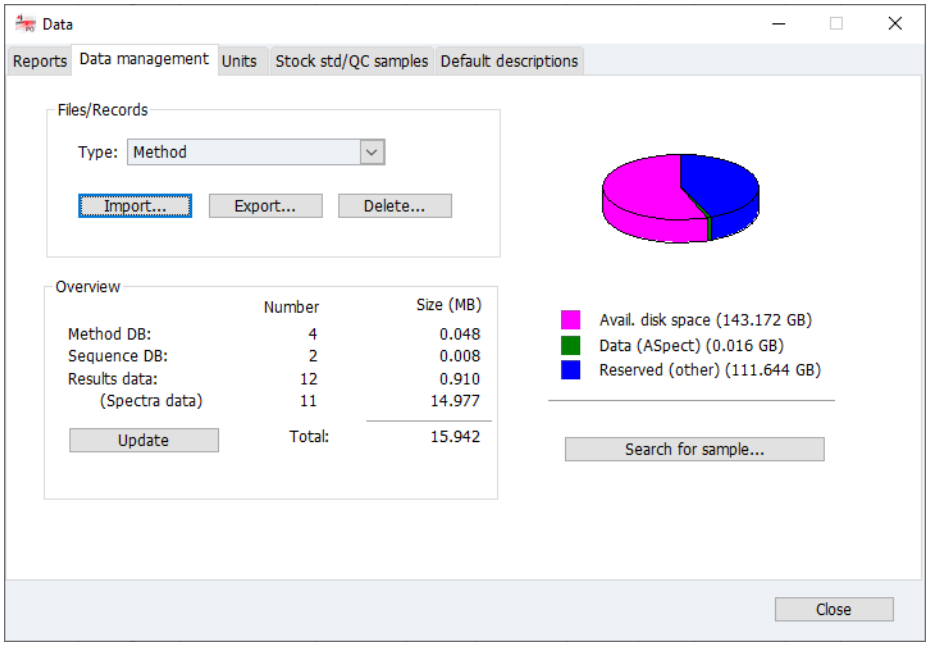
Methods, sequences and correction models are saved separately in a database. The method database is saved as "method.tps". The database holding the sequence data bears the name "sequ.TPS".

For the results obtained by measurements, separate databases are created. Further results can be added to a database by later measurements. Deleting individual samples from a database, however, is impossible. Result databases have the file name extension ".tps".

Sample information data are stored in a table calculation program, e.g. Excel-readable format with the extension ".csv".

Methods, sequences and result data can be organized in the **Data/data management** window. The same dialog functions for the management of methods and sequences are also used for the opening and the saving of these files.

The window **Data / Data management** opens after clicking on or after selecting the menu command **Extras | Data**.



Window Data / Data management

In this window the following data are managed:

- Methods
- Sequences

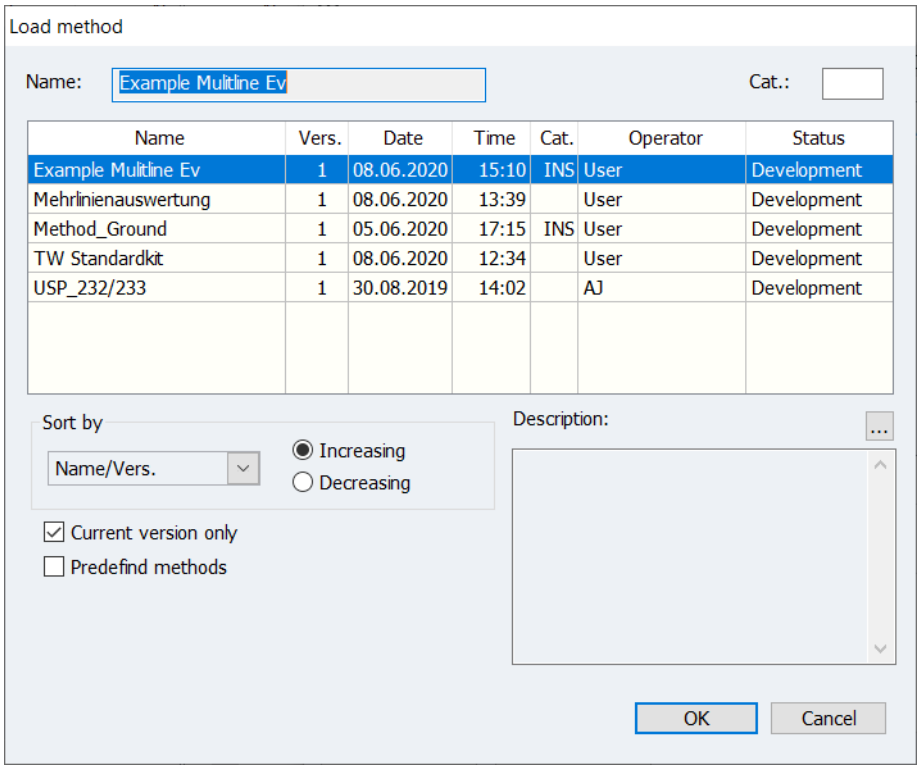
- Results files
- Line/wavelength file
- Correction models
- Correction spectra
- Report templates
- Line favorites
- Worksheets

Choose the desired data type from the **Type** list field.

10.2.1 Managing methods and sequences

The database window for methods and sequences

Methods and sequences are saved to databases. When saving, opening, deleting, importing and exporting methods and sequences, database windows are opened, that have identical elements.



Database window

Displays an input fields

Option/display	Description
Name	Entry or display of the name of the selected method/sequence.
Cat.:	Additional property for the search of the method/sequence in the database. A max. of 3 digits can be entered as category name. You can limit the number of entries to the list by entering a category identifier in the Cat. field. If you want to have the methods/sequences of all categories displayed, delete the entry from the Cat field.

Method list/ Sequence list	Display of the stored methods/sequences with name, version, date, time, category and operator.
Description	Display or entry of additional remarks, e.g. on the use of the method/sequence.
Sort by	Sorts the list of methods/sequences by various criteria, such as name/version or date/time. Depending on the selected option, the entries may be sorted in **ascending** or **descending** order.
Current version only	If several versions of a method/sequence exist in the database, only the method/sequence having the highest version number will be displayed.

Methods/sequences with the same name are not overwritten in the ASpect PQ software. A further version is instead created and the version number is increased by 1.

The databases also provide functions for importing, exporting or deleting individual methods or sequences from the respective databases. In the text of this section, methods and sequences will hereafter be referred to as "data records".

Note
To select several data records in the database window hold down the Ctrl or Shift key during selection with the mouse.

Opening the data management

- Open the window **Data / Data management** by clicking on or the menu item **Extras | Data**.
- In the **Type** list select the data record type to be edited: **Method** or **Sequence**.

Exporting data records

Using the export function, you can make data records accessible to other devices/computers. You may export several data records to a common file. Export files are given the following extensions: method records - ".met", sequence records - ".seq".

- Open the database window with **[Export]**.
- Select the data records and and click on **[Export]**.
- In the default window **Save as** enter a file name and confirm it with **[Save]**.

  The database window with the exported files is displayed.
- Exit the database window with **[close]** and return to the **Data** window.

Importing data records

Using the import function, you can load data records from other devices/computers into your database. An imported file may contain several data records, from which you can select those to be loaded.

- With **[Import]** open the window **Select the file for import** with the default functions for opening files.
- Choose the file to be imported.
- Confirm the selection with **[Open]**.

  This will bring up the database window with the presentation of name, date of creation and category of the data records contained in the file. In the title bar of the window, the name of the import file is displayed.
- Select the data records to be imported in the database window and click on **[Import]**.

✓ The data records are imported into the database. If a method/sequence of the same name should exist already, a new version of the method/sequence will be created. In the database window, the current versions of the available data records appear.

▶ Exit the database window with **[Close]** and return to the **Data** window.

Deleting data records

Using the delete function, you can permanently delete data records from the database.

▶ Open the database window with **[Delete]**.

▶ Select the data records to be deleted.

▶ Click on **[Delete]**.

✓ The database window is updated, displaying only the remaining data records.

For data records of the same name, the version number is reduced by 1.

Deleting data records via the File menu

▶ Alternatively, you can open the database windows **Delete method** or **Delete sequence** with the menu command **File | Delete | Method** or **File | Delete | Sequence**.

▶ Then, proceed as described above.

10.2.2 Managing results files

Results are always saved to the database during the measurement. A database holding result data may be copied or deleted.

