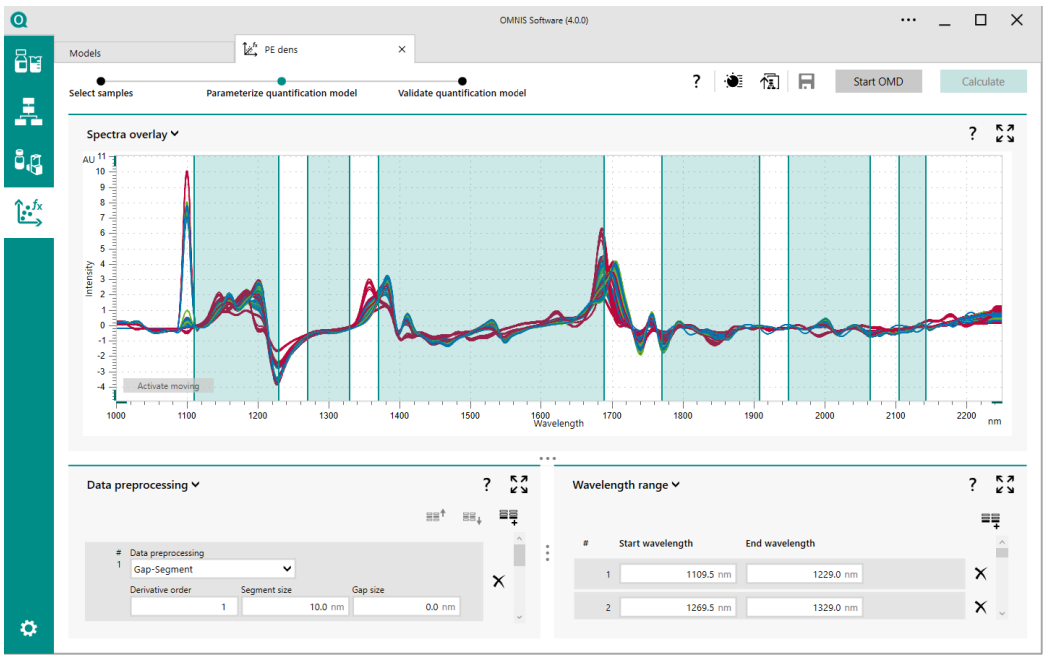
OMNIS NIR (Lab)



OMNIS Spectroscopy step by step

Tutorial

8.0600.8202EN / v6 / 2025-10-10

Metrohm

Metrohm AG
Ionenstrasse
CH-9100 Herisau
Switzerland
+41 71 353 85 85
info@metrohm.com
www.metrohm.com

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OMNIS Software version 4.6

Tutorial

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Technical Communication
Metrohm AG
CH-9100 Herisau

This documentation is an original document.

This documentation has been prepared with great care. However, errors can never be entirely ruled out. Please send comments regarding possible errors to the address above.

Disclaimer

Deficiencies arising from circumstances that are not the responsibility of Metrohm, such as improper storage or improper use, etc., are expressly excluded from the warranty. Unauthorized modifications to the product (e.g., conversions or attachments) exclude any liability on the part of the manufacturer for resulting damage and its consequences. Instructions and notes in the Metrohm product documentation must be strictly followed. Otherwise, Metrohm's liability is excluded.

Table of contents

1 Overview

1.1 Introduction

This tutorial describes the operation of instruments of the **OMNIS NIR Analyzer** product family, based on the OMNIS Software version 4.6.

The tutorial gives a brief overview of the OMNIS Software and describes the instrument setup, model development and prediction.

1.2 About the documentation

Possible depictions in the documentation:

(**1**)	Reference to position number in the figure
1	Instruction step
Method	Parameters, menu items, tabs, and dialogs
Processes ▶ **Operating procedures**	Menu path
[Continue]	Button or key
	Supplementary information to the descriptive text

1.3 OMNIS licenses

OMNIS is a modular platform. Instrument functions and software modules can be freely combined:

- Instrument functions are available as license packages (see *Metrohm Knowledge Base*).
  To work through this tutorial, the following function license is required:
  – Function license Lab NIR Spectroscopy

- Software modules can be licensed and activated individually (see *Metrohm Knowledge Base*).
  To work through this tutorial, the following software licenses are required (example for OMNIS Stand-Alone):
  - Software license OMNIS Stand-Alone
  - For the development of quantification models: Software license Quant Development
  - For the development of identification models and qualification models: Software license Ident Development

Additional information on the licenses can be obtained at the *Metrohm Knowledge Base* or from your regional Metrohm representative.

1.4 User rights

If the user management is activated, then an administrator can set up other users and assign user rights. User roles group together a number of individual rights and thus make it easier to manage rights (see *Metrohm Knowledge Base*).

If user management is activated, then the user role **Method developer** or **Laboratory managers** is required to complete this tutorial.

Alternatively, the OMNIS Software without activated user management can be used.

1.5 Further information

The OMNIS help can be accessed from the OMNIS Software or via a browser.

Calling up help from the OMNIS Software

- **Specify online or offline access**
  - Online access (internet access required): In the title bar under •••, activate the option **Metrohm Knowledge Base**.
  - Offline access: In the title bar under •••, deactivate the **Metrohm Knowledge Base** option.
- **Call up the start page of the help**
  - In the title bar under •••, click on **Help**.
  - Or press the **[F1]** key.
- **Call up context-dependent help**
  - Click on ? in the relevant area or window.
    Note: The start page always appears for offline access.

Calling up help via a browser

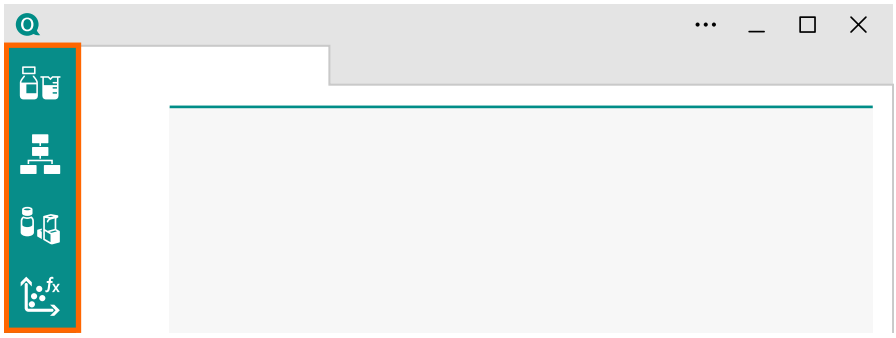
- Go to *https://guide.metrohm.com/*.
- Click on **OMNIS Software**.
- Under the **Release** filter, select the required version of the OMNIS Software.
- For the latest version of the OMNIS Software and for long-term support versions, the help is also available as a PDF file: Use Filter **Information** ▶ **Publication** ▶ **Product manual**.

2 Brief overview of the OMNIS Software

2.1 Structure and functions

2.1.1 Work areas

The OMNIS Software divides the user interface into multiple work areas. Each of the icons on the left side of the screen opens a specific work area.



Work areas

	In the **Samples** work area, samples can be organized and subsamples can be analyzed.
	In the **Processes** work area, operating procedures and methods can be defined to determine how samples will be analyzed.
	Instruments and accessories can be managed in the **Equipment** work area.
	Spectroscopic models can be developed in the **Calibration and evaluation** work area. A model enables the prediction of sample properties.

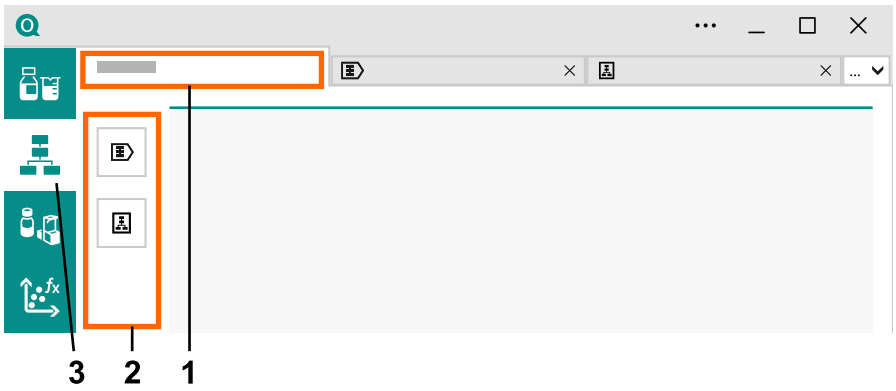
There is also the **Settings** work area.

Depending on the system, the **User management** and **Audit Trail** work areas are also available.

2.1.2 Tabs and subsections

A work area contains one or more **tabs**. Each tab fulfills a particular purpose. For example, in the Processes work area, a tab may enable an operating procedure to be edited.

Each work area has its own tabs. The leftmost tab (**1**) is used to organize the selected work area. Other tabs can be opened from there to execute specific tasks.



Work areas are further divided into **subsections**. The leftmost tab (**1**) shows the subsections (**2**) of the selected work area (**3**).

Relevant subsections

The following *figure 1* shows a schematic depiction. The Samples work area contains a sample list. The sample list contains samples and subsamples. Each subsample has an operating procedure assigned to it.

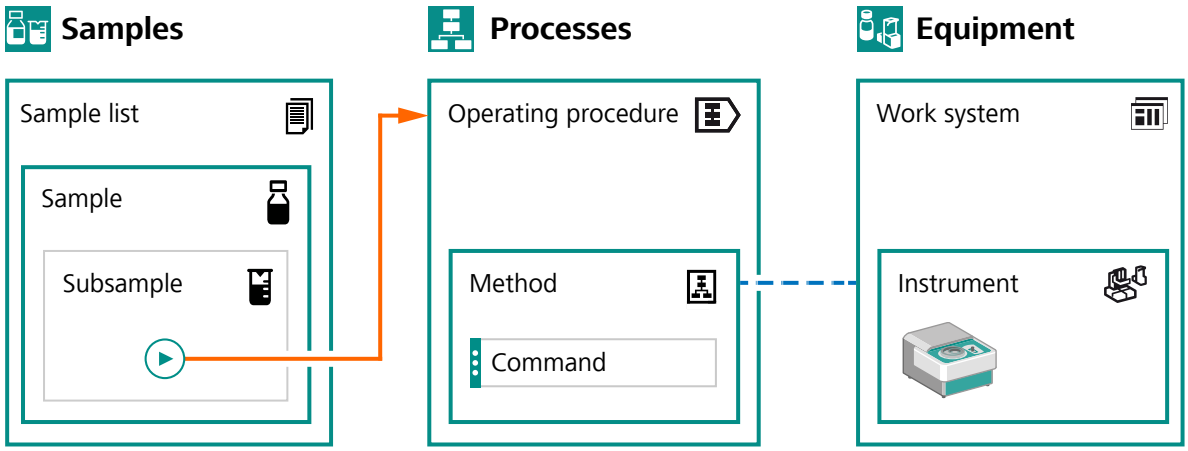


Figure 1 3 work areas with relevant subsections

→	A subsample calls up an operating procedure.
- - - - -	The method has a work system assigned to it.

If a subsample is analyzed, the OMNIS Software starts the assigned operating procedure and executes the methods and commands contained therein.

The method has a work system assigned to it. This allows commands to access the work system and the functional units it contains.

2.1.3 Samples work area

A **sample** is the substance to be analyzed. A sample is divided into one or more subsamples.

A **subsample** has an operating procedure assigned to it. The operating procedure assigned to it is executed when the subsample is analyzed.

A **sample list** organizes samples and subsamples. A sample or subsample can be included in one or more sample lists.

A **sample profile** is a template for creating samples.

Samples – Overview

In the **Samples** work area, samples can be organized and subsamples can be analyzed.

The following *Figure 2* shows a simplified example of a sample list containing a sample, which in turn contains a subsample.

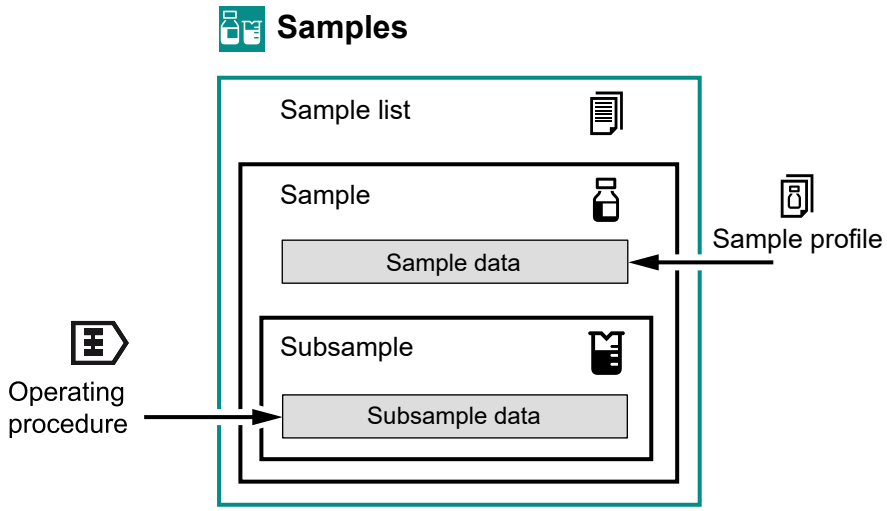


Figure 2 Samples work area

→ Specification of data fields.

Data fields for samples and subsamples can be set up, e.g., sample data for reference values or subsample data for analysis results.

Figure 2 also shows how data fields are created:

- A sample profile can be used to stipulate fields for sample data.
- An operating procedure can be used to stipulate fields for subsample data.

Data fields can be filled with data manually or automatically (e.g., by a command).

Samples – Subsections

The **Sample lists** subsection provides the following functions:

- Create and manage sample lists.
- Within a sample list:
  - Add new samples and subsamples.
  - Process subsamples, whereby for each subsample its assigned operating procedure is executed. The operating procedure can carry out, for instance, a sample analysis or a wavelength calibration.

In the **Search queries** subsection, search queries can filter all samples and subsamples in the database according to various criteria.

The filter criteria can be saved as a search query.

The samples found can be saved as a sample list.

In the **Sample profiles** subsection, a sample profile specifies the following for a series of similar samples:

- The structure of the sample data, i.e., the number and type of sample data fields.
- Default values for sample data fields.
- One or more default operating procedures, each applying respectively to a default number of subsamples.

2.1.4 Processes work area

Processes – Overview

In the **Processes** work area, operating procedures and methods can be defined to determine how samples will be analyzed.

The following *Figure 3* illustrates the modules for processes:

- **Operating procedures**
- **Methods**
- **Commands** (e.g., **MEAS SPEC**)

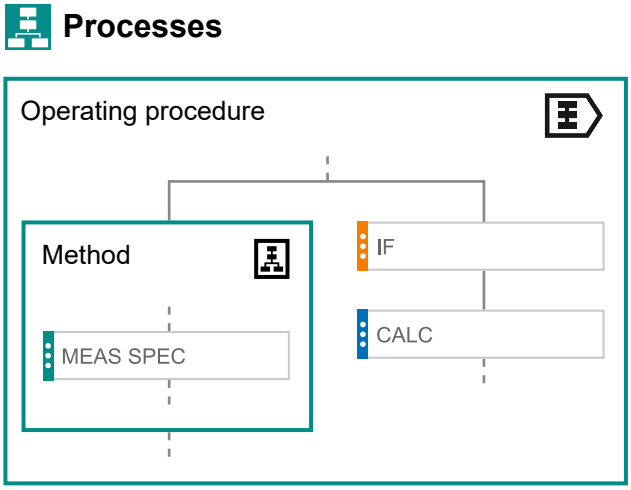


Figure 3 Processes work area

Processes – Subsections

	In the **Operating procedures** subsection, operating procedures can be assembled from methods and commands. The methods and commands can be aligned to be executed sequentially or simultaneously.
	In the **Methods** subsection, methods can be assembled from commands. The commands can be aligned to be executed sequentially or simultaneously. A method can contain commands that control the actions of a work system. These commands are executed on the work system that is assigned to the method.