▶ Open the window **Data / Data management** by clicking on  or the menu item **Extras | Data**.

▶ In the **Type** list select the option **Results**.

Exporting results files

With the this command, you may copy one or several databases as well as existing spectrum files to another folder.

▶ Click on **[Export]** to open the **Export results files** window with the overview of existing result databases.

The results files are listed with name, size and date/time of the last change.

▶ Select the results databases by mouse clicks. To select several databases, keep the ctrl or shift key depressed.

▶ With the **[Export]** command open the window **Select directory**.

▶ Select the target folder and confirm it with **[OK]**.

✓ The results files are being copied to the selected target folder.

Deleting results files

Delete the result data permanently.

▶ In the window **Data / Data management** click on **[Delete]**.

▶ In the window **Delete results files** select the results database to be deleted by mouse click. To select several databases, keep the Ctrl or Shift key depressed.

▶ Click on **[Delete]** to delete the results database.

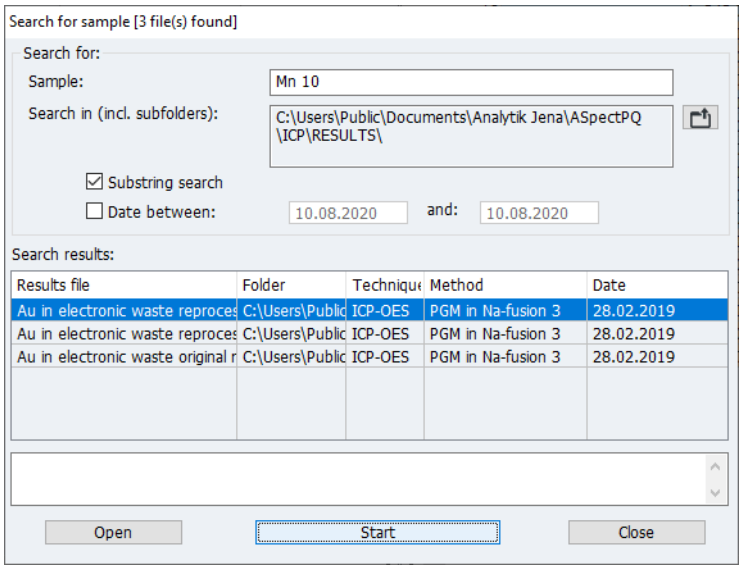
▶ Confirm the query for deleting the files with [OK].

✓ The data are permanently deleted.

Searching for results of individual samples

Individual samples with known sample names can be looked for.

▶ Use **[Search for samples]** to open the window of that name



Window Search for sample

▶ In the **sample** input field enter the sample name.

▶ If you are looking for samples for which the entered string is part of the name, enable the checkbox **Substring search**.

▶ Limit the time of the measurement by enabling the checkbox **Date between**.

▶ Start the search with **[Start]**.

All results which contain samples with the sample name entered are displayed in the table.

▶ In order to open one of the displayed result databases, select this database in the list and confirm with **[Open]**.

✓ The results are displayed in the main window.

10.2.3 Copying the line/wavelength file

The line/wavelength file with the analysis lines and the saved main peaks is device-specific. It has been saved to the computer controlling the ICP-OES device. To use the line/wavelength file on a different computer proceed as follows:

▶ Open the window **Data / Data management** by clicking on or the menu command **Extras / Data management.**

▶ In the **Type** list select the option **Line/Wavelength file** and click on **[Copy]**.

▶ Select a folder to save the file and click on [OK].

✓ The file with the name "lines.dat" is saved in the selected folder.

10.2.4 Managing correction models

Correction models are used for spectral corrections (→ "Removing spectral interference – Spectral corrections " p. 85). They can be transferred from one device to another. Correction model files have the extension ".MOD".

- ▶ Open the window **Data / Data management** with the menu command **Extras | Data management** or by clicking on .
- ▶ In the **Type** list select the option **Correction models**.

Importing correction models

With this command you import correction models to ASpect PQ.

- ▶ Click on **[Import]**.
- ▶ Select the correction model file to be imported and click on **[Open]**.

  The window **Select the correction model file for import** opens.
- ▶ Click on **[Open]**.
  - ✓ The correction model is transferred to the database.

Exporting correction models

With this command you export the correction model for use on another computer.

- ▶ Click on **[Export]**.
- ▶ Select the desired model in the database window **Export Correction model**. Multiple selection is possible.
- ▶ Click on **[Export]**.
- ▶ In the window **Save as** enter the name and storage path and click on **[Save]**.
  - ✓ The file with the correction model is saved.

Deleting correction models

With this command you delete correction models no longer required.

Note

Note that no check takes place whether the correction model is used in a method.

- ▶ Click on **[Delete]**.
- ▶ Select the desired model in the database window **Correction models**.
- ▶ Click on **[Delete]**.
  - ✓ The correction model is deleted from the database.

10.2.5 Deleting correction spectra

Correction spectra no longer required can be deleted from the database.

- ▶ Open the window **Data / Data management** by clicking on or the menu command **Extras | Data.**
- ▶ In the **Type** list select the option **Correction spectra** and click on **[Delete]**.
- ▶ In the database window **Correction spectra** select the spectrum to be deleted and click on **[Delete]**.

✓ A check takes place whether the spectrum is used in a correction model. If this is not the case, the correction spectrum is deleted.

10.2.6 Importing report templates

Print report templates created externally must be imported to ASpect PQ via the data management:

- ▶ Open the window **Data / data management** with the menu command **Extras | data management** or the icon .
- ▶ In the **Type** list select the option **Report templates** and click on **[OK]**.
- ▶ In the **Open** window select the file and click on **[Open]**.
  Report files have the extension ".lst".
  - ✓ The report template is imported to ASpect PQ. Now assign the report template to the print content (→ "Adapting report templates" p. 121).

10.2.7 Managing line favorites

Line favorites can be defined in the **Method** window (→ "Defining own line favorites" p. 31). They contain the analysis line used for a specific application and the line-dependent method parameters. Line favorite files have the extension ".fav".

- ▶ Open the window **Data / Data management** by clicking on or the menu command **Extras | Data**.
- ▶ In the **Type** list select the option **Favorites**.

Importing line favorites

With this command you import a favorite record ASpect PQ.

- ▶ Click on **[Import]**.
- ▶ In the database window **Favorites details** click on **[Import]**.
- ▶ Select the line favorite file to be imported and click on **[Open]**.
  - ✓ After a query the favorite record is added to your line favorites.

Exporting line favorites

With this command you export a line favorite record for use on another computer.

- ▶ Click on **[Copy]**.
- ▶ In the database window **Favorites details** select the desired record. Multiple selection is possible.
- ▶ Click on **[Export]**.
- ▶ In the window **Target file** enter the name and storage path and click on **[Save]**.
  An already existing file can also be used as target file. In this case the record will be integrated there.
  - ✓ The file with the record of line favorites is saved.

Deleting line favorites

With this command you delete line favorites no longer required.

- ▶ Click on **[Delete]**.
- ▶ In the database window **Favorites details** select the record.

▶ Click on **[Delete]**.

✓ The selected record is deleted from the database.

10.2.8 Importing and exporting worksheets

You can import and export worksheets. Optionally, you can specify the methods and sequences stored.

▶ Open the **Data / Data Management** window with the menu command **Extra ▸ Data Management** or the icon  .

▶ In the **Type** list, select **Worksheet**.

Export Worksheet

▶ Click on **[Export]**.

▶ In the **Export Worksheet** window, select the relevant worksheet. To export the methods and sequences as well, activate the **including sequence and method(s)** option.

▶ Click **[Export]** and enter a folder and name for the export file.

▶ Confirm the entries with **[Save]**.

✓ The worksheet is exported with the extension ".WST".

Importing the worksheet

▶ Click on **[Import]**.

▶ In the **Import Worksheet** window, select **[Import]**. To import the methods and sequences exported with the exported methods and sequences as well, activate the option **including sequence and method(s)**.

▶ Select the worksheet in the standard window and click on.

✓ The worksheet is imported.

10.3 Saving results in ASCII/CSV format

You may save measurement results both automatically and manually in ASCII/CSV format. For both export formats, the parameters for the decimal and list separator, and the result columns to be exported are to be defined in the **Options - ASCII/CSV Export** window (→ "Export options" p. 137).

Automatic continuous data export

With activated automatic continuous data export option, every entry in the results table is instantly exported to the defined ASCII file. The name of this ASCII file is to be defined in the **Options / Continuous ASCII Export** window (→ "Options for continuous ASCII export" p. 137).

Manual data export

If you intend to export data manually, you can select the rows of the results table to be exported.

▶ Select the samples in the desired results table.

Holding the ctrl or shift key depressed, select the samples by mouse clicks on the respective rows.
To select all rows of the table, use menu command **Edit / Select All**.

- Then, activate menu command **Edit ▸ Save selection** to open the **Save As** standard window for saving files.
- Enter the file name and confirm it with **[OK]**.
  The data will be saved in the CSV format.

10.4 Specifying units of measurements

You can define the units of measurement available throughout the application in the **Data / Units** window.

- Open the window **Data / Units** by clicking on  or select the menu point **Extras | Data**.

3 preferred versions (for solutions: mg/L, µg/L, ng/L; for solid samples: mg/kg, µg/kg, ng/kg) are available. These units cannot be changed by the user. Units deviating from these can be freely defined. For freely defined units it is necessary to enter the conversion factor under Factor:

Option	Description	
Unit	Name of the unit (max. 10 characters)	
Comment	Remarks (max. 20 characters)	
Factor	Factor 1 corresponds to 1 µg/L or µg/kg, factor 1000 corresponds to 1 ng/L or ng/kg	
Type	**solid**	Unit related to solid sample
	liquid	Unit related to liquid sample (solution)
	liquid grav.	Unit related to liquid sample to be weighed, e.g. oil

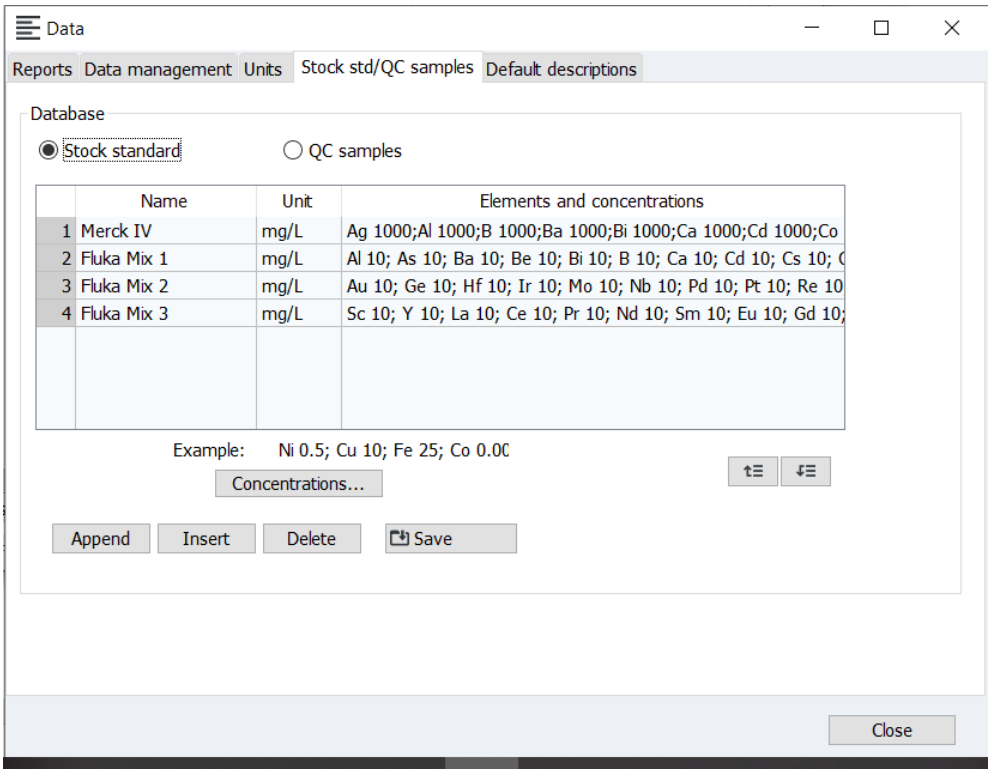
The **[Append]** and **[Insert]** buttons serve to append user-defined units to the end of the list or insert them above the currently selected row. The **[Delete]** button only deletes user-defined units, the preferred default units, however, cannot be deleted. Use **[Save]** to permanently save any changes.

10.5 Managing databases for stocks and QC samples

The databases with the frequently used stock standards and QC samples are entered in the window **Data / Stock std./QC samples**. These frequently used single and multiple element standards are then available throughout the application.

- Open the window **Data / Stock std./QC samples** by clicking on  or select the menu point **Extras | Data**.

- By enabling the options **Stock standard** or **QC sample** you select the display in the table.



Window Data / Stock std/QC samples

Table column	Meaning
Name	Name of the standard (enter max. 20 characters).
Unit	Select the name of the unit (max. 10 characters) for the standard.
Elements and concentrations	Element concentrations are entered in the format "Element symbol concentration" in the selected unit, e.g. Fe 0.5; Cu 10; Co 0.005. Alternatively, use **[Concentration]** to open the input field of the same name where you can assign the concentration to each element.

The buttons have the following functions:

Button	Function
[Append]	Appends a new row to the end of the list.
[Insert]	Inserts a row in the list above the currently selected row.
[Delete]	Delete the marked row.
[Save]	Save the lists of the stock standards/QC samples.
[Concentration]	Opens the input window for the entry of the element(s) and concentration(s) of the selected standard.

10.6 Managing default descriptions

User-defined remarks can be defined for the following actions. These can then be selected via the [...] button and are transferred to the field **Description**:

- save method
- save sequence
- start reprocessing
- start measurement

Use the EDIT TEMPLATE button to create, modify, or delete predefined descriptions or comments.

Creating Comment

- Open the window **Data / Default descriptions** with the menu command **Extras ▸ Data management** or the symbol  .
- Select the operation from the **Select category** list.
- Open the list of remarks by clicking on **[Edit template]**.
- Create a new comment by clicking on **[New]**. Enter a **Name** under which the remark can be selected. Enter the actual comment in the **Text** field.
- A comment can be edited via **[Change]** or removed from the selection list via **[Delete]**.

10.7 Using Windows clipboard

Copying results to the clipboard

The application lets you copy the results of selected samples directly to the Windows clipboard thus making them accessible to other Windows applications.

The corresponding commands are found in the **Edit** menu:

Edit menu | ...	Description
Copy visible Columns only (Ctrl+C)	Copies the visible sample results of the current table.
Copy all Columns	Copies the sample results of all tables.
Column titles	If activated (check mark), the copy action includes the column headers.

▶ Select the samples from the desired table of the results list.

Holding the ctrl or shift key depressed and select the samples by mouse click on the sample row.

To select all rows, use the menu command **Edit | Select all**.

▶ If necessary, activate menu command **Edit | Column Titles** to have the column header included in the copy action.

▶ Activate the desired menu command to copy the results to the Windows clipboard.

Copying graphics as screenshots

You may also copy graphic windows and graphs of calibration curves, intensity signals or emission signals as screenshots to the Windows clipboard.

- ▶ Click on the right mouse button on the graph. A context menu with two copy commands opens.
- ▶ Select the copy command to copy the desired object: copy only the graph or the entire displayed window.

The selected object is copied onto the clipboard and is available for other Windows applications.

11 Customizing ASpect PQ

In the **Options** window, you can make the following settings which apply to all operations in ASpect PQ:

- View options
- Save paths of files
- Parameters for data export
- Generally applicable settings for the analysis sequence

The settings made remain active after exiting and restarting ASpect PQ.