2.1.5 Equipment work area

Equipment – Overview

	Instruments and accessories can be managed in the **Equipment** work area.

The following *Figure 4* shows how an instrument is made accessible:

1. In the **Instruments** subsection, an inventory lists all available network and USB devices.
2. An instrument can be reserved via the **Inventory**. This makes all the instrument's functional units available to the user.
3. In the **Work systems** subsection, a work system can be assembled with all of the functional units required for the determination.

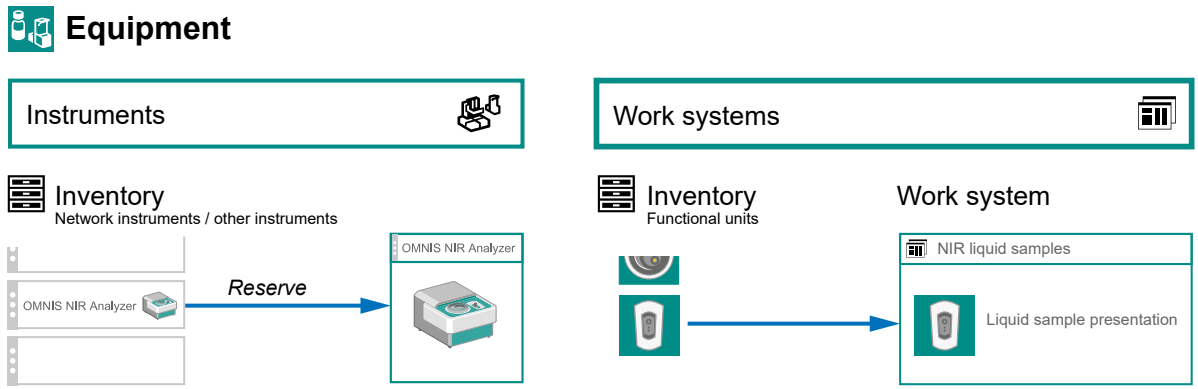


Figure 4 Equipment work area

Equipment – Subsections

Instruments can be reserved and released in the **Instruments** subsection. If an instrument is reserved, then its functional units are available to the user.

After the analysis, the instrument can be released again so that other users can access it.

In the **Work systems** subsection, work systems can be assembled from one or more functional units. The same functional unit may be contained in multiple work systems.

To analyze samples, a method accesses a work system and uses the functional units it contains. A work system can be assigned to multiple methods.

Note: The functional units in the work system can be easily replaced if required. In this way, analyses can be executed on different instruments without having to change the methods.

2.1.6 Calibration and evaluation work area

Spectroscopic models can be developed in the **Calibration and evaluation** work area. A model enables a sample to be analyzed on the basis of the acquired spectrum:

- **Quantification model**: Prediction of a quantitative parameter of interest (e.g., water content 5.1%)
- **Identification model**: Identification or verification of a sample (e.g., fructose)
- **Qualification model**: Qualification of a sample (e.g., the sample corresponds to the specifications)

Calibration and evaluation – Overview

	Models and model hierarchies can be developed in the **Calibration and evaluation** work area.

The work area is divided into subsections.

Calibration and evaluation – Subsections

	Quantification models can be developed in the **Quantification models** subsection. A quantification model describes the dependence of a quantitative parameter of interest on the acquired spectra of the samples.
	Slope/y-intercept corrections can be created in the **Slope/y-intercept corrections** subsection. This enables systematic errors in the application of a particular quantification model to be corrected. Systematic errors can occur, e.g., if there are changes in the samples or in the sample processing.
	Models for the identification and verification of samples can be developed in the **Identification models** subsection. An identification model divides samples into different products (e.g., fructose, glucose, and lactose), based on the acquired spectra.
	Models for the qualification of samples can be developed in the **Qualification models** subsection. A qualification model subdivides samples into positive (usable) and negative (unusable) samples based on the acquired spectra.
	Model hierarchies can be developed in the **Model hierarchies** subsection: ▪ If the samples cannot be distinguished by a single identification model, then a model hierarchy can link multiple identification models hierarchically. ▪ If quantitative analyses are to be carried out for identified samples, then a model hierarchy can link quantification models with products. ▪ If more precise results are required for a quantification model, then subordinate quantification models can be created and linked. Each of these subordinate models is optimized for a part of the reference value range of the superordinate model.

2.2 Practical introduction

The following introduction provides a first look into the OMNIS Software.

Work areas

The OMNIS Software divides the user interface into multiple work areas. Work areas can have different subsections.

In this tutorial, subsections are indicated by a menu path. Example: The **Methods** subsection in the **Processes** work area is indicated by **Processes** ▶ **Methods**.

The following procedure shows how to open such a work area subsection.

1 Opening the work area

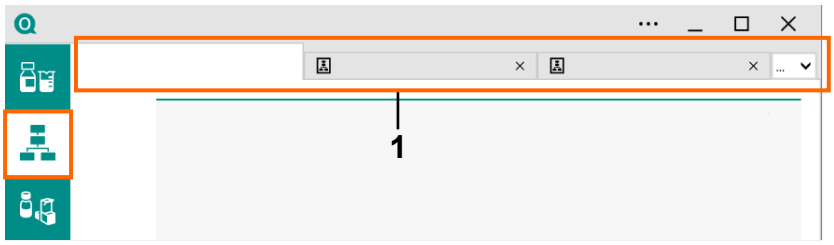
Click on the icons on the left side of the screen to switch between different work areas.

For the above example, open the **Processes** work area by clicking on the corresponding icon  .

Tooltips

When the cursor is placed over an icon, a tooltip appears with the name of the work area, a brief explanation, and a link to further information. Tooltips for other elements of the user interface can be displayed in the same way.

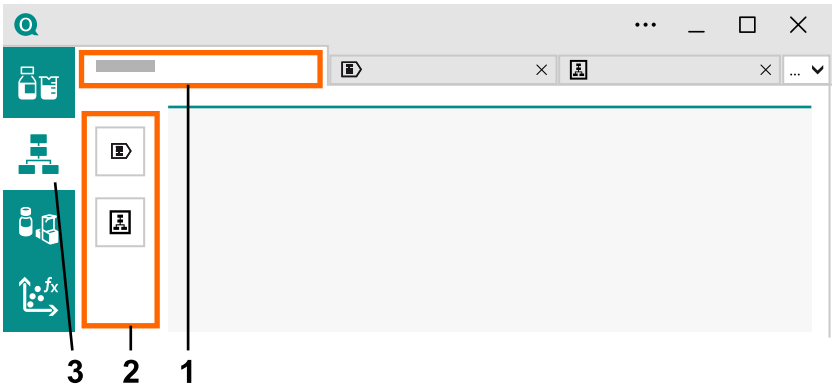
To show tooltips on a touch screen, touch and hold the element.



The work area can contain one or more tabs (**1**).

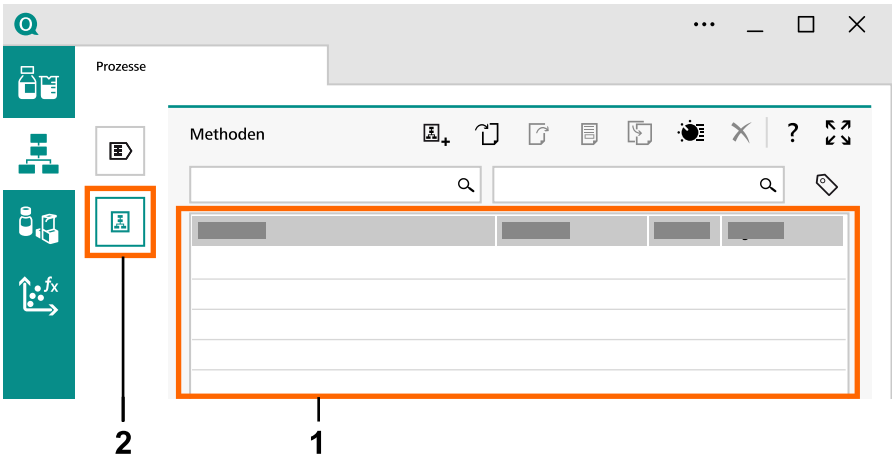
2 Opening the Subsection tab

Click on the leftmost tab (**1**) to show the subsections (**2**) of the selected work area (**3**).



3 Opening a subsection

Open the **Methods** subsection by clicking  (**2**).



The **Methods** subsection contains an overview list showing all of the methods in the database (**1**).

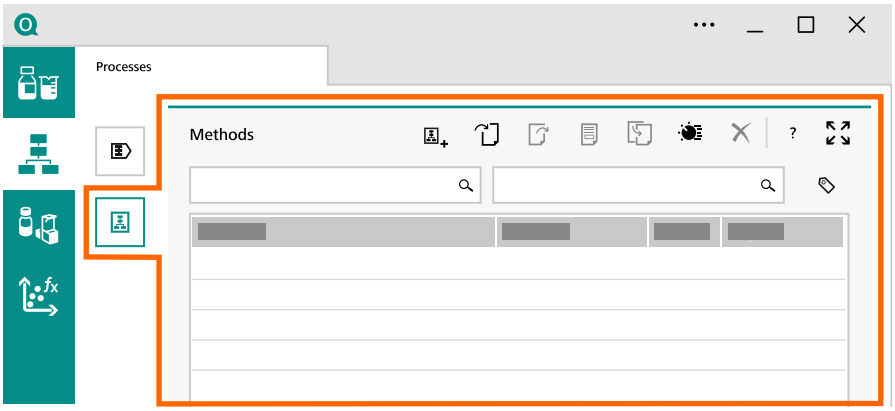
Tabs

As seen above in step 3, a work area subsection can contain an overview list. If an entry in the list is opened or if a new entry is created, then it will be presented in a separate tab.

As an example, the following procedure describes how to create a new method.

1 Opening the Methods subsection

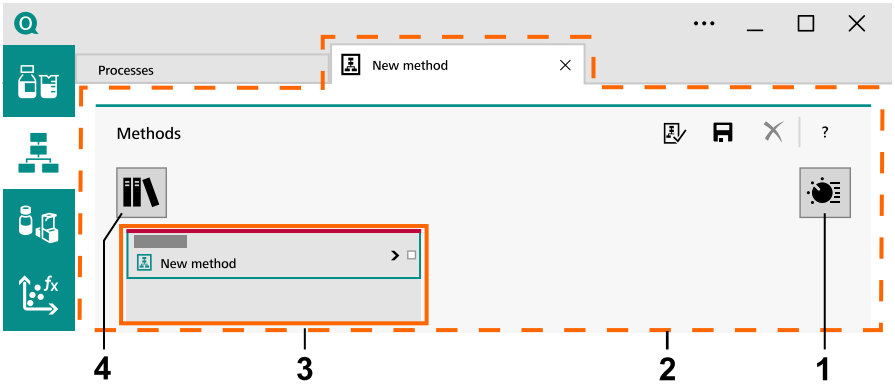
Open **Processes** ▶ **Methods** as described above.



2 Creating a method

- Click on  .

A new tab named *New method* (**2**) appears in which the method can be created (**3**).



The icons (**1**) and (**4**) provide access to other, currently hidden windows, as explained below.

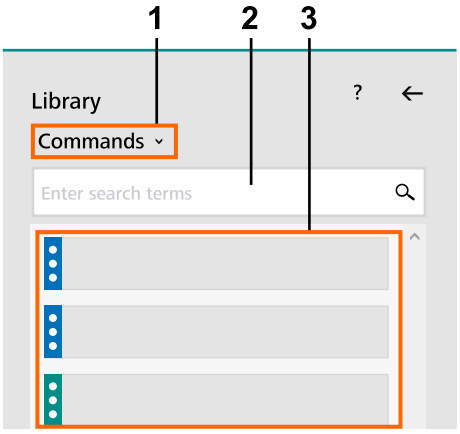
Windows

Windows are part of a tab or of an area inside the tab. Some windows are visible, others are hidden and must be opened with an icon.

1 Library window

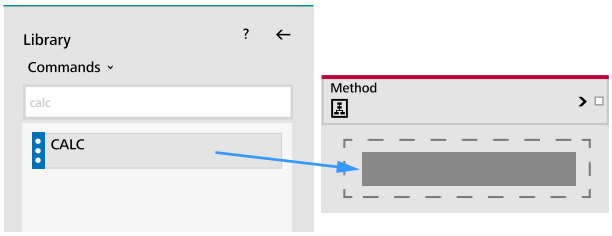
A library contains elements that can be inserted into a process.

- Open the **Library** window by clicking on .
  The library window opens:

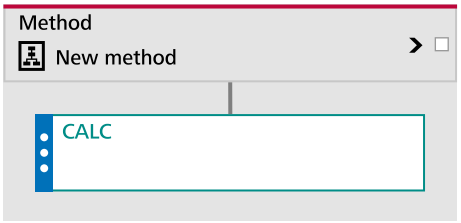


The library window has different **subsections** that can be selected from the selection list (**1**), for instance **Library ► Commands**.
The search field (**2**) allows searching for subsection items, e.g. for commands (**3**).

- As an example, insert the **CALC** command into the method:
  - Under **Library** ▶ **Commands**, search for the **CALC** command.
  - Use drag and drop to insert the **CALC** command into the method.



Now the method contains a command:

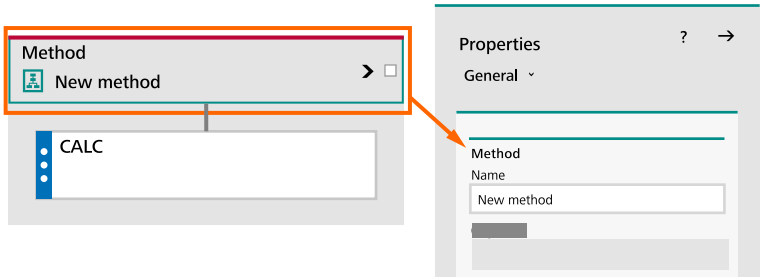


If necessary, additional commands may be added. Commands that are aligned one below the other are executed sequentially. Commands that are aligned side by side are executed in parallel.

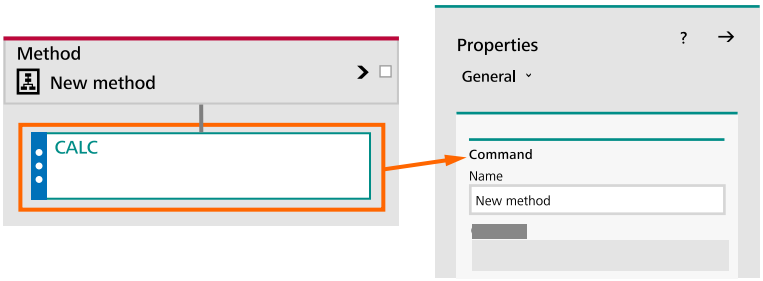
- To close the library window, click on ←.

2 Properties window

- Open the **Properties** window by clicking on .
  The content of the properties window depends on which element is selected:
  - Access the method properties by clicking on
    

    :
    

  - Access the command properties by clicking on
    

    :
    

  - A double-click on one of the elements opens the **Properties** ▶ **Parameters** subsection.
- To close the properties window, click on .

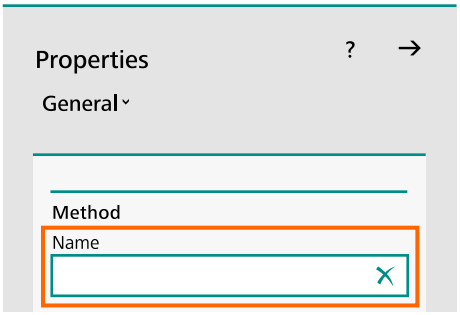
Finishing the introduction

1 Naming the method

- Click on 

  .
- Open **Properties** ▶ **General**.