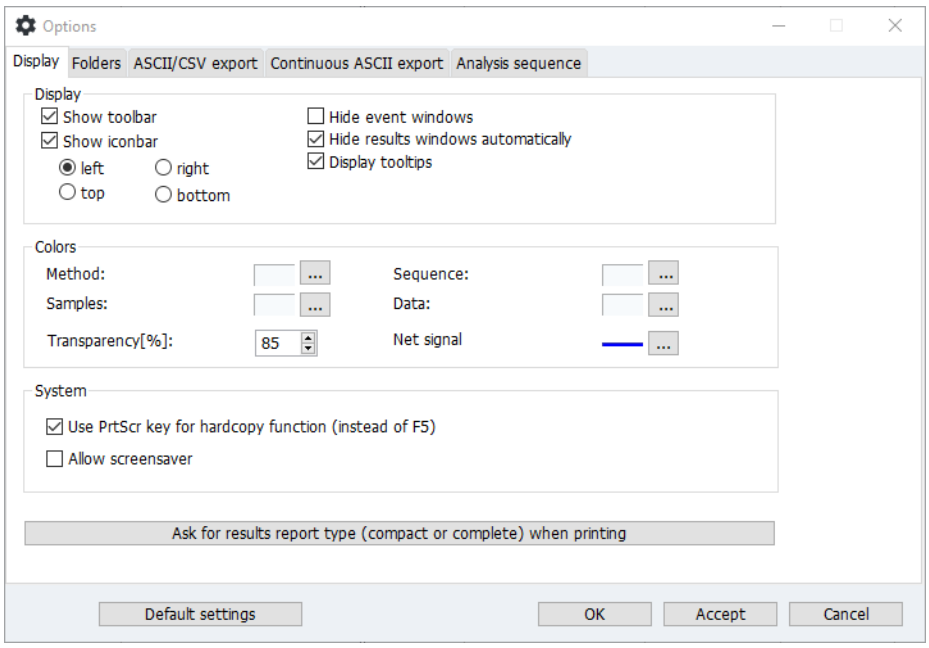
Opening the Options window

The **Options** window you can open the menu command **Extras | Options**.

11.1 View options

In the **Options / Display** window define the functions and elements to be visible on the workspace.

▶ Open **Options / Display** window with the menu command **Extras | Options**.



Window Options / Display

Option	Description
Show toolbar	Display the toolbar with the buttons for the measurement routine.
Show icon bar	Display the icon bar with the large buttons for fast access and select the icon bar position. (The position of the icon bar can also be modified by dragging with the mouse, but the setting will not be saved beyond the next program start).
Hide event window	Hide the event window (e.g. **delay time**). The messages are instead shown in the status bar of the main window.
Hide results windows automatically	Results windows are hidden if sub-windows (e.g. **Method** window) are opened. After closing the sub-windows the result windows are displayed again.
Display tooltips	Displays brief help texts (tool tips) with mouse-over on all icon buttons and for the column headers in the windows **Method**, **Sequence** and **Sample ID**.
Lists and signal colors	The button ••• opens the color selection dialog. There, you may choose predefined or newly defined colors as list background.
Use PrtScr key for hard copy function (instead of F5)	By default, the printout of the screenshot is started with [F5]. In this case, the **[PrtScr/Druck]** key of the keyboard is used for the Windows clipboard function. If this checkbox is enabled, the **[PrtScr/Druck]** button starts the printout of the screenshot. This option only becomes active after restarting ASpect PQ.
Allow screensaver	If enabled, the Windows screensaver activates during input pauses.
Ask for results report type (compact or complete) when printing using menu	When printing result windows via the menu item **File** | **Print** | **Active window** it is possible to choose between a complete and compact report. Clicking on this button resets the selection **Always use this results report type** again and allows for the report type to be selected again.

The button **[Default settings]** resets all currently set options and window positions to default values.

11.2 Storage paths

During the installation the storage paths for data are defined. These are displayed in the window **Options / Folders** and can be partially edited here.

▶ Open **Options / Folders** window with the menu command **Extras | Options**.

Folder	Description
Program	Installation path for executable program files.
work directory	Directory for user data The work directory contains additional sub-folders. It is defined during installation or by the optional user administration.

temporary data	Directory for temporary application files.
Sample information	Default path for opening and saving sample information files. This path can be modified. Click on **[...]** to select the new folder. A different path can also be selected during the opening and saving of sample information files.
Export/Import	Default path for export and import of method and sequence data and export of result data as CSV files. Click on **[...]** to select the new folder. During export and import a different path can also be selected.
Results	Directory for results data This default directory may contain additional sub-folders for result storage. These folders are available for saving results files at the start of measurements.
Application data	Directory for data in which ASpect PQ stores necessary data.

With the **[Add]** button you can create new sub-folders to the Results folder for saving results. Besides, it is possible to delete and rename empty folders here.

11.3 Export options

In the window **Options / ASCII/CSV-Export** you can define the parameters for exporting results to ASCII files. The parameters apply to both forms of the data export, for the automatically continuous and also the manual export (→ "Saving results in ASCII/CSV format" p. 130).

- Open **Options / ASCII/CSV-Export** window with the menu command **Extras | Options**.

For exporting the results lists select the **Decimal separator** and the **List separator.**

In the **Result fields for export** area you can define which columns of the result table shall be exported to the ASCII file. **All** Exports all columns of the results list (with all sub-tabs). The option **only selected fields** opens a list in which the columns to be exported can be selected.

[Default settings] resets all currently set options and window positions to default values.

11.4 Options for continuous ASCII export

In the window **Options / Continuous ASCII export** the automatic export of results data during the analysis sequence is enabled. The export file is updated respectively after the output of a new line in the process and result window . The result data will be appended to already existing files.

Further export options may be defined in the window **Options / ASCII/CSV-Export**.

- Open **Options / Continuous ASCII export** window with the menu command **Extras | Options**.

Export of results data

The checkbox **Continuous ASCII export** activates the export function. Next an option for the file name must be selected:

Option	Description
"Method name".csv	The file name corresponds to the name of the method. The file name extension is ".csv". The file is saved to the default export/import path (window **Options / folders**).
"**Results file name".csv**	The file name corresponds to the name of the results file. The file extension is ".csv". The file is saved to the default export/import path (window **Options / folders**).
other	You may freely define file name and save path. The button [...] opens the default window **Save as** to issue a storage path and a file name. The data will be continuously written to this file until a new name is given or another naming option is selected.
Create separate file for each sample (result row and sample name is appended to filename)	The file name is appended with the row number of the results list and the sample name. Impermissible characters are replaced by underscores (e.g. Testmethod-001 QC 1 mg_L.csv).

Spectral export

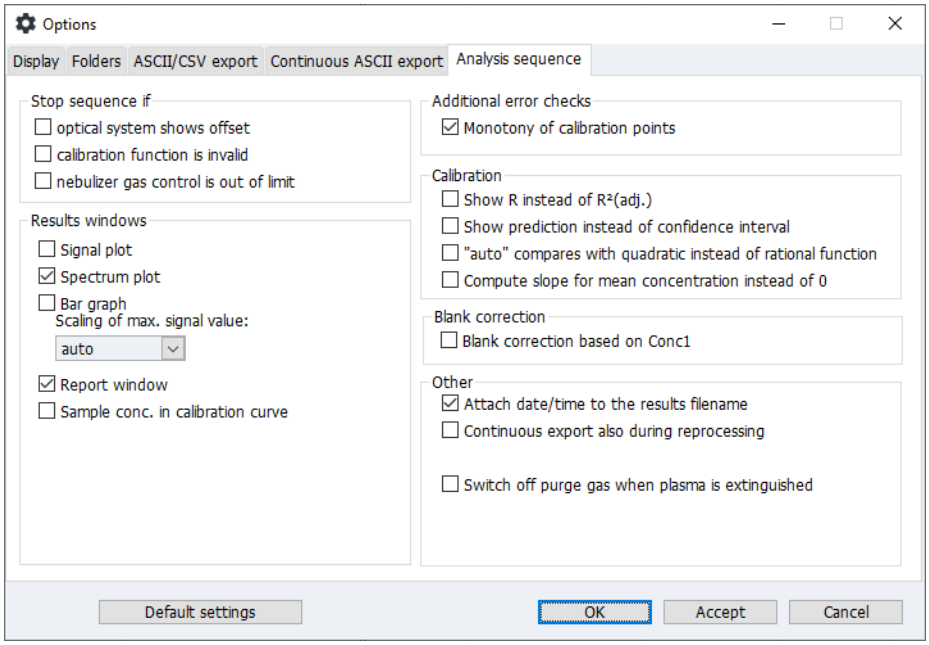
For the spectral export enable the option **Continuous export of spectra (CSV)** and select a storage path.

The spectra are additionally exported as CSV files to the specific export path. The file name is generated based on the schema "ListRow-SampleName-LineName-RepeatMeasurement", e.g. 0007-sample-AI309-02.csv.

11.5 Options for analysis sequence

In the window **Options / Analysis sequence** you can define general options for the analysis sequence.

- Open **Options / Analysis sequence** window with the menu command **Extras | Options**.