- Enter a name for the method:



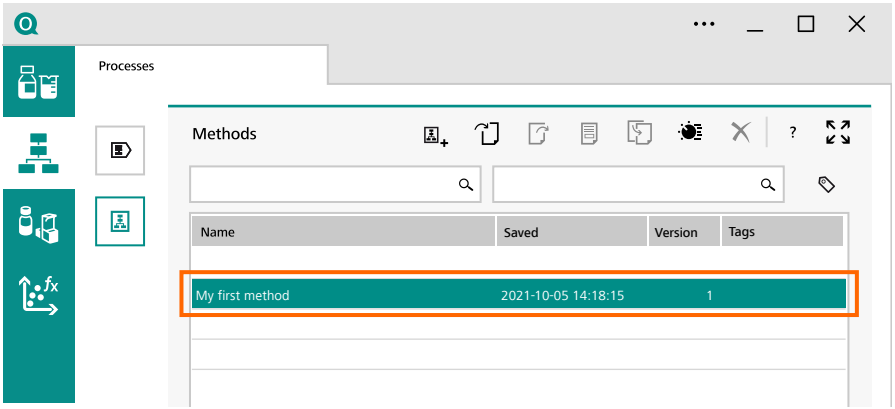
2 Saving a method

- Save the method by clicking on or pressing the **[CTRL]+[S]** keys.

3 Opening the overview list

- Close the tab and switch back to the subsection tab (the leftmost tab) **Processes** ▶ **Methods**.

The created method is now shown in the overview list:



4 Deleting a method

- Select the created method.
- Delete the selected method by clicking on .
  Note: As long as a method is opened in a tab, it cannot be deleted.
- A confirmation prompt appears.
  Verify the name of the method to be deleted.
- Confirm with **Delete**.

The method is deleted from the database and from the overview list.

2.3 OMNIS commands

Commands perform specific tasks. The **MEAS SPEC** command is used, for example, to acquire a spectrum. The **MEAS SPEC** command is used in a method and can access the work system assigned to the method.

Some commands can also be inserted in operating procedures, e.g., the **IF** command.

Commands are displayed in two-line format. The first line contains the name of the command type (e.g., **MEAS SPEC**) and the second line contains a user-specific command name.

It is good practice to change the default command name (e.g., Acquire spectrum 1), to a more specific name. The cross-references to the command are automatically adjusted.

The left edge of the command element has a different colour, depending on the type of command:

- Measuring commands, calibration commands and titration commands

- Structure commands that control the method run (e.g. branches and loops)

- Dosing commands, automation commands and other commands

Command variables

Each command has at least one command variable that is created in the process sequence and can be used under the '**Variable name**.*Command name*' designation.

The following variable is available for all commands:

'**Finished**.*Command name*'

Status of the command.

- **Invalid**: The command has not been started (yet).
- **0**: The command is still running.
- **1**: The command has been completed correctly.
- **2**: The command has not been completed correctly. An error or a warning occurred.
- **3**: The command was skipped either by a **SKIP** command or manually in the **Live data**.
- **4**: The command was stopped either with a manual action by the user (stop or emergency stop), by a **STOP** command or because of an error in a command that is running in parallel.

2.3.1 Spectra acquisition

Command name	Description	Resulting command variables (followed by *.Command name*)
PREP SPEC For functional units of the **Liquid Sample Presentation** type	Prepares the analysis of liquid samples: ▪ Ensures that the inserted sample holder fits the stipulated sample vessel. Otherwise the determination is canceled. ▪ Ensures that a sample vessel is inserted. Otherwise, a prompt to insert the sample appears. ▪ Enables temperature control on the sample vessel or on the sample holder *(see "Temperature control (Liquid Sample Presentation)", chapter 2.5, page 29)*.	

Command name	Description	Resulting command variables (followed by *.Command name*)
MEAS REF SPEC	Records a reference spectrum on the assigned functional unit. The reference spectrum is stored on the instrument. There is 1 reference spectrum per functional unit. Each execution of a **MEAS REF SPEC** command overwrites the previous reference spectrum. The stored reference spectrum is used by all **MEAS SPEC** commands that are executed on the respective functional unit.	▪ **Date.Result** Time at which the reference spectrum was acquired. Additionally for functional units of the **Liquid Sample Presentation** type: ▪ **TemperatureControlMode.Result** Place of temperature control – **Inactive**: No temperature control. – **Sample holder**: The temperature was controlled on the sample holder. – **Sample vessel**: The temperature was controlled on the sample vessel. ▪ **CurrentTemperature.Result** Current temperature while the command is being executed. Unit: °C
MEAS SPEC	Records a spectrum of a sample on the assigned functional unit. Calculation of the **absorption spectrum** of the sample is based on the spectrum and the reference spectrum stored on the instrument for the corresponding functional unit.	For functional units of the **Liquid Sample Presentation** type: ▪ **TemperatureControlMode.Result** Place of temperature control – **Inactive**: No temperature control. – **Sample holder**: The temperature was controlled on the sample holder. – **Sample vessel**: The temperature was controlled on the sample vessel. ▪ **CurrentTemperature.Result** Current temperature while the command is being executed. Unit: °C

Command name	Description	Resulting command variables (followed by *.Command name*)
VESSEL REMOVAL For functional units of the **Liquid Sample Presentation** type	▪ Can ensure the removal of the sample vessel. The process sequence is interrupted until the sample vessel has been removed. This enables a controlled run with series determinations. ▪ If the temperature is controlled at the sample vessel, then the temperature sensor is moved away from the sample vessel. The sample vessel can be removed without damaging the temperature sensor as soon as the prompt to remove the sample vessel appears. ▪ Temperature control can be deactivated or continued on the sample holder.	

2.3.2 Prediction

PREDICT – Quantification

The **PREDICT** command applies a model to an absorption spectrum that was acquired with the **MEAS SPEC** command.

The quantification model provides a prediction of a quantitative parameter of interest. A slope/y-intercept correction can be applied if required.

Resulting command variables (followed by ***.Command name***)

- **Predicted.Quantification.Result**
  Predicted result for the parameter of interest.
- **Uncorrected.Quantification.Result**
  Predicted value for the parameter of interest without the use of a slope/y-intercept correction.
- **Unit.Quantification.Result**
  Unit of the parameter of interest.
- **IsOutlier.OutlierDetection.Result**
  Assessment indicating whether the spectrum is considered an outlier.
  0: The spectrum is **not** considered an outlier.
  1: The spectrum is considered an outlier (Hotellings T^2 or Q residuals).
- **HotellingsT2.OutlierDetection.Result**
  Hotellings T^2 of the spectrum.

- **LimitHotellingsT2.OutlierDetection.Result**
  Hotellings T^2 limit value for the identification as outlier. The limit value depends on the significance level defined in the model.
- **QResiduals.OutlierDetection.Result**
  Q residuals of the spectrum.
- **LimitQResiduals.OutlierDetection.Result**
  Q residuals limit value for the identification as outlier. The limit value depends on the significance level defined in the model.
- **NearestNeighborDistance.OutlierDetection.Result**
  Nearest Neighbor Distance (NND) of the spectrum *(see "Publish quantification model", page 92)*.
  If the model was published without NND: **Invalid**
- **LimitNearestNeighborDistance.OutlierDetection.Result**
  NND limit value.
  If the model was published without NND: **Invalid**

PREDICT – Identification and verification

The **PREDICT** command applies a model to an absorption spectrum that was acquired with the **MEAS SPEC** command.

Depending on its use, an identification model provides an identification of an unknown sample (e.g., fructose) or a verification of the product membership of a sample.

Resulting command variables (followed by ***.Command name***)

- **Product.Identification.Result**
  Determined product or determined product group of the identified sample.
  If the identification failed, then no result will be displayed.
- **Status.Identification.Result**
  - **Identified**: Identification successful. A product or product group was determined.
  - **Ambiguous**: Identification failed. The probability levels of multiple products exceeds the probability threshold.
  - **Unidentified**: Identification failed. No probability level of a product exceeds the probability threshold.
- **Probability.Identification.Result**
  - **0.01–100**: The probability in percent expresses the plausibility with which the sample belongs to the product or product group.
  - **Invalid**: Identification failed.

If the model was used for the verification:

- **Status.Verification.Result**
  - **1**: Verification successful.
  - **0**: Verification failed.

PREDICT – Qualification

The **PREDICT** command applies a model to an absorption spectrum that was acquired with the **MEAS SPEC** command.

A qualification model qualifies a sample as a positive (usable) sample.

Resulting command variables (followed by ***.Command name***)

- **Status.Qualification.Result**
  - **1**: Qualification successful.
  - **0**: Qualification failed.

PREDICT – Model hierarchy

The **PREDICT** command applies a model hierarchy to an absorption spectrum that was acquired with the **MEAS SPEC** command.

Depending on its use, the model hierarchy provides an identification of an unknown sample (e.g., fructose), a verification of the product membership of a sample or a quantification of parameters of interest of a sample.

Resulting command variables (followed by ***.Command name***)

- **Model hierarchy (identification)**
  - **Product.Identification.Result**
    Determined product or determined product group of the identified sample.
    If the identification failed, then no result will be displayed.
  - **Status.Identification.Result**
    Identified: Identification successful. A product or product group was identified.
    Ambiguous: Identification failed. The probability levels of multiple products exceeds the probability threshold.
    Unidentified: Identification failed. No probability level of a product exceeds the probability threshold.
  - **Probability.Identification.Result**
    0.01–100: The probability in percent expresses the plausibility with which the sample belongs to the product or product group.
    Invalid: Identification failed.
- **Model hierarchy (verification)**
  - **Status.Verification.Result**
    0: Verification failed.
    1: Verification successful.

- **Model hierarchy (quantification)**
  Note: **x = Index of the quantification model** *(see "Model hierarchy – Index for quantification models", chapter 11.4.1, page 176)*
  If no reference to a quantification model can be established, then the following variables will return the value *Invalid*.
  - **Predicted.Quantification{x}.Result**
    Calculated end value for the parameter of interest.
  - **Uncorrected.Quantification{x}.Result**
    Predicted value for the parameter of interest without the use of a slope/y-intercept correction.
  - **Unit.Quantification{x}.Result**
    Unit of the parameter of interest.
  - **ParameterName.Quantification{x}.Result**
    Name of the reference parameter.
  - **IsOutlier.OutlierDetection{x}.Result**
    Assessment indicating whether the spectrum is considered an outlier.
    0: The spectrum is **not** considered an outlier.
    1: The spectrum is considered an outlier (Hotellings T^2 or Q residuals).
  - **AnyOutlier.OutlierDetection.Result**
    0: None of the quantification models classifies the spectrum as an outlier.
    1: At least one quantification model classifies the spectrum as an outlier. (Hotellings T^2 or Q residuals).
  - **HotellingsT2.OutlierDetection{x}.Result**
    Hotellings T^2 of the spectrum.
  - **LimitHotellingsT2.OutlierDetection{x}.Result**
    Hotellings T^2 limit value for the identification as outlier. The limit value depends on the significance level defined in the model.
  - **QResiduals.OutlierDetection{x}.Result**
    Q residuals of the spectrum.
  - **LimitQResiduals.OutlierDetection{x}.Result**
    Q residuals limit value for the identification as outlier. The limit value depends on the significance level defined in the model.
  - **NearestNeighborDistance.OutlierDetection{x}.Result**
    Nearest Neighbor Distance (NND) of the spectrum *(see "Publish quantification model", page 92)*.
    If the model was published without NND: **Invalid**
  - **LimitNearestNeighborDistance.OutlierDetection{x}.Result**
    NND limit value.
    If the model was published without NND: **Invalid**

2.3.3 Calculations and statistics

Command name	Description	Resulting command variables (followed by *.Command name*)
CALC	Carries out calculations, for instance to further process the predicted result. The formula can be created using a formula editor.	▪ '***Result name***' Result value of the calculation. Note: The '*Result name*' can be defined or calculated in the command parameters. The default name is '*Result 1*'. ▪ '**MeanValue**.*Result name*' Mean value of all results that have already been determined before with the same operating procedure version and with the same method versions. ▪ '**StandardDeviation**.*Result name*' Absolute standard deviation. The values of the current subsample and of all subsamples that have already been determined before with the same operating procedure version and with the same method versions are used for the calculation.
EVAL BASE STATISTICS	Determines basic statistical values of a spectrum. The data preprocessing and wavelength ranges to be used can be defined.	▪ **Mean.Result** Mean of the absorbance values. ▪ **StandardDeviation.Result** Standard deviation of the absorbance values. ▪ **Minimum.Result** Minimum of the absorbance values. ▪ **Maximum.Result** Maximum of the absorbance values. ▪ **First.Result** First absorbance value ▪ **Last.Result** Last absorbance value ▪ **Integral.Result** Integral value of the spectrum.
In addition, structure commands are available, such as **IF**, **LOOP**, **SKIP**, **STOP**, **SYNC** or **WAIT**. The **EXPORT** or the **REPORT** command may be used to create an output of the determination data.		

2.3.4 Wavelength calibration

Command name	Description	Resulting command variables (followed by *.Command name*)
CAL WL	Carries out a wavelength calibration of the instrument. The wavelength calibration standardizes the wavelength values, i.e. the x-axis of the spectra.	▪ **Date.Result** Time at which the wavelength calibration was carried out.
VAL WL	Validates the wavelength calibration.	▪ **Date.Result** Time at which the wavelength calibration was validated. ▪ **OverallStatus.Result** **1**: The validation was successful. **2**: The validation failed. ▪ **ExpectedWavelength.Peak{X}** Expected wavelength (in nm) of peak X. ▪ **MeasuredWavelength.Peak{X}** Measured wavelength (in nm) of peak X. ▪ **ExpectedBandwidth.Peak{X}** Expected bandwidth (in nm) of peak X. ▪ **MeasuredBandwidth.Peak{X}** Measured bandwidth (in nm) of peak X. ▪ **Index.Peak{X}** Peak number of peak X. Example: 'Index.Peak{2}' provides the result 2. If no peak exists for X, then the above command variables provide the result: *Invalid*

2.3.5 Instrument performance tests

Command name	Description	Resulting command variables (followed by *.Command name*)
TEST WL	The wavelength test checks the wavelength accuracy and wavelength precision. **Internal** (obligatory): The internal wavelength standard is used. **External** (optional): An external wavelength standard is used.	▪ **Date.Result** Time at which the wavelength test was carried out. ▪ **OverallStatus.Result** **1**: The test was successful. **2**: The test failed.
TEST NOISE	The noise test checks the signal noise. **Internal** (obligatory): The reference path of the sample presentation used is used. **Low flux test** and **high flux test** (optional): External reference standards are used.	▪ **Date.Result** Time at which the signal noise was tested. ▪ **OverallStatus.Result** **1**: The test was successful. **2**: The test failed.
TEST PHOTOMETRIC LINEARITY	The external, optional test checks the photometric linearity with the help of external reference standards.	▪ **Date.Result** Time at which the photometric linearity was tested. ▪ **OverallStatus.Result** **1**: The test was successful. **2**: The test failed.

2.4 Reserving and releasing the instruments

A particular instrument can be used by only one OMNIS system at a time.

The instrument must be reserved before it can be used. As long as the instrument is reserved, no other OMNIS system can access it.

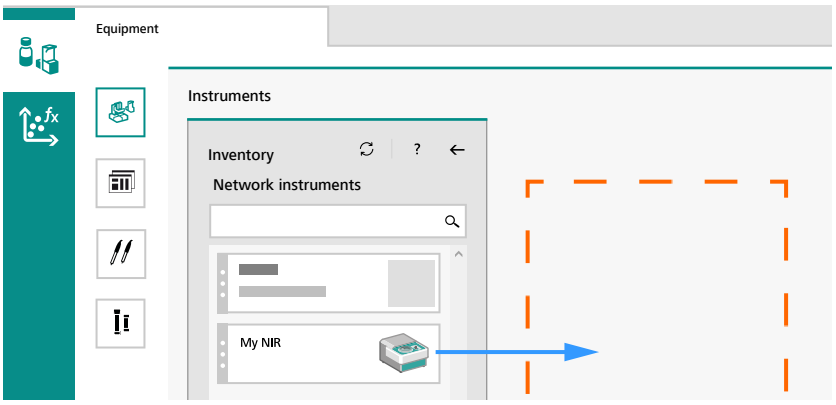
Reserving the instrument

1 Searching for available instruments

- Open **Equipment** ▶ **Instruments**.
- Open the **Inventory** window by clicking on  .
- Find the required instrument.
  Note: Instruments with grayed-out icons are not available.