Window Options / Analysis sequence

Stop after following errors

The analysis is monitored for the following errors and can be canceled if these errors occur:

Option	Description
optical system shows offset	Stops if the wavelength configuration (Ne correction) is faulty.
calibration function is invalid	Stops if the calibration function could not be calculated.
nebulizer gas control is out of limit	Stops when the atomizer control value is exceeded. During calibration, the atomizer flow control value is determined. If the control value changes during the following analysis, this is an indication that particles are clogging the nebulizer.

Additional error checking

Option	Description
Monotony of calibration points	The calibration points will be tested for monotony. The monotony test serves to determine if higher standard concentrations also lead to higher measured values.

Results displays

Option	Description
Signal plot	A window is displayed during the analysis process showing a graph of the measured signal as a function of time.
Spectrum plot	A window is displayed during the analysis process showing a graph of the recorded spectral range.
Bar graph	Displays measured intensities as a bar graph.

Scaling of maximal signal value	Defines the maximum of the measurement value axis for the presentation of the signal curve. **auto:** Automatic axis scaling. Alternatively, this setting can also be made using the menu function **View | Scaling**.
Report window	A window is displayed during the analysis process showing status information about the plasma.
Sample conc. in calibration curve	Displays the window **Sample conc. in reference graph** with the current calibration, and if measured already, the recalibration graph. After the measurement of the sample, the calculation of the uncorrected concentration from the emission is highlighted by red auxiliary lines. If addition calibration is used, the converted calibration curve will be displayed.

Calibration

On this tab you make basic settings for the calibration. All checkboxes are disabled as default.

Option	Description
Show R instead of R2 (adj.)	If enabled, the correlation coefficient is displayed. By default the corrected (adjusted) coefficient of determination is provided.
show prediction instead of confidence interval	If enabled the prognosis band for the calibration is displayed. The confidence band is provided as default.
"auto" compares with quadratic instead of rational function	"auto" designates the automatic selection of the calibration function (→ "Entering calibration parameters – Calibration tab" p. 39. If enabled the quadratic function is used for the comparison. The default setting is the broken ratio function.
Compute slope for mean conc instead of 0	If enabled the slope of the calibration graph is calculated for the mean concentration of the calibration range. As default the slop is calculated for 0 concentration.

Note

All options mentioned above must be enabled for compatibility of the calculation of the quadratic calibration function in accordance with DIN 38402 and ISO 8466-2.

Options for blank correction

For the blank value correction it is possible to choose between 2 different calculation methods: Conc.1-based or Conc.2-based.

With the Conc.2-based calculation method, the original concentration of the blank value ($conc2_{Blank}$) is first calculated based on the sample IDs of the blank value. $conc2_{Blank}$ is used to determining the $conc2_{Sample}$ of the sample.

In the Conc.2-based calculation method, the concentration ($conc1_{Blank}$) determined directly from the measured value of blank sample is used for the calculation of the sample concentration. This procedure can be used if the sample ID data (e.g. dilutions) do not strongly influence the concentration of the blank value solutions and therefore no sample ID data are entered for the blank values.

Calculation example for liquid original sample with pre-dilution:

Conc.1-based: $conc2_{Sample} = (conc1_{Sample} - conc1_{Blank}) * DF\_sample$

Conc.2-based: $conc2_{Sample} = (conc1_{Sample} * DF\_sample) - conc2_{Blank}$

$Conc1_{Sample}$	Concentration of the sample without consideration of the information in the sample ID
$Conc2_{Sample}$	Original concentration of the sample
$Conc1_{Sample}$	Concentration of the blank without consideration of the information in the sample ID
$Conc2_{Sample}$	Original concentration of the blank
DF_{Sample}	Dilution factor of the sample

For the blank value correction, the Conc.2-based method is preset by default. If you want to use the shortened Conc.1-based method without considering the sample ID of the blank value, activate the option **Blank value correction based on Conc.1**.

Other

Option	Description
Attach date/time to the result file name	Current PC/time at the start of measurement is automatically appended to the name of the result file.
continuous export also during reprocessing	After reprocessing the results are automatically exported.
do not update time stamp when reprocessing	After reprocessing of the results the original measurement times are retained.
Switch off purge gas when plasma is extinguished	To save gas the purging gas is switched off if the plasma is extinguished.

12 Optional Modul 21 CFR Part 11 Compliance ASpect PQ

An optional Modul 21 CFR Part 11 Compliance ASpect PQ includes the following functions according to the FDA requirements for Electronic Records and Electronic Signatures (21 CFR Part 11). It provides extended functionality for operation of ASpect PQ in accordance with the FDA requirements on Electronic Records & Electronic Signatures (21 CFR Part 11).

- User management
- Electronic signatures
- Audit trail
- AJ File Protection to protect files against intentional and unintentional data manipulation

The user management provides one administrator level and four user levels. The following functions are accessible for a user with administrator rights:

- Flexible system configuration (code word and login gidelines, audit trailing, signatures, data directories)
- Creation of a user level for each user with a stepped pattern of user rights
- Assignment of passwords for access to ASpect PQ software
- Assignment of personalized working directories for methods, sequences and results to users
- Inspection and exporting of created audit trail (events log sheet)

Once the user management package is installed and configured, the **System** menu item in ASpect PQ will be active. You can use this menu item to access one or more desired functions of user identity management.

Any change in user data will be permanently saved in an encoded data base on exiting a pertaining window.

Note

In order to meet safety requirements, a Microsoft Windows operating system with adequate configuration resources must be available. This applies to file access rights and other setting actions of a kind that should be performed by an authorized system administrator.

12.1 User management

12.1.1 Hierarchy and access to functions

The user identity management package provides for one administrator level and four user levels.

The hierarchy structure for user levels is as follows:

Administrator > level 1> level 2 > level 3 > level 4.

Administrator level

The user has full access rights to ASpect PQ and to any function of user identity management.

Level 1

Level 1 users have unlimited access to all Aspect PQ functions, but are denied access to user identity management.

Level 2

Same as level 1 users, except:

- Deletion of methods (M1 ID code)
- Deletion of sequences (P1 ID code)
- Deletion of QC rule tabs (Q1 ID code)
- Deletion of results files (R1 ID code)

An ID code is used in advisory notes to operators.

Level 3

Same as level 2 users, except:

- Saving of methods (creating methods in a method data base) (M2 ID code)
- Saving of sequences (creating sequences in a sequence data base) (P2 ID code)
- Accepting of peak offsets (W1 ID code)

Level 4

Same as level 3 users, except:

- Changes in method parameters (E1 ID code)

(users of this category can only load previously created methods and sequences and perform measurement).

Function	ID code	Admin.	Level 1	Level 2	Level 3	Level 4
Work with user ID management		+	-	-	-	-
Delete methods	M1	+	+	-	-	-
Delete sequences	P1	+	+	-	-	-
Delete QC rule tabs	Q1	+	+	-	-	-
Delete results files	R1	+	+	-	-	-

Save methods	M2	+	+	+	-	-
Save sequences	P2	+	+	+	-	-
Make changes in peak offsets	W1	+	+	+	-	-
Make changes in report templates	L1	+	+	-	-	-
Make changes in methods	E1	+	+	+	+	-
Load methods and sequences		+	+	+	+	+
Perform measurement		+	+	+	+	+

*ID code is used in operating advice.

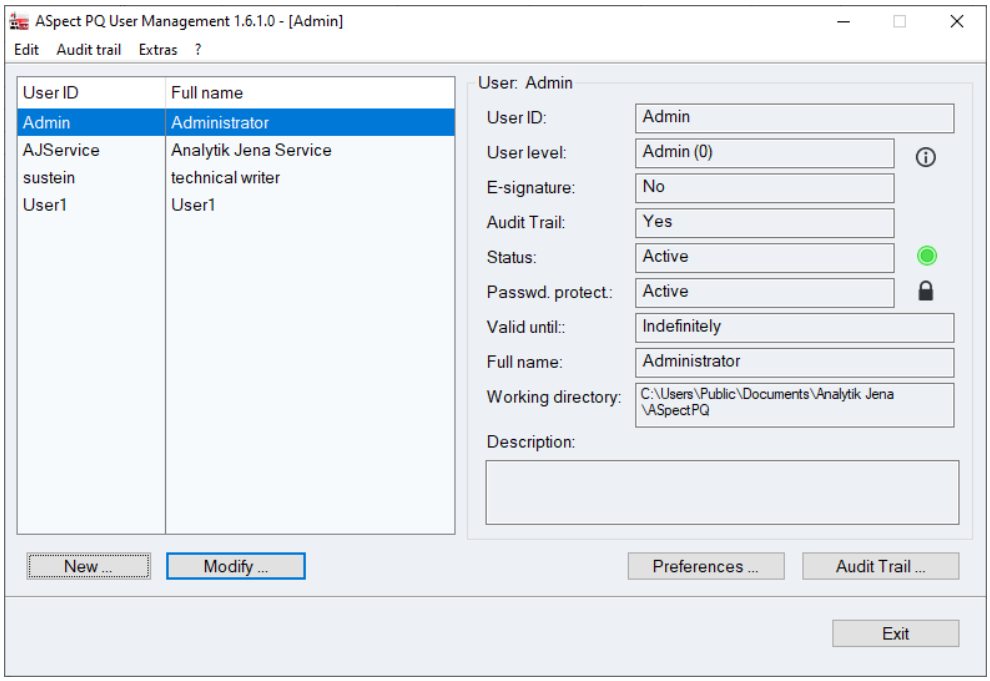
12.1.2 User Management setups

The user management can be set up at the first start of the user management after installation or at a later time by users with ASpect PQ administrator rights.