2 Reserving the instrument

Drag and drop the instrument onto the adjacent work surface.



The instrument is now reserved:

A green status lamp next to the instrument indicates that the instrument can be used.

If needed, additional instruments can be reserved.

The instrument remains reserved when the OMNIS Software is exited.

The instrument is released as soon as Windows is exited. The instrument will be reserved once more as soon as the computer is switched on again. It makes no difference whether the same user logs in or another one.

Releasing the instrument

To release a reserved instrument permanently, proceed as follows:

1 Opening the Instruments subsection

- Open **Equipment** ▶ **Instruments**.

2 Releasing the instrument

- Select the instrument to be released.

- Remove the selected instrument by clicking on ✕.

The instrument is released and is once again available for other users.

2.5 Temperature control (Liquid Sample Presentation)

The temperature control regulates the temperature, either in the sample holder or in the sample itself.

Temperature control in the sample holder

- Supports sample holders for disposable vials, cuvettes, and flow-through cells.
- Target temperature in the sample holder: Between 25 °C and 80 °C (not lower than 5.0 K below the ambient temperature).
- Accuracy of the temperature sensors: < 0.5 K

Temperature control in the sample

- Support for disposable vials.
- Target temperature of the sample: Between 25 °C and 80 °C (not lower than 5.0 K below the ambient temperature).
- Accuracy of the temperature sensors: < 0.5 K
- Control algorithm:
  - The control algorithm takes into account the defined target temperature of the sample and the measured temperature at the sensors. The spectroscopic measurement can start once the modeled temperature in the sample is achieved with sufficient stability and does not deviate more than 0.5 K from the target temperature. It is possible that the spectroscopic measurement will begin shortly after the disposable vial has been inserted.
  - Typical accuracy: 1.0 K (checked in water samples for sample temperatures of 25 °C to 80 °C at an ambient temperature of 23 °C).

Switching on the temperature control

- By a **PREP SPEC** command (command parameter **Temperature control**).
- In manual control (only for temperature control on the sample holder):
  - Under **Equipment** ▶ **Instruments**, double-click on the reserved device to open the **Manual control**.
  - In the **Regulate temperature** area in the **Target temperature sample holder** input field, enter the desired temperature and click on ▶.

Switching off the temperature control

- Via a **VESSEL REMOVAL** command (option **Deactivate**).

- In manual control:
  - Under **Equipment** ▶ **Instruments**, double-click on the reserved device to open the **Manual control**.
  - Click on  in the **Regulate temperature** area. Temperature control is terminated, regardless of whether the temperature is measured in the sample holder or in the sample.
- Temperature control is generally terminated after 2 hours of inactivity or by switching the instrument off.

3 Preparing the instrument

Before the instrument can acquire spectra, the following preparations are required:

- A **work system** must be set up.

After that, the following tasks ensure that the spectra are comparable with one another:

- The **wavelength calibration** calibrates the x-axis of the spectra.
- The **instrument performance tests** ensure that the instrument performance corresponds to the requirements.
  The instrument performance tests must be performed regularly *(see "Instrument performance tests", chapter 10.1, page 165)*.

In addition, the absorbance values on the y-axis of the spectra must be standardized. For this purpose, a **MEAS REF SPEC** command is used before the spectra are acquired.

An illustration of the runs in the OMNIS Software can be found in the Appendix *(see "Preparing the instrument", page 182)*.

3.1 Creating a work system

Prerequisite:

- The spectrometer is reserved *(see "Reserving and releasing the instruments", chapter 2.4, page 27)*.

1 Creating a work system

- Under **Equipment** ▶ **Work systems**, click on .

A new tab opens.

2 Naming the work system

- Select the **New work system** subsection.
  The frame of the subsection turns green.
- Open the **Properties** window by clicking on .
- Enter a matching name in the **Name** field under **Properties** ▶ **General**.

3 Adding the functional unit

- Open the **Inventory** window by clicking on  .
- Use drag and drop to insert the **Liquid Sample Presentation** or **Solid Sample Presentation** functional unit into the work system.

2 functional units are available with instruments of the **OMNIS NIR Analyzer Liquid/Solid** type. Metrohm recommends the creation of separate work systems, each with one of the two functional units.

A functional unit can be removed from the work system once again:

- Select the functional unit that is to be deleted.
- Click on  or press the **[Delete]** key.

If needed, the name of the functional unit can be changed:

- Select the functional unit whose name is to be changed.
- Enter a matching name under **Properties** ▶ **General** ▶ **Name**.

4 Saving the work system

- Click on  or press the **[CTRL]**+**[S]** keys.

One functional unit can be assigned to multiple work systems.

Once the work system is created, the instrument can be released and reserved again as needed.

3.2 Wavelength calibration

The wavelength calibration ensures that the wavelength values of the spectra are comparable with one another. It uses an internal, metrologically traceable wavelength standard.

The wavelength calibration is done in 2 steps:

1. The **CAL WL** command standardizes the wavelength values, i.e. the x-axis of the spectra.
2. The **VAL WL** command validates the wavelength calibration. Validation must be successfully performed before the functional unit can be used to acquire spectra.

2 functional units are available with instruments of the **OMNIS NIR Analyzer Liquid/Solid** type. Wavelength calibration and validation must be carried out separately for both functional units.

3.2.1 Preparing the wavelength calibration

For first-time use of the OMNIS Software, read the introduction before proceeding *(see "Practical introduction", chapter 2.2, page 11)*.

Follow the instructions below to create a method containing the **CAL WL** and **VAL WL** commands. Afterwards, create an operating procedure, a sample profile and a sample list. This makes it possible to start the wavelength calibration in the same way as a sample determination.

Creating a method

1 Creating a method

- Under **Processes** ▶ **Methods**, click on  .

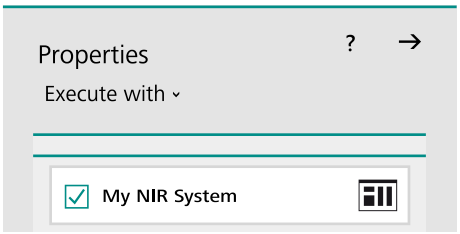
A tab opens with the newly created method and the title **New method**.

2 Naming the method

- Open the **Properties** window by clicking on  .
- Under **Properties** ▶ **General**, enter the following name in the **Name** field: **Wavelength Cal/Val**.

3 Assigning the work system to the method

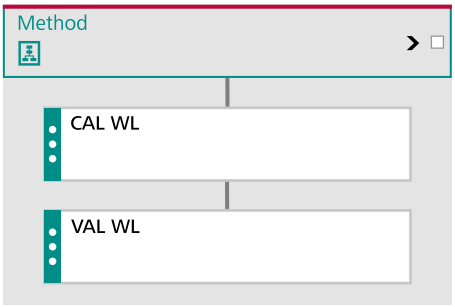
- Under **Properties** ▶ **Execute with**, select the work system to be used.



Use the same work system for all of the methods in this document.

4 Inserting commands

- Open the **Library** window by clicking on .
- Under **Library** ▶ **Commands**, search for the **CAL WL** command.
- Use drag and drop to insert the **CAL WL** command into the method.
- Search for the **VAL WL** command and place it below the **CAL WL** command.



The sequence of commands is important. Commands that are aligned one below the other are executed sequentially. First, the **CAL WL** command is executed, and then the **VAL WL** command.

The commands automatically acquire a reference spectrum. The **MEAS REF SPEC** command is therefore not required.

5 Saving a method

- Click on or press the **[CTRL]**+**[S]** keys.

Creating an operating procedure

1 Creating an operating procedure

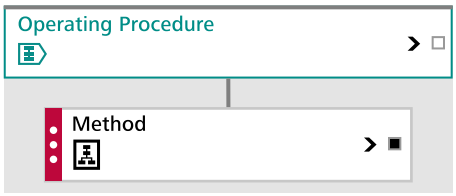
- Open **Processes** ▶ **Operating procedures** by clicking on , then on .
- Create a new operating procedure by clicking on .

2 Naming the operating procedure

- Open the **Properties** window by clicking on .
- Under **Properties** ▶ **General**, enter the following name in the **Name** field: **Wavelength Cal/Val**

3 Inserting the method

- Open the **Library** window by clicking on .
- Use drag and drop to insert the created method from **Library** ▶ **Methods** into the operating procedure.



4 Saving the operating procedure

- Click on or press the **[CTRL]**+**[S]** keys.

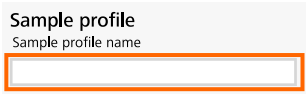
Creating a sample profile

1 Creating a sample profile

- Under **Samples** ▶ **Sample profiles**, click on .

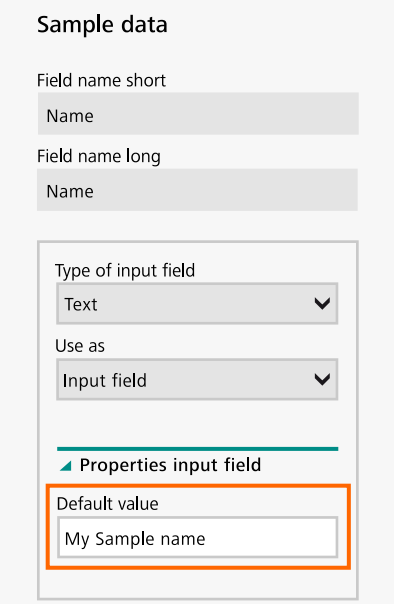
2 Naming the sample profile

- Enter the following name in the **Sample profile name** field: **Wavelength Cal/Val**



3 Input field for the sample name

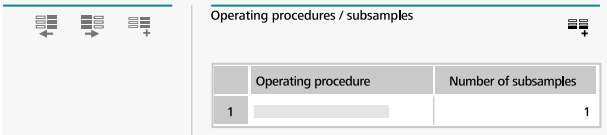
The **Sample data** area contains a field for the name of the sample:



- Enter a **Default value** for the sample name.

4 Defining the operating procedure and the number of subsamples

- In the **Operating procedures / subsamples** area, select the **Wavelength Cal/Val** operating procedure.
- The **Number of subsamples** defines how many subsamples are added automatically for each sample. Enter: **1**.



5 Saving the sample profile

- Click on or press the **[CTRL]+[S]** keys.

Creating a sample list

1 Creating a sample list

- Under **Samples** ▶ **Sample lists**, click on .

2 Naming a sample list

- Enter the following name in the **Name** field: **Wavelength Cal/Val**

Confirm with **[Enter]**.

3 Saving the sample list

- Click on or press the **[CTRL]**+**[S]** keys.

Samples will be added later.

3.2.2 Starting the wavelength calibration

Observe execution intervals *(see "Wavelength calibration", chapter 10.2, page 166)*.

Metrohm recommends waiting 1 hour after switching on the instrument before starting the wavelength calibration.

Starting the wavelength calibration

Prerequisite:

The wavelength calibration is prepared *(see "Preparing the wavelength calibration", chapter 3.2.1, page 33)*.

1 Reserving the instrument

Reserve the spectrometer *(see "Reserving and releasing the instruments", chapter 2.4, page 27)*.

2 Opening the 'Wavelength Cal/Val' sample list

- Open the **Samples** work area.
- If the **Wavelength Cal/Val** sample list has been closed, then open the **Sample lists** subsection in the **Samples** tab and double-click on the **Wavelength Cal/Val** sample list.

3 Selecting the 'Wavelength Cal/Val' sample profile

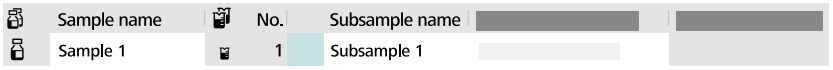
- In the selection list to the left of the + icon, select the **Wavelength Cal/Val** sample profile.

Subsequently added samples are created according to the specifications in the selected sample profile.

4 Adding a sample

- Add a new sample to the sample list by clicking on .

A new entry appears in the sample list. It contains a sample marked with , followed by its subsample marked with .



In accordance with the sample profile, the new sample contains 1 subsample using the **Wavelength Cal/Val** operating procedure.

- Edit the sample names and subsample names as needed.
- Save the sample list by clicking on or by pressing the **[CTRL]**+**[S]** keys.

5 Executing the wavelength calibration

- Select the added sample.
- Start the wavelength calibration by clicking on .
  After completion of the calibration, the status of the subsample is displayed as .

6 Checking the result

- Open **Results** ▶ **Raw data** in the bottom right area.

The results of the calibration and the validation are displayed. Check the overall validation status:

Status warning: If the validation has failed, then the subsample icon will be highlighted in red in the sample list:

Information on the most recently performed wavelength calibration and wavelength validation can be viewed in the instrument properties:

- Select the reserved instrument under **Equipment** ► **Instruments**.
- Click on  to open the **Properties** window.
- **Specific data** ► **Calibration and test data**

The **VAL WL** command variable **OverallStatus.Result** shows the overall validation status:

1: The validation was successful.

2: The validation failed.

3.3 Instrument performance tests

Internal and external instrument performance tests are available:

- **Internal instrument performance tests (obligatory)**
  Internal instrument performance tests must be carried out successfully before the corresponding functional unit can be used to acquire spectra.
  - The wavelength test checks the wavelength accuracy and wavelength precision using the **TEST WL** command. The wavelength test uses an internal, metrologically traceable wavelength standard.
  - The noise test checks the photometric noise, the peak-to-peak noise, and the baseline bias of the noise using the **TEST NOISE** command.
- **External instrument performance tests (optional)**
  The external instrument performance tests support the validation according to pharmacopoeia such as USP <856>, Ph.Eur 2.2.40, and JP 2.27. The following commands are used: **TEST WL** (wavelength accuracy and wavelength precision), **TEST NOISE** (photometric noise, peak-to-peak noise, and baseline bias of the noise for low and for high light intensity), and **TEST PHOTOMETRIC LINEARITY** (photometric linearity).
  The external instrument performance tests require external, metrologically traceable reference standards *(see "External instrument performance tests (optional)", chapter 3.3.3, page 45)*.

3.3.1 Preparing internal instrument performance tests

Follow the instructions below to create a method containing the **TEST WL** and **TEST NOISE** commands. Afterwards, create an operating procedure, a sample profile and a sample list. This makes it possible to start the instrument performance tests in the same way as a sample determination.

Setting up the method

1 Creating a method

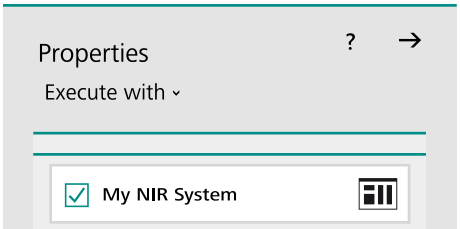
- Under **Processes** ▶ **Methods**, click on .

2 Naming the method

- Open the **Properties** window by clicking on .
- Under **Properties** ▶ **General**, enter the following name in the **Name** field: **Instrument performance test**.

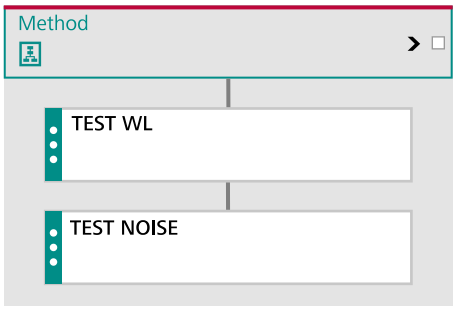
3 Assigning the work system to the method

- Under **Properties** ▶ **Execute with**, select the work system to be used.



4 Inserting commands

- Open the **Library** window by clicking on .
- Under **Library** ▶ **Commands**, search for the **TEST WL** command.
- Use drag and drop to insert the **TEST WL** command into the method.
- Search for the **TEST NOISE** command and place it below the **TEST WL** command.



The commands automatically acquire a reference spectrum. The **MEAS REF SPEC** command is therefore not required.

The instrument performance tests use the respective reference path, depending on the sample presentation. The wavelength test uses an internal, metrologically traceable wavelength standard.

The external instrument performance tests (optional) require external reference standards *(see "External instrument performance tests (optional)", chapter 3.3.3, page 45)*.

5 Saving a method

- Click on or press the **[CTRL]+[S]** keys.

Creating an operating procedure

1 Creating an operating procedure

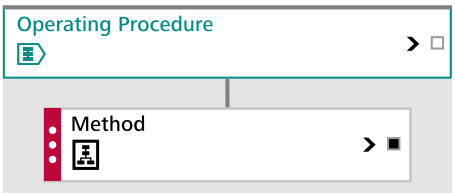
- Under **Processes** ▶ **Operating procedures**, click on .

2 Naming the operating procedure

- Open the **Properties** window by clicking on .
- Under **Properties** ▶ **General**, enter the following name in the **Name** field: **Instrument performance test**

3 Inserting the method

- Open the **Library** window by clicking on .
- Use drag and drop to insert the created method from **Library** ▶ **Methods** into the operating procedure.



4 Saving the operating procedure

- Click on or press the **[CTRL]+[S]** keys.

Creating a sample profile

1 Creating a sample profile

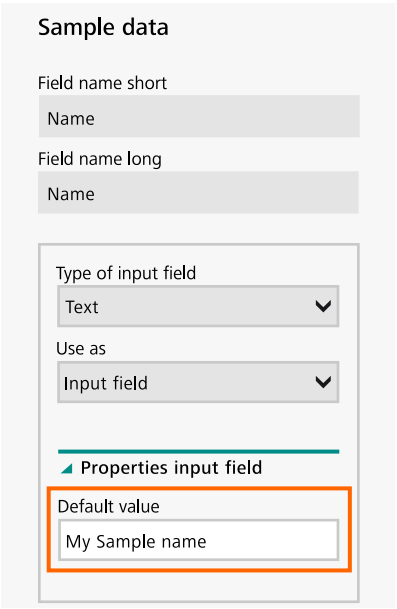
- Under **Samples** ▶ **Sample profiles**, click on .

2 Naming the sample profile

- Enter the following name in the **Sample profile name** field: **Instrument performance test**.

3 Input field for the sample name

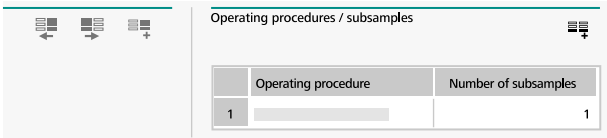
The **Sample data** area contains a field for the name of the sample:



- Enter a **Default value** for the sample name.

4 Defining the operating procedure and the number of subsamples

- In the **Operating procedures / subsamples** area, select the **Instrument performance test** operating procedure that has been created.
- The **Number of subsamples** defines how many subsamples are added automatically for each sample. Enter: **1**.



5 Saving the sample profile

- Click on or press the **[CTRL]**+**[S]** keys.

Creating a sample list

1 Creating a sample list

- Under **Samples** ▶ **Sample lists**, click on .

2 Naming a sample list

- Enter the following name in the **Name** field: **Instrument performance test**.

Confirm with **[Enter]**.

3 Saving the sample list

- Click on or press the **[CTRL]**+**[S]** keys.

Samples will be added later.

3.3.2 Executing internal instrument performance tests

Observe recommended execution intervals *(see "Instrument performance tests", chapter 10.1, page 165)*.

Executing the instrument performance test

Prerequisite:

The instrument performance tests are prepared *(see "Preparing internal instrument performance tests", chapter 3.3.1, page 40)*.

1 Reserving the instrument

Reserve the spectrometer *(see "Reserving and releasing the instruments", chapter 2.4, page 27)*.

2 Opening the 'Instrument performance test' sample list

- Open the **Samples** work area.
- If the **Instrument performance test** sample list has been closed, open the **Sample lists** subsection in the **Samples** tab and double-click on the **Instrument performance test** sample list.

3 Selecting the 'Instrument performance test' sample profile

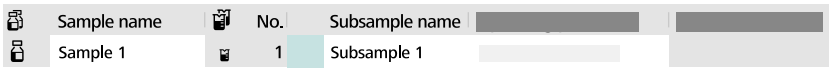
- In the selection list to the left of the + icon, select the **Instrument performance test** sample profile.

Subsequently added samples are created according to the specifications in the selected sample profile.

4 Adding a sample

- Add a new sample to the sample list by clicking on +.

A new entry appears in the sample list. It contains a sample marked with , followed by its subsample marked with .



In accordance with the sample profile, the new sample contains 1 subsample using the **Instrument performance test** operating procedure.

- Edit the sample names and subsample names as needed.
- Save the sample list by clicking on or by pressing the **[CTRL]**+**[S]** keys.

5 Carrying out the instrument performance test

- Select the sample that carries out the instrument performance tests.

- Start the test by clicking on  .

After completion of the instrument performance test, the status of the subsample will be displayed as  .

6 Checking the result

- Open **Results** ▶ **Raw data** in the bottom right area.

The results of the wavelength tests and the noise tests are displayed. Check the overall status for both tests:

Overall status
Successful

Status warning: If an instrument performance test has failed, then the subsample icon will be highlighted in red in the sample list:

Information on the most recently performed instrument performance test can be viewed in the instrument properties:

- Select the reserved instrument under **Equipment** ▶ **Instruments**.
- Click on  to open the **Properties** window.
- **Specific data** ▶ **Calibration and test data**

The **OverallStatus.Result** variable of the **TEST WL** and **TEST NOISE** commands shows the respective overall status of the test:

1: The test was successful.

2: The test failed.

Observe troubleshooting steps for failed tests *(see "Instrument performance tests", chapter 10.1, page 165)*.

3.3.3 External instrument performance tests (optional)

The external instrument performance tests support the validation according to pharmacopoeia such as USP <856>, Ph.Eur 2.2.40, and JP 2.27. External, metrologically traceable reference standards are required. For each external reference standard, the corresponding OMNIS standard file (*.ostd) must be imported into the OMNIS Software (see *Metrohm Knowledge Base*).

So that the internal and external instrument performance tests can be performed independently of one another:

- Create separate methods, operating procedures, and sample profiles.
- For the external instrument performance tests, proceed in the same way as for the internal instrument performance tests, with the following modifications and additions.

The commands automatically acquire a reference spectrum. The **MEAS REF SPEC** command is therefore not required.

TEST WL

- Insert and select the **TEST WL** command in the method.
- Open the **Properties** window by clicking on .
- Under **Properties** ▶ **Parameters** ▶ **Measuring parameters**, enter the measuring parameters:
  - Select the **External** measuring mode.
  - **Liquid Sample Presentation**: Select the **WL Standard Transmission OMNIS NIR** standard.
  - **Solid Sample Presentation**: Select the **WL Standard Reflection OMNIS NIR** standard.

TEST NOISE – Low flux test

- Insert and select the **TEST NOISE** command in the method.
- Open the **Properties** window by clicking on .
- Under **Properties** ▶ **Parameters** ▶ **Measuring parameters**, enter the measuring parameters:
  - Select the **Low flux test** measuring mode.
  - **Liquid Sample Presentation**: Select the **ND Standard Transmission 4 (OD 1.0) OMNIS NIR** standard.
  - **Solid Sample Presentation**: Select the **ND Standard Reflection 2 (R10) OMNIS NIR** standard.

TEST NOISE – High flux test

- Insert and select another **TEST NOISE** command in the method.
- Open the **Properties** window by clicking on .
- Under **Properties** ▶ **Parameters** ▶ **Measuring parameters**, enter the measuring parameters:
  - Select the **High flux test** measuring mode.
  - **Liquid Sample Presentation**: Select the **ND Standard Transmission 0 (OD 0) OMNIS NIR** standard.
  - **Solid Sample Presentation**: Select the **ND Standard Reflection 5 (R99) OMNIS NIR** standard.

TEST PHOTOMETRIC LINEARITY

- Insert and select the **TEST PHOTOMETRIC LINEARITY** command in the method.
- Open the **Properties** window by clicking on  .
- Enter the reference standard under **Properties** ▶ **Parameters** ▶ **Measuring parameters**:
  - **Liquid Sample Presentation**:
    ND Standard Transmission 1 (OD 0.1) OMNIS NIR
    ND Standard Transmission 2 (OD 0.3) OMNIS NIR
    ND Standard Transmission 3 (OD 0.6) OMNIS NIR
    ND Standard Transmission 4 (OD 1.0) OMNIS NIR
    ND Standard Transmission 5 (OD 1.7) OMNIS NIR
  - **Solid Sample Presentation**:
    ND Standard Reflection 1 (R05) OMNIS NIR
    ND Standard Reflection 2 (R10) OMNIS NIR
    ND Standard Reflection 3 (R40) OMNIS NIR
    ND Standard Reflection 4 (R80) OMNIS NIR
    ND Standard Reflection 5 (R99) OMNIS NIR

Executing external instrument performance tests

Observe recommended execution intervals *(see "Instrument performance tests", chapter 10.1, page 165)*.

To perform the tests, proceed in the same way as for the internal instrument performance test. To place the reference standards, follow the instructions in the **Curves and data** ▶ **Live data** area.

NOTICE

Incorrect standard

If the placed standard and the standard selected in the corresponding command do not match, then the test results will be incorrect.

- The serial number of the placed standard must match the serial number displayed in the. **Curves and data** ▶ **Live data** area.

4 Preparing the model development

Models for liquid samples are developed with functional units of the **Liquid Sample Presentation** type. Models for solid samples are developed with functional units of the **Solid Sample Presentation** type.

Model development begins with the collection of calibration samples and validation samples.

Collecting samples

Collect the samples for the development of a model with care:

- The samples should include any typical sample variations to be expected in the future, as well as seasonal fluctuations or environmental conditions.
- The samples should be evenly distributed over the range of variations.
- Preferably, collect separate sample sets for the calibration and the validation.
- All samples should be handled in the same way.

Quantification

Metrohm recommends a minimum number of approx. 50 samples, or approx. 20 samples for a first model. The more variations in conditions, chemical components or particle sizes that must be covered, the more samples that are required.

1. A spectrum is acquired for each sample.
2. The reference value for the parameter of interest will be measured by the reference method, e.g., titration, for each sample.
   If multiple reference values per sample are available for a particular parameter of interest, then the arithmetic mean value of the reference values should be calculated for each sample. The mean value is used as the reference value for the respective sample. Each mean value should be calculated from the same number of reference values. In this case, the figures of merit will be expressed relative to a defined number of reference values.

If the sample is not altered or destroyed by the reference scan, then the measurements can also be performed in reverse order.

Identification

The samples must cover the expected variations for each product. The products can have a different number of samples; the minimum number is 3.

- A spectrum is acquired for each sample.
- The identity of the sample must be known.

Qualification

The calibration samples must cover the expected variations. The minimum number of samples in the calibration dataset is 3.

As an option, validation samples can be assigned to the positive validation dataset or the negative validation dataset.

- A spectrum is acquired for each sample.

Workflows

An illustration of the runs in the OMNIS Software can be found in the Appendix:

- Acquiring spectra of the calibration samples *(see "Acquiring spectra of the calibration samples", page 184)*
- Recording reference values or the product names *(see "Recording reference values or the product names", page 183)*

4.1 Preparing the spectra acquisition

A spectrum must be acquired for each calibration sample and validation sample.

Acquire only 1 spectrum for each sample. For heterogeneous solids, use the **Multi-point measurement** option (see below).

To prepare the spectra acquisition, create a method, an operating procedure, a sample profile, and a sample list as follows.

Setting up the method

Prerequisite:

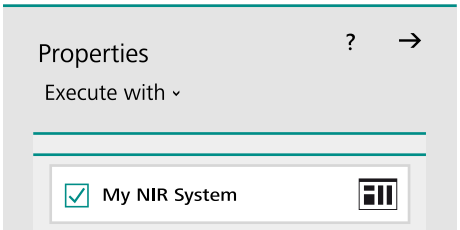
A suitable work system is created *(see "Creating a work system", chapter 3.1, page 31)*.

1 Creating and naming the method

- Under **Processes** ▶ **Methods**, click on .
- Open the **Properties** window by clicking on .
- Enter a matching name in the **Name** field under **Properties** ▶ **General**.

2 Assigning a work system

- Under **Properties** ▶ **Execute with**, select the work system to be used:
  - For liquid samples, select a work system that contains a functional unit of the **Liquid Sample Presentation** type.
  - For solid samples, select a work system that contains a functional unit of the **Solid Sample Presentation** type.



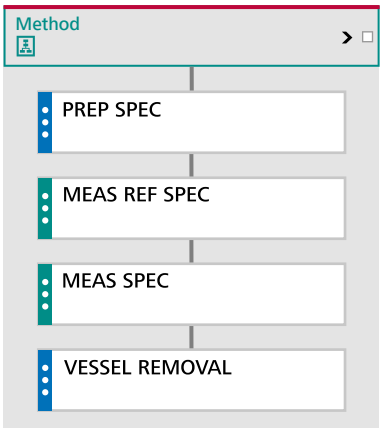
3 Inserting commands

- Open the **Library** window by clicking on .
- Under **Library** ▶ **Commands**, search for the following commands and use drag and drop to insert them into the method:
  - Only for liquid sample presentation: **PREP SPEC** prepares the analysis of liquid samples.
  - **MEAS REF SPEC** records the reference spectrum.
  - **MEAS SPEC** acquires the spectrum of a sample.
  - Only for liquid sample presentation: **VESSEL REMOVAL** is used for controlled removal of the sample vessel from the sample holder of the liquid sample presentation.

Observe the correct sequence of the commands:

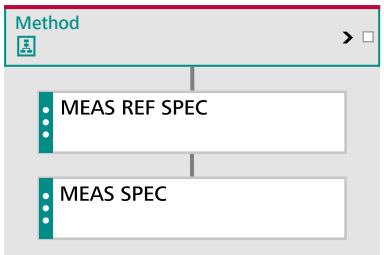
Liquid samples

Basic structure



Solid samples

Basic structure



The absorption spectrum of the sample is calculated, based on the reference spectrum and the acquired spectrum of the sample.

There is only 1 reference spectrum per functional unit. Each execution of the **MEAS REF SPEC** command overwrites the previous reference spectrum.
For this reason, the **MEAS SPEC** command always uses the most recently acquired reference spectrum of the respective functional unit.

Metrohm recommends giving each command a meaningful name under **Properties** ▶ **General**.

4 Configuring the MEAS SPEC command parameters (only for solid sample presentation)

- Select the **MEAS SPEC** command.
- Open the **Properties** window by clicking on  .
- Under **Properties** ▶ **Parameters** ▶ **Measuring parameters**, enter the measuring parameters:
  - Select the holder to be used for measuring the sample.
  - Select the measuring mode that determines the number of measurements. Recommendation:
    Multi-point measurement for heterogeneous solids.
    Single-point measurement for homogeneous solids.
  - Select the sample vessel to be used for measuring the sample.

5 Configuring the PREP SPEC command parameters (only for liquid sample presentation)

The **PREP SPEC** command enables a temperature control. The temperature of the sample or sample holder can be regulated to a value between 25 °C and 80 °C *(see "Temperature control (Liquid Sample Presentation)", chapter 2.5, page 29)*.

In addition, the **PREP SPEC** command ensures that the inserted sample holder fits the stipulated sample vessel. Otherwise the determination is canceled. If no sample vessel is used, then a prompt to insert the sample will appear.

- Select the **PREP SPEC** command.

- Open the **Properties** window by clicking on  .
  Open the **Parameters** subsection.
  - Under **Sample vessel**, select the type used and the exact designation of the sample vessel.
  - Switch the temperature control on or off under **Temperature control**. If required, specify the location of the temperature control and the target temperature. For the temperature control of the sample, select the **Sample vessel** option.
    Note: The target temperature must not be more than 5.0 K below the ambient temperature.
  - If required, select the associated **VESSEL REMOVAL** command.