For each user, an account is created in which the user profile is stored. If a user account is no longer needed, it can be deactivated or locked. User accounts cannot be deleted.

- Select the menu command **System | User Management** or start the user management via the entry in the Windows start menu.
- Log on with an administrator profile.

  ✓ The **User Management** window opens.



User management window

The window contains a list of registered usernames with assigned passwords. It shows the user profile details for a selected user in its right-hand subarea.

Indicator and control elements

Option	Description	
UserID	Login name of user	
User	Administrator, level 1 to level 4	
E-signature	**Yes**:	User is authorized to electronically sign result data.
	No:	User has no authorization for electronic signature.
Status	**Active**:	Username allowed for use (green circle).
	Disabled:	Username was disabled and cannot be used (red circle).
Password protection	**Active**:	User login requires a code word (key)
	Inactive:	User login allowed without a code word (key crossed out)
Valid until	**Unlimited**:	Password never expires.
	Date/days:	User must change his/her password on expiry of specified term.
Full name	Complete name of user	
Description	Optional description of user	

Control buttons

Button	Description
[New ...]	Creates new user. Opens the **Add user data** window.
[Modify ...]	Changes user data for a marked table line. The **Modify user data** window will open for a marked table line. The same dialog screen may be called up by double-clicking the particular table line.
[Preferences...]	Allows changes in user management configuration.
[Audit trail....	Audit trail (events logsheet)
[Exit]	Terminates this application.

12.1.2.1 Configuring user management

For configuration, the **Preferences** window provides selection options as follows:

- Password policies
- Login and audit trail
- Signature meanings
- Selected data directories

▶ Click onto **[Preferences...]** in ASpect PQ **User Management** window.

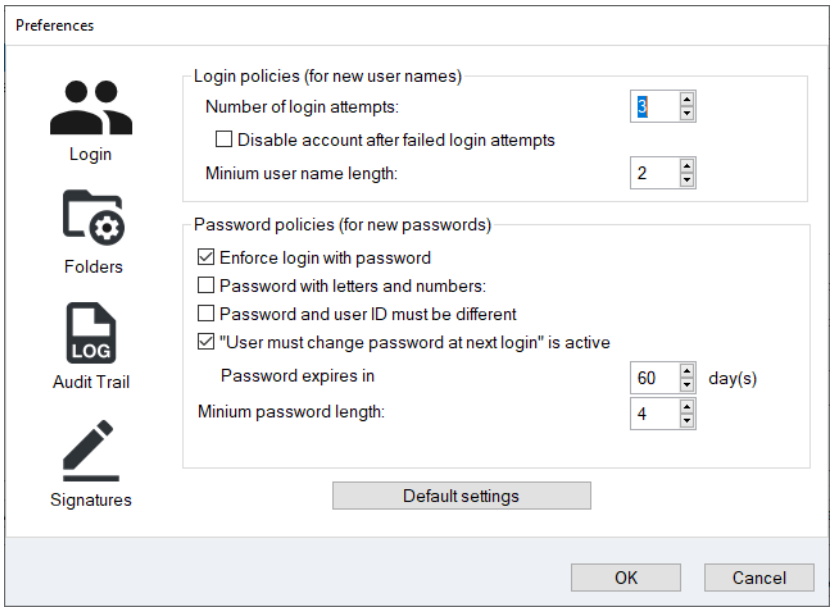
The **Preferences** window opens.

▶ Select an option group you want to change from the left column.

The settings apply to newly created user accounts and should therefore be carried out after installation. When you close the window with [OK], all settings are saved, when

you press [Cancel], the changed settings are discarded. You can use the [Set to Defaults] button to restore the default values for a selected option. The settings of the other areas are not affected.

Login/password



Setups for login and password policies

Login policies (for new usernames)

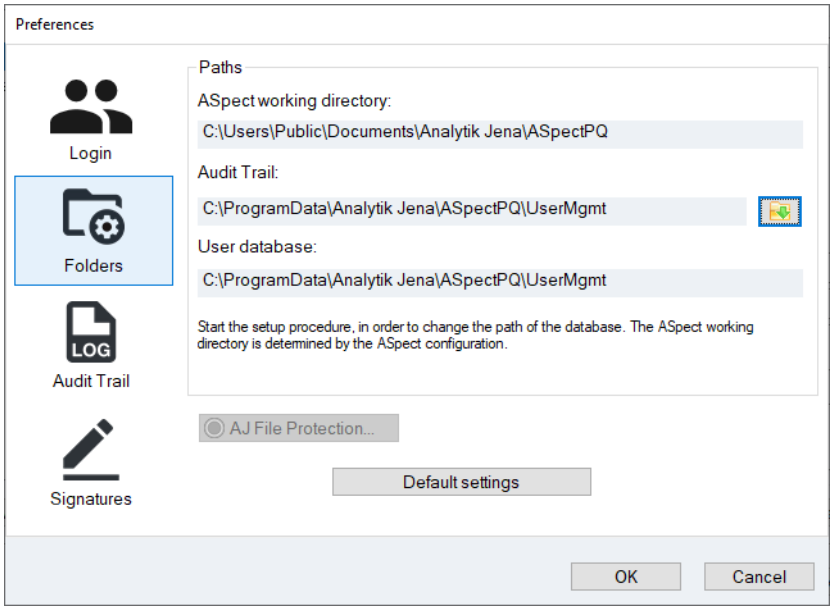
Button	Description
Number of login attempts	Shows the number of invalid login attempts (max. 10). On excision of the maximum allowed number, the ASpect PQ session will be terminated after a certain waiting time and must be restarted for another login. An entry (warning) is added to the audit trail file.
Disable account after failed login attempts	If enabled, the user will be locked after exceeding the number of login attempts.
Minimum username length	Defines the minimum prescribed number of characters for a newly created username Maximum number of characters: 10

Password policies (for new usernames)

Option	Description
Enforce login with password	A password must be assigned to newly created usernames.
Password with letters and numbers	Only passwords consisting of letters and numeric characters can be assigned. This policy equally applies to changes in password.
Pasword and user ID must be different	Only passwords which are different from the respective username will be accepted. This policy equally applies to changes in password.

"User must change assword at next logon" is active	For newly created users, the **Change password at next logon** checkbox will be turned on by default. The user will be prompted to change his/her password when logging in to ASpect PQ for the next time.
Password expires in ...day(s)	On expiry of a preset term, the user will be prompted to change his/her password when logging in to ASpect PQ. The password is then extended by a term as set in Policies. This value is then acknowledged as a template that can be modified for other single users (max. 999 days).
Minimum length of password	Designates the minimum required number of characters for a newly created password; number of characters: 3 to 10

Folders



Setups for paths

The ASpect-PQ working directory and the audit trail directory provide specification options.