NOTICE

Damage to the temperature sensor

A temperature sensor will be in direct contact with the sample vessel as long as the temperature is controlled at the sample vessel. To avoid damaging the temperature sensor, the temperature sensor must be moved away from the sample vessel before the sample vessel is removed. The **VESSEL REMOVAL** command is used for this.

6 Configuring the VESSEL REMOVAL command parameters (only for liquid sample presentation)

The **VESSEL REMOVAL** command can ensure removal of the sample vessel. The process sequence is interrupted until the sample vessel has been removed. This enables a controlled run with series determinations.

If the temperature is controlled at the sample vessel, then the temperature sensor is moved away from the sample vessel. The sample vessel can be removed without damaging the temperature sensor as soon as the prompt to remove the sample vessel appears.

Temperature control can be deactivated or continued on the sample holder.

- Select the **VESSEL REMOVAL** command.

- Open the **Properties** window by clicking on .
  Open the **Parameters** subsection.
  - Activate the **Ensure removal of the sample vessel** option. This interrupts the process sequence and prompts the user to remove the sample vessel from the sample holder. The process sequence will be continued as soon as the sample has been removed.
  - For the **Sample holder temperature control** parameter, enable the **Continue** option. This continues an existing temperature control on the sample holder, regardless of the previous location of the temperature control.

7 Saving a method

- Validate the method by clicking on .
- Save the method by clicking on or pressing the **[CTRL]**+**[S]** keys.

Creating an operating procedure

1 Creating an operating procedure

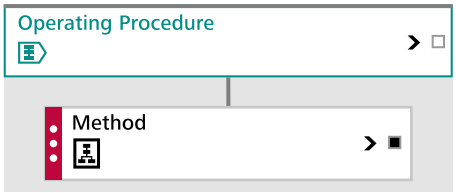
- Under **Processes** ▶ **Operating procedures**, click on .

2 Naming the operating procedure

- Open the **Properties** window by clicking on .
- Enter a matching name in the **Name** field under **Properties** ▶ **General**.

3 Inserting the method

- Open the **Library** window by clicking on .
- Use drag and drop to insert the created method from **Library** ▶ **Methods** into the operating procedure.



4 Saving the operating procedure

- Click on or press the **[CTRL]+[S]** keys.

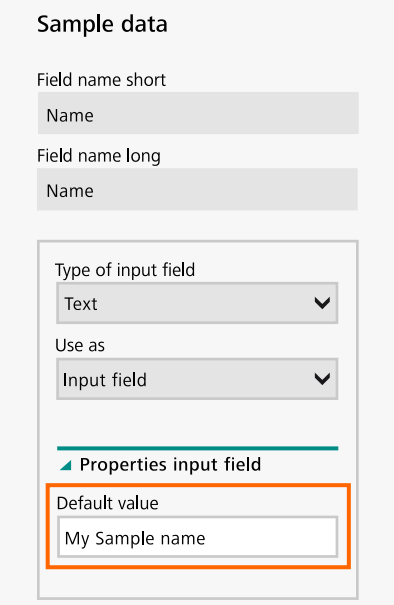
Creating a sample profile

1 Creating and naming the sample profile

- Under **Samples** ▶ **Sample profiles**, click on .
- Enter a matching name in the **Name of the sample profile** **Name of the sample profile** field.

2 Input field for the sample name

The **Sample data** area contains a field for the name of the sample:



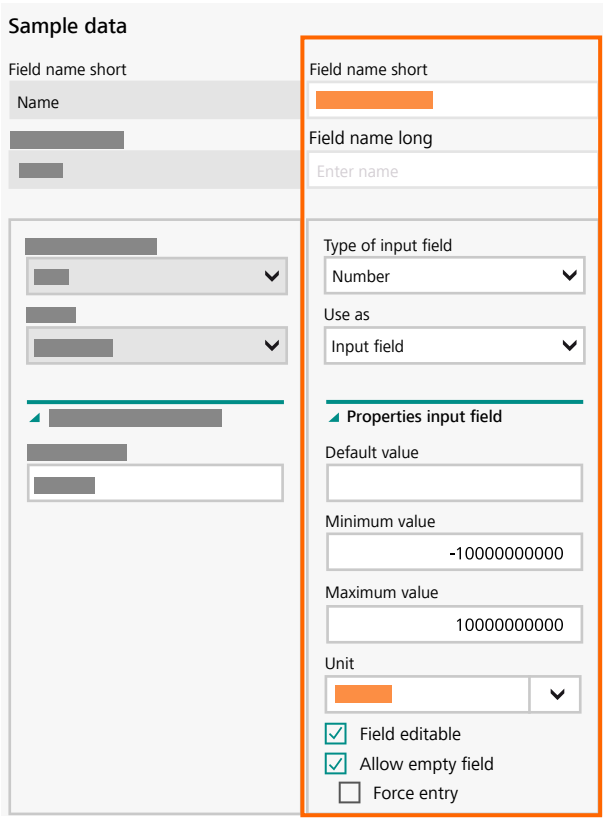
- Enter a **Default value** for the sample name.

3 For quantification: Adding the input field for the reference parameter

Only sample data can be used as reference parameters, not subsample data.

- In the **Sample data** area, add an input field by clicking on . A new input field is added on the right.
- **Field name short**: Enter the name that should be used as column header in the sample list.

- **Field name long**: Optionally, enter the name that should be used as designation in reports.
  Note: If the **Field name long** field is empty, then the name in the **Field name short** field will be used in the reports.
- **Type of input field**: **Number**.
- **Use as**: **Input field**
- In the **Properties input field** section:
  - Enable the **Allow empty field** check box.
    Disable the **Force entry** check box.
  - Leave the **Default value** field empty.
  - Enter the **Unit** in which the reference parameter is specified.
  - Optionally, change the **Minimum value** and the **Maximum value** for the input field.
  - To be able to edit the input field, make sure that the **Editable field** check box is enabled.



Multiple reference parameters

If more than one parameter of interest must be predicted, then add a separate input field for each reference parameter *(see "Multiple parameters of interest (quantification)", chapter 9.1.1, page 156)*.

To delete an input field, right-click on **Field name short** and select **[Delete input field]** in the context menu.

4 For identification and verification: Adding the input field for the product parameter

Only sample data can be used as product parameters, not subsample data.

- In the **Sample data** area, add an input field by clicking on  . A new input field is added on the right.
- **Field name short**: Enter the name that should be used as column header in the sample list.
- **Field name long**: Optionally, enter the name that should be used as designation in reports.
  Note: If the **Field name long** field is empty, then the name in the **Field name short** field will be used in the reports.

Product names can be entered in the sample list, either as text or as a list selection. If verifications are carried out later, use the list selection.

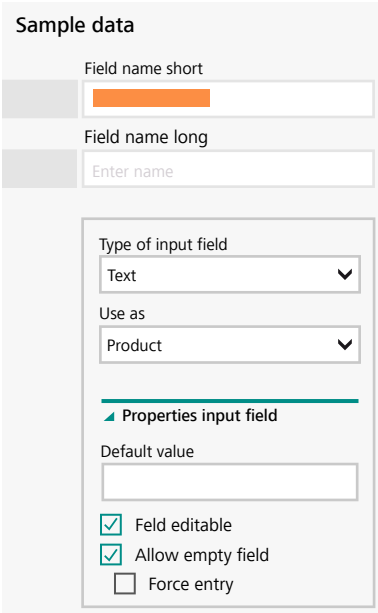
Write product names in the text field:

- **Type of input field**: **Text**.
- **Use as**: **Product**
- Fill in the **Properties input field** section if required.

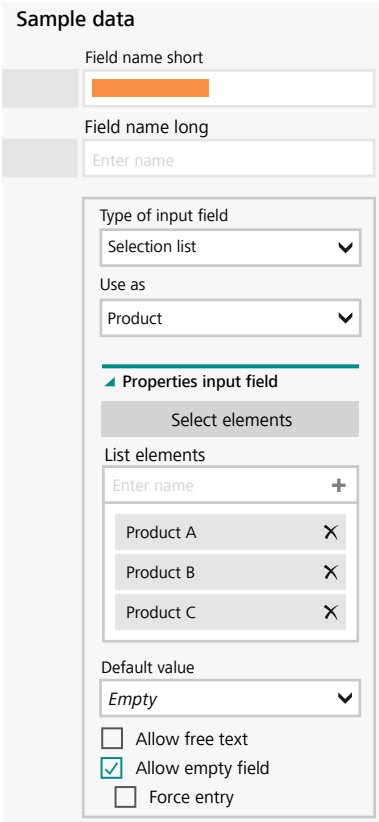
Select product names from the list:

- **Type of input field**: **Selection list**.
- **Use as**: **Product**
- In the **Properties input field** section, select the product names from a model or add them manually:
  - **Select elements**: Click on **Select elements**. Select an identification model or a model hierarchy. Click on **Select** to transfer the product names from the model.
  - **Add elements manually**: Under **List elements**, enter the desired product names and add each entered product name by clicking on **+**.
- If it should also be possible to enter free text in addition to the predefined list elements, enable the **Allow free text** check box.
- Make further settings as required.

Write product names in the text field

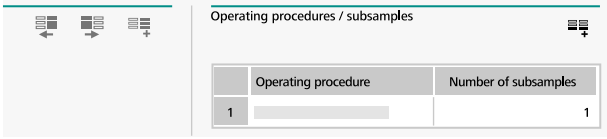


Select product names from the list



5 Defining the operating procedure and the number of subsamples

- In the **Operating procedures / subsamples** area, select the created operating procedure.
- The Number of subsamples defines how many subsamples are added automatically for each sample. Enter: **1**.



6 Saving the sample profile

- Click on or press the **[CTRL]+[S]** keys.

If multiple sample profiles are required (e.g. for different products):

1. Select the sample profile already created under **Samples** ▶ **Sample profiles**.

2. Duplicate the selected sample profile by clicking on .
3. Open the duplicated sample profile and make the necessary adjustments.

Creating a sample list

1 Creating and naming the sample list

- Under **Samples** ▶ **Sample lists**, click on .
  A new tab opens.
- Enter a matching name in the **Name** field.

2 Adding samples

- In the selection list to the left of the icon, select the created sample profile.

  Subsequently added samples are created according to the specifications in the selected sample profile.
- Add a new sample to the sample list by clicking on . Add as many samples as needed.

  Each line of the sample list contains a sample marked with the icon. The sample data follows to the right. This is followed by the subsample marked with and the subsample data.
  The samples are created according to the specifications in the selected sample profile:
  - Quantification: With the defined input field for the reference parameter and its unit, if a unit has been defined.
    Identification and verification: With the defined input field for the product parameter.
  - Each sample contains 1 subsample which uses the defined operating procedure.

Sample name	My reference parameter	No.	Subsample name	Operating procedure
Sample 1	%	1	Subsample 1	
Sample 2	%	2	Subsample 2	
Sample 3	%	3	Subsample 3	

Figure 5 Sample list (example for quantification)

- Edit the sample names and subsample names as needed.

- If the reference values (quantification) or the product names (identification, verification) are already known, then enter these in the corresponding fields.

3 Saving the sample list

- Click on or press the **[CTRL]**+**[S]** keys.

If multiple sample lists are required (e.g. for different products):

1. Under **Samples** ▶ **Sample lists**, select the sample list already created.
2. Duplicate the selected sample list by clicking on .
3. Open the duplicated sample list and make the necessary adjustments.

Reference values or product names can also be entered in the sample list in other ways (see *Metrohm Knowledge Base*).

- Manually during the determination, e.g. in a pop-up window.
- For quantification, also automatically from a subsequent or previous determination with the reference method.

The sample lists and processes are ready for acquiring the spectra *(see "Acquiring spectra", chapter 4.2, page 59)*.

4.2 Acquiring spectra

WARNING

Flammable substances on a hot surface

Risk of fire and burns if flammable substances are spilled. Samples, sample vials, sample holders and the sample presentation can reach temperatures of up to 85 °C.

- Avoid sources of ignition.
- Use protective grounding.
- Use exhaust equipment.
- Clean up spilled liquids and solids immediately.

CAUTION

Volume expansion of the sample due to heating

Injuries and damage to health due to overflowing or breakage of the sample vessel or due to the stopper being blown off.

- Fill the sample vessel only up to the minimum filling height of 2 cm. The liquid can expand in the remaining volume of air.
  Alternatively, use stoppers a with capillary bore.
- Press the stopper in gently so that the sample vessel is not damaged.

CAUTION

Hot sample vials

Burns to the skin due to contact with hot surfaces or hot liquids. Samples, sample vials, sample holders and the sample presentation can reach temperatures of up to 85 °C.

- Wear personal protective equipment and heat-resistant protective gloves.
- Clean up spilled liquids and solids immediately.

Acquiring the spectra for model development

Prerequisites:

- The spectra acquisition is prepared *(see "Preparing the spectra acquisition", chapter 4.1, page 49)*.
- The spectrometer is reserved *(see "Reserving and releasing the instruments", chapter 2.4, page 27)*.
- The correct sample holder is inserted. The sample holder must be matched to the sample vessel to be used.

1 Opening a sample list

- Open the **Samples** work area.
- If the sample list has been closed, then open the sample list with a double-click under **Samples** ► **Sample lists**.

Quantification: The input fields for the reference parameter may still be empty at this point. The reference values can be determined and entered after the spectra acquisition.
Identification and verification: The product names can be entered before or after the spectra acquisition.

2 Adding additional samples (optional)

If additional samples are needed:

- In the selection list to the left of the icon, select the created sample profile.

  Subsequently added samples are created according to the specifications in the selected sample profile.
- Add new samples to the sample list by clicking on .
- Edit the sample names and subsample names as needed.
- Click on to save the sample list.

3 Carrying out the determinations

NOTICE

Damage to the temperature sensor during temperature control on the sample vessel

If the sample vessel is removed while the sensor is in direct contact with the sample vessel, then the sensor may be damaged.

- Do not remove the sample vessel until the measurement is complete and the temperature sensor has been moved away from the sample vessel.

- Select the subsample that should be analyzed, using any of the following ways:
  - Select the subsample by clicking on the icon.
  - For analysis purposes, it is sufficient to select a single cell of the subsample.
- Prepare the corresponding physical sample.
  Insert the sample vessel into the sample holder.
- Start the determination by clicking on . A number on the button indicates how many subsamples will be executed.
- The operating procedure assigned to the subsample is started. Follow any instructions that may appear in the **Curves and data** ▶ **Live data** area. If the temperature is controlled at the sample vessel, then do not remove the sample vessel until prompted.
  As soon as the analysis is successfully completed, the status of the subsample will be displayed as .
- Carry out the determinations for all further samples the same way.

The target temperature must not be more than 5.0 K below the ambient temperature.

If the processes are suitable for series determinations, then multiple subsamples can be selected simultaneously. Alternatively, starts all executable subsamples in the sample list.

- Liquid samples: The **VESSEL REMOVAL** command enables series determinations.
- Solid samples: User actions must be provided for carrying out series determinations (e.g., with the **WAIT** command).

Visual inspection of the spectra

A visual inspection of the spectra enables identification of noisy wavelength ranges and possible erroneous measurements.