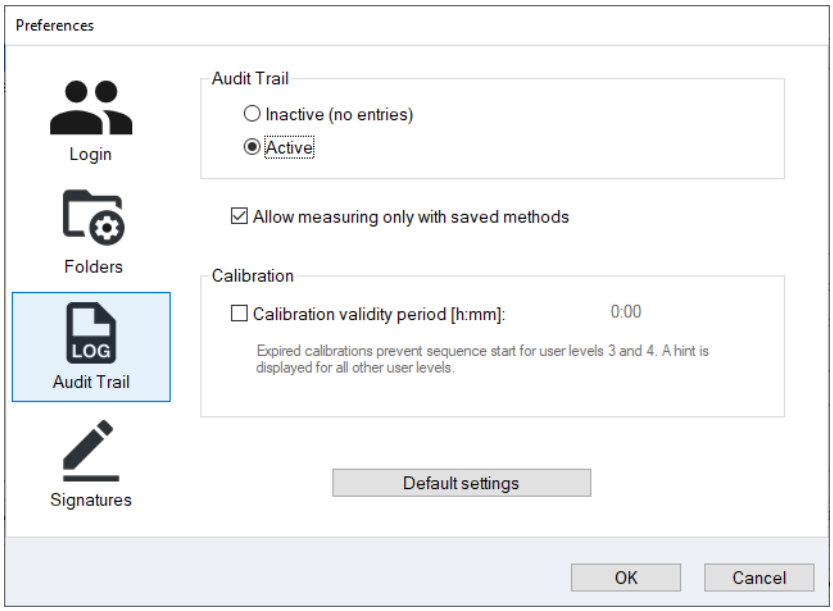
Directory	Description
ASpect-working directory	Working directory of Aspect PQ The working directory contains a data base of methods and sequences and results files. The working directory was defined as part of ASpect PQ installation and may be changed at this point.
Audit Trail	Path of audit trail file. This path may be changed.
User database	Path of user data base. This path may only be changed with the help of the installation program.

Additional protection is offered by the optional software **AJ File Protection**.

This software protects files against any intentional and unintentional manipulation, e.g. by deleting or modifying data.

When AJ File Protection is installed, the button is active and displays the protection status. Green – File protection is active; Red - File protection is inactive. Clicking on the button shows a list of protected.

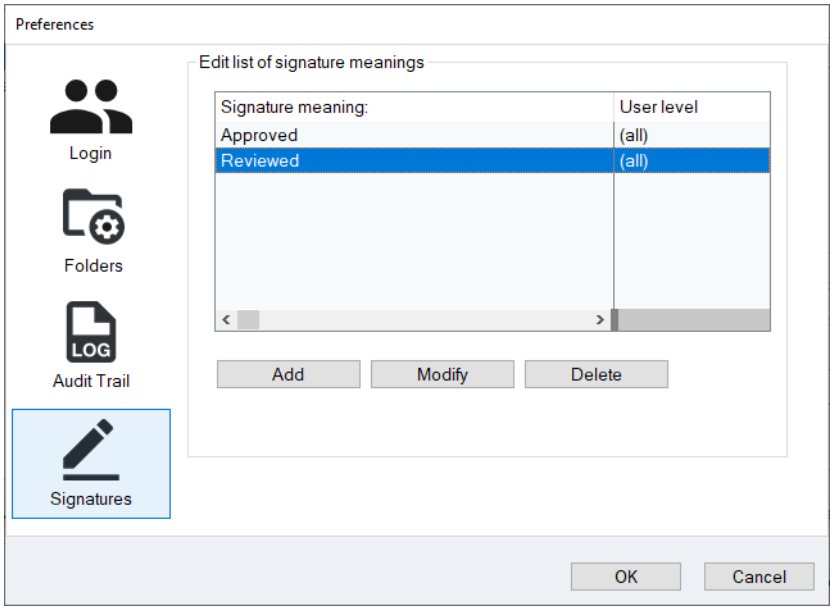
Audit Trail



Settings for audit trail data

Option	Descriptions
INACTIVE (NO ENTRIES)	No entries will be added to the audit trail file (event log sheet).
ACTIVE	Entries will be added to the audit trail file (event log sheet).
ALLOW MEASURING ONLY WITH SAVED METHODS	When active, this checkbox allows a measurement to be triggered in ASpect PQ only if a method was loaded and no change was made in this method since it was saved for the last time.
CALIBRATION VALIDITY PERIOD [H:MM]	When active, the validity period of the calibration can be specified. For user levels 3 and 4 the calibration must be updated before starting the measurement sequence. For all other levels a hint is displayed.

Signatures



Editing of signature meanings

This list shows those signature meanings that are selectable for signing.

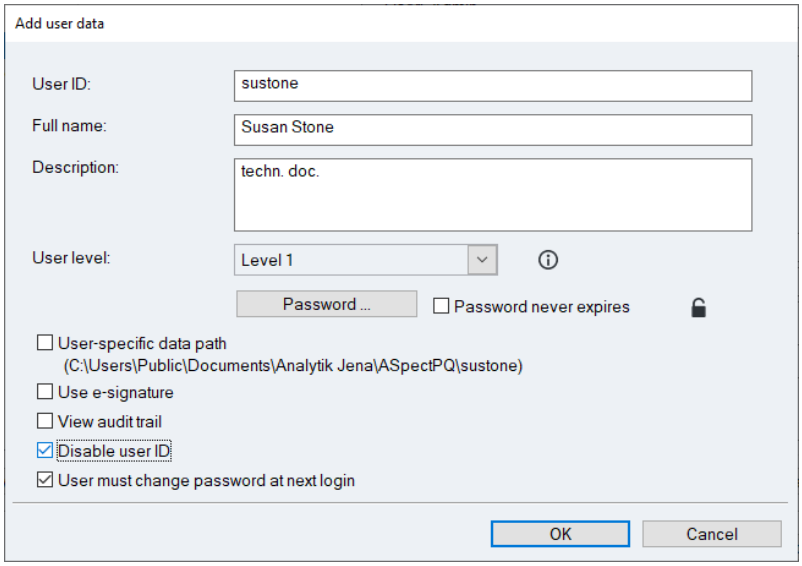
Button	Description
Add	Adds a signature meaning, e.g. created, reviewed, approved (max. 30 characters).
Modify	Changes a signature meaning that was marked in the list.
Delete	Deletes a signature meaning that was marked in the list.

12.1.2.2 Creating a new user account

Only users with administrator access rights are authorized to create a new user account.

- Click the **[New]** button in the **User Management** window, in order to create a new user account.

  The **Add user data** window appears.



Editing of user data

The input fields in this window provide setting options as follows:

Option	Description
User ID	The name that is required for a user to log in. No checks are made for capital and small lettering. The minimum name length depends on configuration settings made in **Login/Password** (→ "Configuring user management" p. 145). et seq.). Max. length: 32 characters
Full name	Full name of user. This name will serve as a constituent of the electronic signature. Max. length: 30 characters
Description	A field for note or comment text. Entry is optional.
User level	User level
Password...	Opens a dialog screen for password entry. Max. password length: 20 characters The minimum length and other password policy rules may be configured. Capital lettering and small lettering are distinguished for passwords. If the password dialog is acknowledged without a password entry, the password protection will be canceled. **Lock** icon: Password protection is active. **Unlock** icon: Username uses no password.
Password never expires	Password will remain valid for unlimited time if this box is active. If it was disabled, the given password will expire within a preset term. The specified value is sourced from password policies. A user may also extend his/her password in advance.
User-specific data path	If active, this box will create the user's own working directory in this format: \ASpect-Working directory\Username. As part of an initial login to ASpect PQ the appropriate directory structure will be created.
Use e-signature	Allows the user to electronically sign for measured results if active.
View audit trail	Allows user to view the audit trail.
Disable user ID	Prohibits the use of a username if active. Usernames can be temporarily disabled. A disabled (as opposed to a removed) username cannot be reassigned to another newly created user.
User must change password at next logon	Will prompt the user to change his/her password as the next ASpect PQ session is triggered.

▶ Click **[OK]** to confirm all features of a newly created user account.

12.1.2.3 Modifying a previously created user account

▶ To select a user account you want to modify, click this account in the table of the **User Management** dialog window, then click the **[Modify]** button.

The **Edit user data** window with selected accounts will open (→ "Creating a new user account" pg. 149).

12.1.3 Changing a password

Depending on his/her user account settings, a user may or may have to change his/her password at regular intervals.

▶ Select the **System | Change password** menu command in Aspect PQ.

The **Change password** dialog screen opens.

▶ Enter your previous password at the input field, then enter the new password twice and click **[OK]**.

✓ On successful entry, a “Password changed“ message will appear.

12.2 Viewing, printing and exporting Audit Trail

An audit trail file keeps track of certain system events and any warning or error message that occurred during an ASpect PQ session.