Prerequisite:

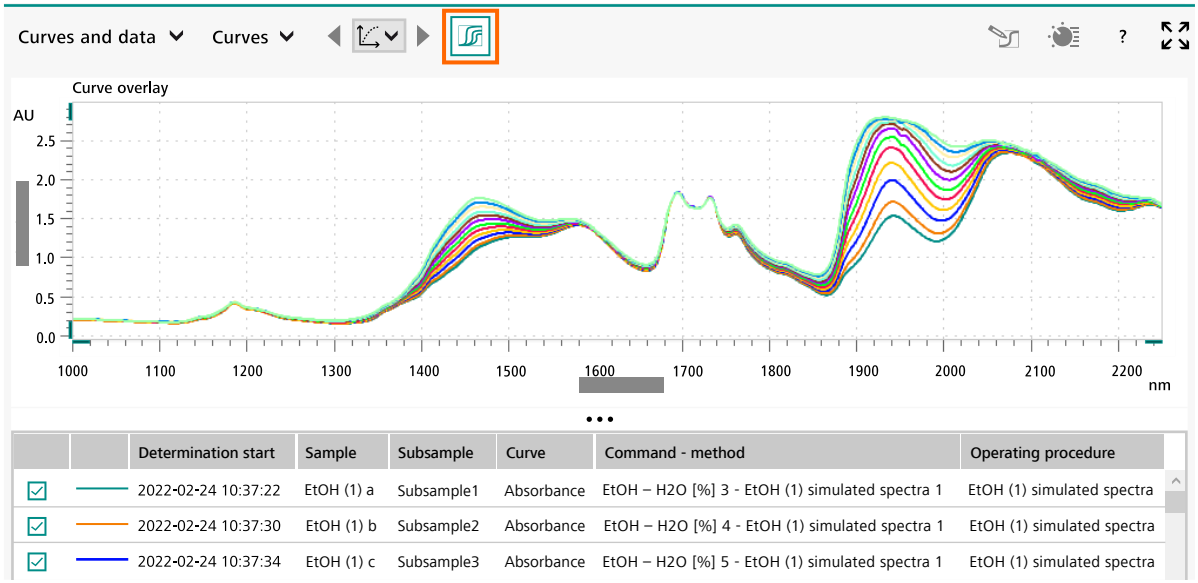
The analysis of the subsamples was successfully completed.

1 Opening the 'Curves' subsection

- In the sample list tab, open **Curves and data** ▶ **Curves**.

2 Displaying and checking spectra

- Display a single spectrum:
  - Select the corresponding subsample in the sample list (marked with the icon).
- Displaying multiple spectra:
  - Enable the curve overlay by clicking on .
  - Select multiple subsamples in the sample list by using the **[CTRL]** key or the **[SHIFT]** key.
- Check the spectra *(see "Handling the charts", chapter 11.3, page 168)*.



		Determination start	Sample	Subsample	Curve	Command - method	Operating procedure
☑		2022-02-24 10:37:22	EtOH (1) a	Subsample1	Absorbance	EtOH – H2O [%] 3 - EtOH (1) simulated spectra 1	EtOH (1) simulated spectra
☑		2022-02-24 10:37:30	EtOH (1) b	Subsample2	Absorbance	EtOH – H2O [%] 4 - EtOH (1) simulated spectra 1	EtOH (1) simulated spectra
☑		2022-02-24 10:37:34	EtOH (1) c	Subsample3	Absorbance	EtOH – H2O [%] 5 - EtOH (1) simulated spectra 1	EtOH (1) simulated spectra

Reference values (quantification) and product names (identification, verification)

1 Reference method (quantification)

- Measure the reference values of the samples using a suitable reference method, e.g., titration.

2 Entering reference values or product names

- In the sample list, enter the reference values or the product names into the corresponding fields.

Add sample data in the sample list

If there was no sample data for the reference parameter or the product parameter at the start of the measurement, then an input field can be added as follows:

- Select the samples for which an input field is to be added by clicking on . To select all samples, click on .
- Right-click on the selected samples to open the context menu and select **Add sample data**.
- Add sample data for the reference parameter or the product parameter *(see "Preparing the spectra acquisition", chapter 4.1, page 49)*.

3 Saving the sample list

- Click on or press the **[CTRL]**+**[S]** keys.

If multiple sample lists have been prepared, then process each sample list as described above.

Quantification: To develop a quantification model, continue with *Quantification model, chapter 5, page 65.*.

Identification, verification: To develop an identification model, continue with *Identification model, chapter 6, page 102.*.

Qualification: To develop a qualification model, continue with *Qualification model, chapter 7, page 120.*.

5 Quantification model

5.1 Creating the quantification model

An illustration of the runs in the OMNIS Software can be found in the Appendix *(see "Developing a model", page 184)*.

Multiple parameters of interest
To predict more than one parameter of interest for each sample, create a separate model for each parameter *(see "Multiple parameters of interest (quantification)", chapter 9.1.1, page 156)*.

Creating the quantification model

Prerequisite:

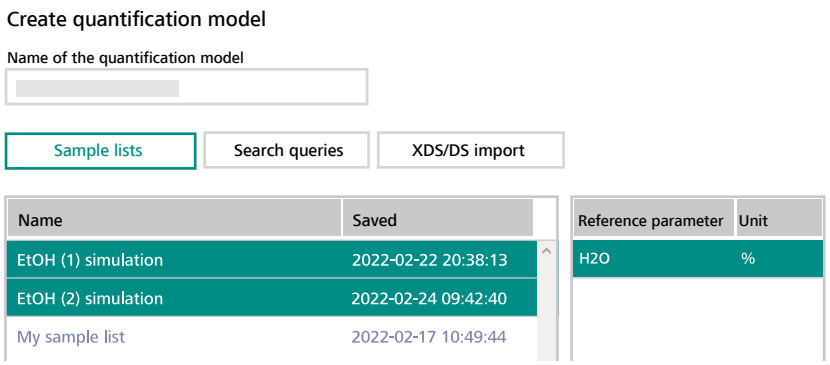
- Spectra for the development of the model are acquired *(see "Acquiring spectra", chapter 4.2, page 59)*.

1 Creating and naming the quantification model

- Under **Calibration and evaluation** ▶ **Quantification models**, click on .
  A new quantification model appears in a new tab.
- Enter a matching name in the **Name of the quantification model** input field.

2 Selecting the samples and the reference parameters

- Display all sample lists by clicking on **Sample lists**.
- Select all of the sample lists that were prepared for the development of the model.



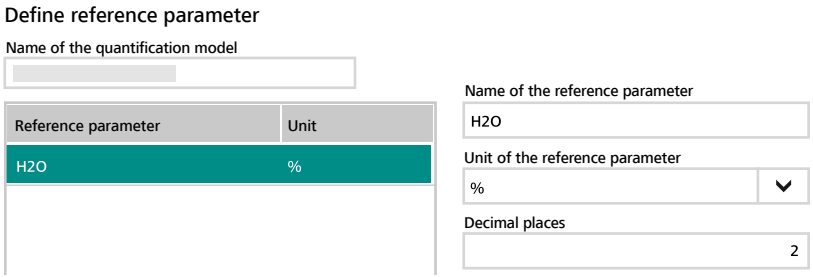
Samples can also be selected from a search query.
Samples from XDS devices and DS devices can also be imported if required *(see "Switching from XDS/DS Analyzers (quantification)", chapter 11.6, page 178)*.

The sample selection can be adjusted later.

- The **Reference parameter** list shows all sample data that is suitable as reference parameters.
  Select the reference parameter for which the model is developed.
  If the reference parameter has different designations, then select all designations.
- Click on **[Continue]**.

3 Defining the reference parameters

- The **Reference parameter** list shows all of the designations for the reference parameter that were selected in the previous step.
  Select all required designations from this list.



- Enter the name that the model should use in the **Name of reference parameter** field.
- Select the **Unit of reference parameter** that the model is to use.
- Enter the number of **Decimal places** for the depiction of the results.

All spectra that have the selected designations of the reference parameter are added to the model.

4 Automatic or manual model development

If the model is to be developed automatically but the sample selection needs to be adjusted beforehand, then continue with manual model development.

- **Automatic model development**
  Automatic model development with the **OMD (OMNIS Model Developer)** and the selected samples. The model can be published, validated or further developed after completion of the automatic model development.
  - Click on **[Start OMD]**.
    The duration of the OMD execution depends on the number of spectra.
  - Continue with *chapter 5.2, Automatic model development – OMD, page 67*.
- **Manual model development**
  - Click on **[Create]**.
    The new model opens in a tab.
  - Save the model: Click on or press the **[CTRL]+[S]** keys.
  - Continue with *chapter 5.3, Manual model development, page 70*.

5.2 Automatic model development – OMD

The **OMD (OMNIS Model Developer)** automates the development of quantification models, presents a selection of the most suitable models, and assesses their predictive ability.

Prerequisite:

The OMD was started with **[Start OMD]**.

1 Checking the calculated quantification models

Once the calculations are complete, the OMD offers 5 models to choose from.

Calculated quantification models

Name of the quantification model

Decimal places: 4

Model#	SEC	SECV	SEP	R^2P
1	0.0019	0.0020	0.0030	0.990
2	0.0017	0.0018	0.0030	0.990
3	0.0019	0.0020	0.0031	0.990
4	0.0012	0.0014	0.0023	0.994
5	0.0012	0.0015	0.0023	0.994

The models are sorted according to their predictive ability. Figures of merit are listed for each model.

The models are marked with the following colors on the left-hand side of the table:

- Models highlighted in **green** have good predictive ability. If the number of samples is sufficiently large, then the model will work well with all unknown samples of the same type. The figures of merit provide a reliable estimate of future errors.
- Models highlighted in **yellow** have medium predictive ability. If the number of samples is sufficiently large, then the model can be expected to perform well. The figures of merit may be too optimistic for future samples. A separate validation is recommended.
- Models highlighted in **red** do not have sufficient predictive ability. The model has serious defects. It should not be used.

If a quantification model can be improved even further, then suggestions for its improvement will be displayed in the tooltip for the model.

2 Checking the figures of merit

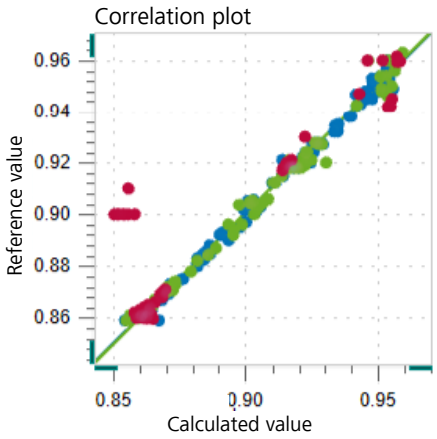
The following standard errors are shown for each model:

- **SEC**: Standard error of calibration.
- **SECV**: Standard error of cross validation.
- **SEP**: Standard error of prediction. This figure is the best estimate for the prediction error when analyzing unknown samples. The SEP is displayed only if a validation dataset is available.
  Note: The OMD creates a validation dataset only if at least 100 spectra remain after the outlier detection.

Model 1 has the best predictive ability, but not necessarily the smallest standard error.

3 Checking the correlation plot

Clicking on an individual model causes the associated Correlation plot to be displayed.



The **Correlation plot** enables an assessment of the model's performance at a glance. The plot visualizes the correlation between the values calculated with the model (x-axis) and the reference values (y-axis).

Each point represents a sample:

- Blue points represent the samples in the calibration dataset.
- Green points represent the samples in the validation dataset (if available).
- Red points represent the samples in the outlier dataset (if available).

A regression line is laid through the blue or green points in such a way that the relationship between the reference values and the calculated values is described as well as possible.

Judging a correlation plot

- The slope of the blue and green regression line should be as close as possible to 1, the y-intercept as close as possible to 0.
- The blue and green points should be as close as possible to the corresponding regression line.

The regression line and the points can overlap.

4 Validating, further developing or publishing the models

The model must first be published before it can be used in determinations and repredictions.

Publishing one of the 5 models directly

- If the OMD was started when the model was created:
  - Select a model and click on **[Save and publish]**. The other 4 models are discarded.
  - The last published version is displayed under **Calibration and evaluation ▶ Quantification models**.
    The **PREDICT** command can now access the published version of the model.
- If the OMD was started in an open model:
  - Select a model and click on **[Edit]**.
  - Save the model: Click on or press the **[CTRL]**+**[S]** keys.
  - Continue with *chapter 5.4, Publishing a quantification model, page 92*.

Validating or further developing one or more models

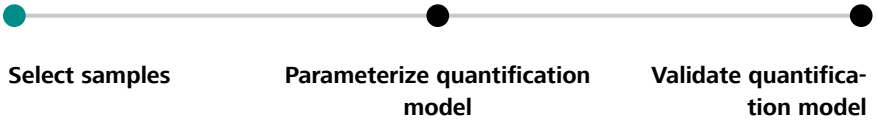
- Select one or more models. For a multiple selection, use the **[SHIFT]** key or the **[CTRL]** key.

- Click on **[Edit]**.
  Each selected model is opened in a new tab.
- Save the new models: Click on in the corresponding tabs or press the **[CTRL]+[S]** keys.
- Continue with *chapter 5.3, Manual model development, page 70*.

5.3 Manual model development

5.3.1 Selecting the samples and splitting the dataset

The tab of the quantification model shows a horizontal navigation bar at the top, the **navigator**. The navigator guides you through the next steps of model development.



Depiction of spectra

In the 3 process steps, the individual spectra are displayed in the form of curves, points or table cells.

Selected spectra are highlighted simultaneously in all depictions and in all process steps.

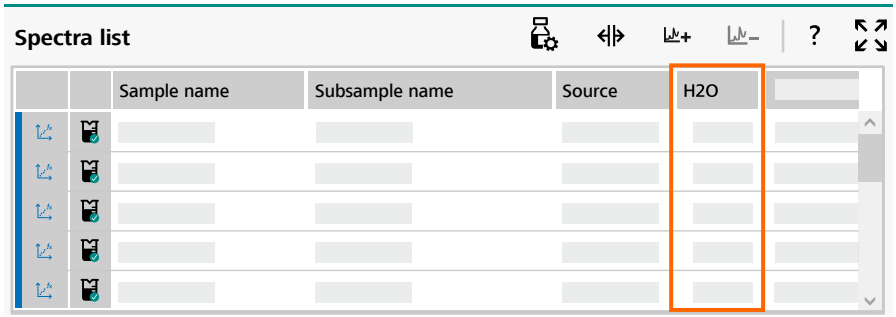
Tables and charts

The handling of tables and charts is described in the appendix:

- Handling the tables *(see "Handling the tables", chapter 11.2, page 167)*
- Handling the charts *(see "Handling the charts", chapter 11.3, page 168)*

Select samples process step

The **Spectra list** area shows the spectra of the selected samples:



An input field shows in each case the associated reference value (highlighted in orange in the picture).

The **Select samples** process step enables the following:

- **Adjusting the sample selection**
  Add additional spectra or delete spectra.
- **Splitting the dataset**
  Automatic or manual splitting of the dataset:
  - **Calibration dataset**: The model is calculated using the spectra and reference values of the calibration dataset.
  - **Validation dataset**: The spectra and reference values of the validation dataset are used solely to validate the model.
  - **Outlier dataset**: The outlier dataset has no influence on the model or its validation. Outliers are shown only in some charts, and then for information purposes.

A model can be developed without a validation dataset, e.g., if only a limited number of samples are available in an initial phase.

Adjusting the sample selection (optional)

Prerequisite:

- In the **Calibration and evaluation** work area, the model is open and at the front *(see "Creating the quantification model", chapter 5.1, page 65)*.
- The navigator is in the **Select samples** process step.

1 Adding or deleting spectra

Sample selection and reference parameters can be adjusted in the **Spectra list** area at any time:

- To select samples whose spectra should be added to the spectra list, click on .
- To remove spectra from the spectra list, select the spectra and click on .
  Note: The associated samples and their spectra are retained in the database.
- Click on to change the following:
  - the name or unit of the reference parameter
  - the selection of designations for the reference parameter
  - the number of decimal places for the reference parameter and for all results

If the reference value of a spectrum must be edited, then open the respective sample list or search query and double-click on the reference value.

2 Saving the model

- Click on or press the **[CTRL]**+**[S]** keys.

Switch to automatic model development

The other settings in this tab have no influence on the **OMD (OMNIS Model Developer)**. A switch to automatic model development can therefore be made at this point:

- If necessary, check the uniform distribution of the reference values in the histogram *(see "Histogram", page 75)*.
- The OMD independently searches for outliers and excludes them from the model development.