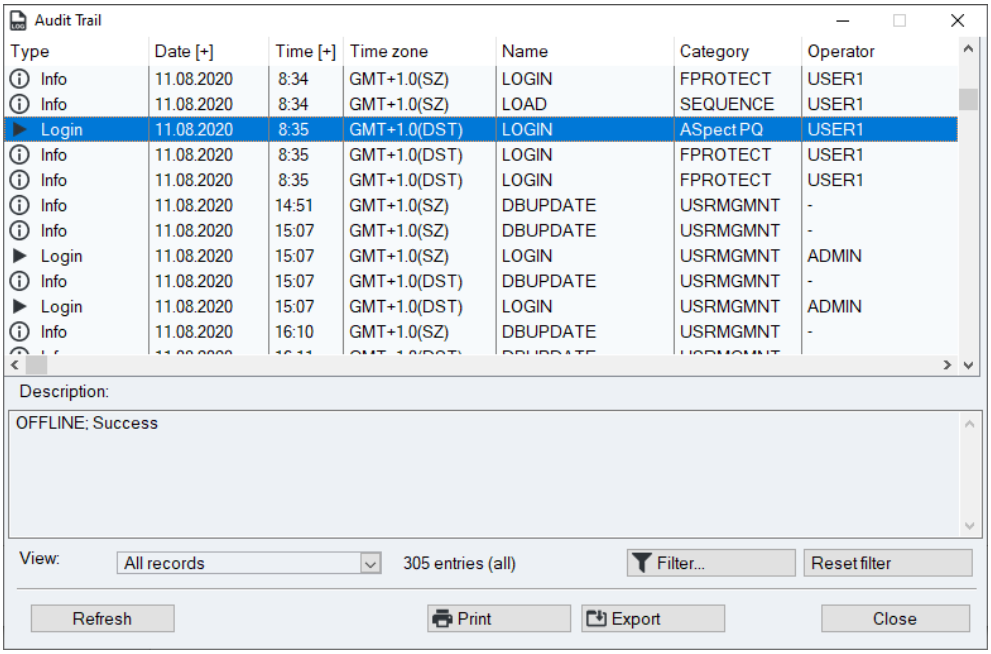
▶ Click **[Audit Trail...]** in the User Management window

or:

Select the **System | Audit Trail** menu command in Aspect PQ.

The audit trail can now be:

- viewed
- updated
- exported as a CSV-file (only if audit trail was opened from the **User management** window)



Window Audit Trail

The following parameters are documented in an audit trail file:

Column	Description
Type	Indicates the type of an event An audit trail keeps track of the following types of events and marks these with symbols: **Info**, **Warning**, **Error**, **Login** and **Logout**
Date/Time	Designates the date and exact time of an entry (PC watch) The **[+]** and **[-]** buttons in the table header of both columns can be used to sort the entries by ascending or descending date.
Time Zone	Indicates the time zone to which the time of an entry is referenced (Windows system control)
Name	Describes an event, for details refer to **Description** field
Categoy	Shows the category of an event. "USRMGMNT" is the category used to mark those entries made in User Management. All other categories will be entered by ASpect PQ.
Operator	Designates the user in login state at the moment of an entry.
Description	Provides more detail about the cause of a selected entry.

Updating an audit trail

You may use **[Refresh]** to update the list of entries in an audit trail.

This may be necessary if further entries were added to a previously created audit trail display.

Exporting Audit Trail

- ▶ Use **[Export]** to open the **Save as** standard window.
- ▶ Type in a name, then save your export file with **[OK]**.
  - ✓ The audit trail file will be exported.

Filter audit trail

You can filter the audit trail by specific names, categories or users and limit the time period of the entries.

- To do this, click on **[Filter]** and specify the search filter in the **Filter Audit Trail** window.
- Click **[Delete Filter]** to remove the restrictions imposed by the filter.

Print Audit Trail

You can print the audit trail. If you have filtered the entries, only the filtered entries are printed.

- Start printing the current audit trail view with **[Print]**.
  The ASpect PQ print window opens.
- If necessary, change to the output format in the **Output** list.
- Start the printout with **[Start]**.
  - ✓ The audit trail is output in the selected output format.

12.3 Electronic signatures

ASpect PQ allows users to electronically sign up for their result data provided the given user has authorization to **Use e-signature** (→ "Creating a new user account" pg. 149). A signature will close work on a particular file so changes in this file made at a later point in time will cause an invalid signature state.

A sign-up procedure will encode a given file and assign to this file a signed state and the data of the signing user. In addition, an encoded signature file of identical name (same as results file) will be created, except that this signature file carries a *.sig data extention. It contains the checksums of the related results file, including those of (if included) a spectrum file.

A file may be signed by more than one user.

12.3.1 Signing measured results

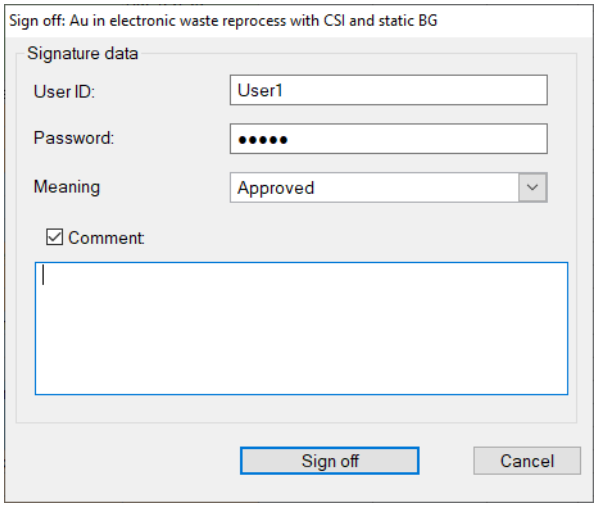
A file of measured results may be sealed with an electronic signature by one or more users authorized to do so on completion of measurement or when this file is loaded at some later point in time.

- Select the **System | Sign off results** menu command in ASpect PQ.

  The **Sign off** dialog screen opens.

Option	Description
User ID	The login name of a current user. A user ID may be modified to facilitate signing by more users (max. 32 characters).
Password	Code word of a user (max. 20 characters).
Meaning	The measning of a signature, for example, Created, Reviewed, Approved. To define a list of signature meanings is the responsibility of the User Management administrator.

Comment	For optional comment (max. 256 characters)
[Sign off]	Signs a given document with any settings it contains. After actuation of **[Sign off]**, you will be asked if this signature is granted or if the process is to be aborted. Successful granting of a signature will be confirmed.



Dialog screen for measured data sign off

12.3.2 Displaying signatures

A print preview of signed result data includes a **Signatures** section that is attached to the end of the related log sheet. This section contains all electronic signatures belonging to the file.

Option	Description	
Issued by	States the full username and ID user of the individual who signed this file.	
Signed on:	Date/time of signature granting	
Status	The signature state may take on one of the following meanings:	
	Valid	Signature and result data are complete and correct. Calculated checksums of a file reveal no variance against the checksums contained in the signature file at the moment of signing.
	Invalid (signature file missing or invalid)	Failure to find signature file that belongs to data set or signature file contains faults.
	Invalid (TPS data)	Results file was modified following signature. Comparison between newly calculated checksums and previously saved checksums reveals variances.
	Invalid (SPK data)	A file with raw spectrum data was modified after being signed. Comparison between newly calculated checksums and previously saved checksums reveals variances.
Meaning	The meaning of signatures	
Remarks	For comment text (optional)	

13 Supplement

13.1 Overview of markings used in the display of values

Remark	Meaning	Values	Output
> Cal	The mean value is larger than the working range of the calibration curve.	Mean values	Sequence and results window
< Cal	The mean value is smaller than the working range of the calibration curve.	Mean values	Sequence and results window
< LOD	The value is smaller than the limit of detection.	Mean values	Sequence and results window
< LOQ	The value is smaller than the limit of quantitation and larger than the limit of detection.	Mean values	Sequence and results window
RSD!	The mean value is outside the range of the specified relative standard deviation.	Mean values	Sequence and results window
RR!	The mean value is outside the range of the specified relative range.	Mean values	Sequence and results window
Factor!	Limit of recalibration factor for the calibration curve was exceeded	Calibration curve	Sequence and results window
R2 (adj.)	The quality of regression R2 (adj.) of the calibration curve falls below the specified value.	Calibration curve	Sequence and results window Calibration curve window
#MAN.	The value was manually excluded from the calculation of the mean value.	Single values	Sample individual values window
#COR.	The value was automatically excluded from the calculation of mean values by the Grubbs outlier test	Single values	Sample individual values window