  However, if outliers are to be excluded manually, then they must be removed from the sample list. An assignment to the outlier dataset has no effect on the OMD.
- Click on **[Start OMD]**.

  The duration of the OMD execution depends on the number of spectra.

Significance level for outlier detection

Prerequisite

- In the **Calibration and evaluation** work area, the quantification model is open and at the front.

1 Editing the properties of the model

- Open the **Properties** window by clicking on .
- Under **Properties** ▶ **Parameters** ▶ **Outlier limits**, define the **Significance level** for outlier detection. The higher the significance level, the more spectral outliers will be detected. Typical values are 5% or 1%.

  The significance level is used as follows:
  - The optional automatic outlier detection during model development takes into account the significance level at the time of outlier detection (see below).
  - The outlier detection when predicting sample properties takes into account the significance level at the time the model is published.

2 Saving the model

- Click on or press the **[CTRL]**+**[S]** keys.

Determining the outlier dataset and the validation dataset

Outlier detection enables the automatic creation of an outlier dataset. The remaining spectra can be automatically split into a calibration dataset and a validation dataset.

If separate samples were collected for calibration and validation, then the samples can be assigned manually.

Prerequisite:

- In the **Calibration and evaluation** work area, the model is open and at the front.
- The navigator is in the **Select samples** process step.

1 Calling up the dataset splitting

- Click on  in the **Spectra list** area.

The **Dataset splitting** dialog opens.

2 Determining the outlier dataset

- To assign spectra to the outlier dataset automatically, enable the **Detect outliers** toggle switch. The automatic outlier detection detects the following types of outliers:
  - Spectral outliers due to deviations in the spectra
  - Reference value outliers due to anomalies in the reference values

  Adjust the **Significance level** if required. The higher the significance level, the more spectral outliers will be detected. Typical values are 5% or 1%.

3 Determining the validation dataset

The automatic splitting is done in a way which ensures that the calibration dataset and the validation dataset are representative of the population and independent of one another.

- To automatically assign spectra to the validation dataset, activate the **Determine validation dataset** toggle switch.
  - In the **Percentage** field, define the percentage of spectra that is to be used for the validation dataset, e.g., between 20% and 30%.

4 Defining the options

Define the options for the dataset assignment:

- **Apply parameterization**: Apply data preprocessing and wavelength selection to the spectra *(see "Parameterizing the quantification model", chapter 5.3.4, page 84)*.
  Note: Subsequent changes to the parameterization or the significance level will not affect the dataset assignment. Unless the dataset is to be split again.
- **Keep outliers**: Retain existing outliers and do not take them into account in the split. This option can lead to an increase in the size of the outlier dataset, even if the **Significance level** is not changed.
- **Keep validation dataset**: Retain the existing validation dataset and do not take it into account in the split. This option leads to an increase in the size of the validation dataset, even if the **Percentage** is not changed.

5 Starting the automatic splitting

- Click on **[Split]**.

The split of the dataset corresponds to the settings made.

6 Checking the splitting

As soon as at least one spectrum is selected in the spectra list, the selected spectra will be highlighted in the **Spectra overlay** area.

In the **Histogram** and **Spectra overlay** areas, spectra in the calibration dataset are marked **blue**, spectra in the validation dataset are marked **green**, and spectra in the outlier dataset are marked **red**.

In the **Spectra list** area, the assignments are represented by the following icons:

	The spectrum is assigned to the calibration dataset.
	The spectrum is assigned to the validation dataset.
	The spectrum is assigned to the outlier dataset.
	Indicates missing or invalid data. Consult the tooltip.

7 Manual splitting (optional)

A manual splitting can be done with or without prior automatic splitting.

- Right-click on a spectrum to open the context menu. Assign the spectrum to the corresponding dataset:
  - **Calibration dataset**
  - **Validation dataset**
  - **Outlier dataset**

Assigning multiple spectra at once:

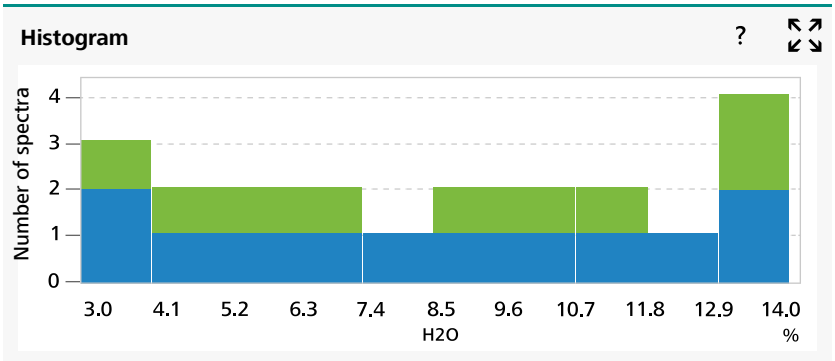
- Optionally, put the spectra in a suitable order. Sort the overview list by clicking on a column header.
- Select multiple spectra by using the **[CTRL]** key or the **[SHIFT]** key.
- Right-click on the selection to open the context menu. Assign the selected spectra.

8 Saving the model

- Click on or press the **[CTRL]**+**[S]** keys.

Histogram

The histogram illustrates how evenly the reference values are distributed. For this purpose, the histogram divides the reference value range into 10 equal-sized bins.



In the example shown, 12 integer reference values from 3% bis 14% are distributed across the 10 bins. The first bin comprises the reference values 3% and 4%; the last bin comprises the reference values 13% and 14%. The other 8 bins include only 1 reference value each.

The example confirms that the spectra in the calibration dataset (blue) and in the validation dataset (green) are evenly distributed across the range of reference values.

Outliers

Spectra assigned to the outlier dataset are displayed in red. They can be either spectral outliers or reference value outliers.

Outliers should be investigated. If an outlier proves to be a valid spectrum with a valid reference value, then it can be assigned to either the calibration dataset or the validation dataset.

5.3.2 Calculating the quantification model

A first model may be calculated without parameterization. This provides a first comparative benchmark for the figures of merit. The influence of a subsequent parameterization can be better assessed.

If noise or other artifacts make some wavelengths unusable, then these wavelengths can be excluded from the start *(see "Parameterizing the quantification model", chapter 5.3.4, page 84)*.

Calculating the model

Prerequisite:

- In the **Calibration and evaluation** work area, the quantification model is open and at the front.

1 Starting the calculation

- Calculate the model by clicking on **[Calculate]**.

If the **[Calculate]** button is disabled, then the following causes may apply:

- The model has already been calculated and no changes have been made since then.
- One of the process steps contains an incorrect entry. The process step of the affected area is displayed in **red** in the navigator. The field with the incorrect entry is outlined in red.

5.3.3 Validating the quantification model

Defining the cross-validation method

Prerequisite:

- In the **Calibration and evaluation** work area, the quantification model is open and at the front.

1 Editing the properties of the model

- Open the **Properties** window by clicking on  .

- Under **Properties** ▶ **Parameters** ▶ **Cross-validation**, define the cross-validation method:
  - For spectra lists with up to 70 spectra, the **Leave-One-Out** procedure is recommended.
  - For larger spectra lists, the **K-fold** procedure is recommended. The larger the **Number of blocks**, the longer the calculation of the model takes. A typical value for k is 5.
    The **Splitting algorithm** determines how spectra of the calibration dataset are split into individual blocks. The splitting algorithm **Random** selects the blocks at random. The **Fixed Blocks (DUPLEX)** splitting algorithm selects the blocks in a reproducible manner.

2 Saving the model

- Click on or press the **[CTRL]**+**[S]** keys.

Validating the quantification model

Prerequisite:

- In the **Calibration and evaluation** work area, the quantification model is open and at the front.
- The quantification model is calculated *(see "Calculating the quantification model", chapter 5.3.2, page 76)*.

1 Switching to the validation process step

- In the navigator, click on **Validate quantification model** to switch to the validation process step.

The calculated quantification model data is displayed in the **Figures of merit**, **Correlation plot**, and **Influence plot** areas.

The charts **Loading plot** and **Score plot** can also be displayed by clicking on .

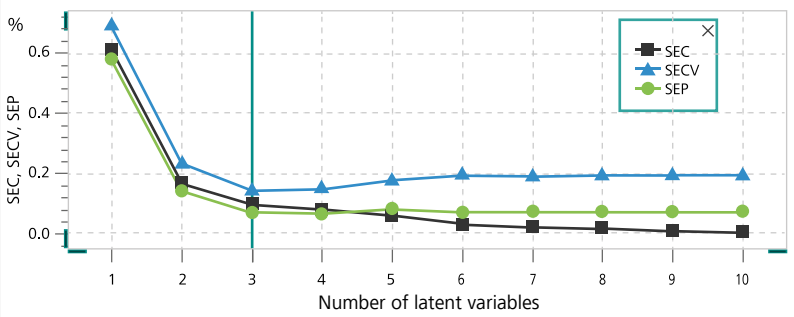
2 Checking the figures of merit

The **Figures of merit** area displays a chart with the following figures of merit:

- **SEC**: Standard error of calibration.
- **SECV**: Standard error of cross validation.
- **SEP**: Standard error of prediction. This figure is the best estimate for the prediction error when analyzing unknown samples. The SEP is displayed only if a validation dataset is available.

The figures of merit (y-axis) are presented for different numbers of latent variables (x-axis).

Figures of merit



The vertical green line indicates the currently selected number of latent variables. In the figure above, 3 latent variables were selected. With 3 latent variables, the SECV has an initial minimum value of 0.14%.

Number of decimal places in the table

To change the number of decimal places for the figures of merit, click on in the **Select samples** process step in the **Spectra list** area.

3 Defining the number of latent variables

The final quantification model uses a fixed number of latent variables. Finding the optimum number of latent variables is fundamental for the quantification model performance.

More latent variables explain more spectral variations in the calibration dataset. But too many latent variables explain too-specific variations or noise, resulting in less accurate predictions with unknown samples. This is called **overfitting**.

Fewer latent variables may lead to a more reliable quantification model. However, if the number of latent variables is too small, then relevant spectral variations will not be captured. The predictions will then be less accurate. This is called **underfitting**.

- Provisionally select a reasonable number of latent variables. To accomplish this, double-click the corresponding line in the table.

  The number of selected latent variables is indicated by ✓ in the table.
  In case of doubt, select the number suggested by the OMNIS Software. If the number of latent variables selected differs from the number suggested by the system, then the suggested number is indicated by →.

4 Checking the correlation plot

The **Correlation plot** shows at a glance an evaluation of the quantification model performance. The plot visualizes the correlation between calculated values (x-axis) and reference values (y-axis). Each point represents a sample.

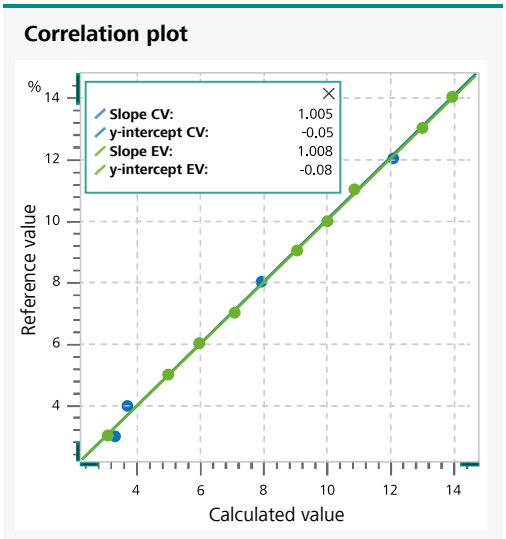
The correlation plot and the table next to it display for each sample the following values:

Reference value	Value for the reference parameter
Calculated value	Result of the quantification model
Residual	Difference between calculated value and reference value

Number of decimal places in the table
To change the number of decimal places for the above values, click on in the **Select samples** process step in the **Spectra list** area.

The blue points generate a blue regression line. The green points generate a green regression line.

The regression line reveals the systematic relationship between calculated values and reference values. Ideally, the regression line has a slope of 1 and an intercept of 0. Ideally, all samples are located directly on the line. In this case, the calculated value corresponds to the reference value for each sample.



The correlation plot reveals different kinds of errors:

- **Systematic errors** can be seen as deviations of the regression line from the ideal line (slope = 1, y-intercept = 0).
- **Random errors**: The more scattered the points around the regression line, the higher the number of random errors.

In the figure, several points are hidden behind other points. The blue regression line is hidden behind the green regression line.

- Select a different number of latent variables in the **Figures of merit** area. Observe the changes in the correlation plot.

Handling the chart

The display of the chart can be adjusted and individual or multiple points can be selected *(see "Handling the charts", chapter 11.3, page 168)*.

5 Checking the influence plot

The **Influence plot** describes the characteristic properties of the spectra and helps identify spectral outliers.

An influence plot can be displayed for either the PLS or the PCA calculation method. Select a calculation method from the list:

- **PLS** (Partial least squares regression)
  PLS uses the relevant information from both the spectra and the reference values. PLS is the basis of the quantification model.
- **PCA** (Principal Component Analysis)
  PCA extracts the relevant information from the spectra.

Both influence plots, PLS and PCA, take into account the defined data preprocessings and wavelength ranges *(see "Parameterizing the quantification model", chapter 5.3.4, page 84)*.

Reference values influence the PLS influence plot, but not the PCA influence plot. The only exception is that any point can be highlighted as a potential outlier due to an extreme reference value.

The selected number of latent variables influences the PLS influence plot, but not the PCA influence plot. For PCA, the number of principal components is chosen so that the explained variance is at least 95%.

Example: PLS influence plot of the EtOH spectra, based on 3 latent variables

Handling the chart

The display of the chart can be adjusted and individual or multiple points can be selected *(see "Handling the charts", chapter 11.3, page 168)*.

Each point represents a spectrum. High values for Hotellings T^2 and Q residuals indicate possible outliers.

Spectra with high values for Hotellings T^2 indicate an extreme composition of the relevant samples. These samples have a major influence on the model. If the reference value of such a sample is incorrect, then the prediction of similar samples can lead to incorrect results.

Spectra with high Q residuals indicate features that have not been successfully modeled. For example, because the relevant samples contain unusual chemical components.

The dashed lines show the critical values (limit values) for the specified significance level.

The above figure does not show any potential outliers. All points are well within the dashed lines.

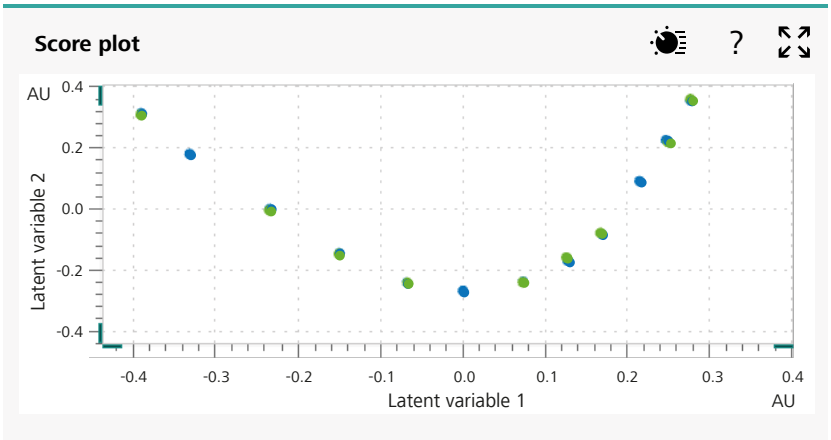
6 Checking the score plot

While the Hotellings T² value of a spectrum combines the scores of all latent variables in a single value, the score plot enables an even more detailed analysis of the scores.

The score plot for quantification models is based on the **PLS** calculation method and takes into account the defined data preprocessing and wavelength ranges *(see "Parameterizing the quantification model", chapter 5.3.4, page 84)*.

Each point represents a spectrum. The scores for the first two latent variables can be read on the x-axis and the y-axis. Every other pair of latent variables can also be displayed under **Properties**. The scores are normalized, each latent variable is assigned the same weight.

Example: Score plot of the EtOH spectra for the latent variables 1 and 2:



Handling the chart

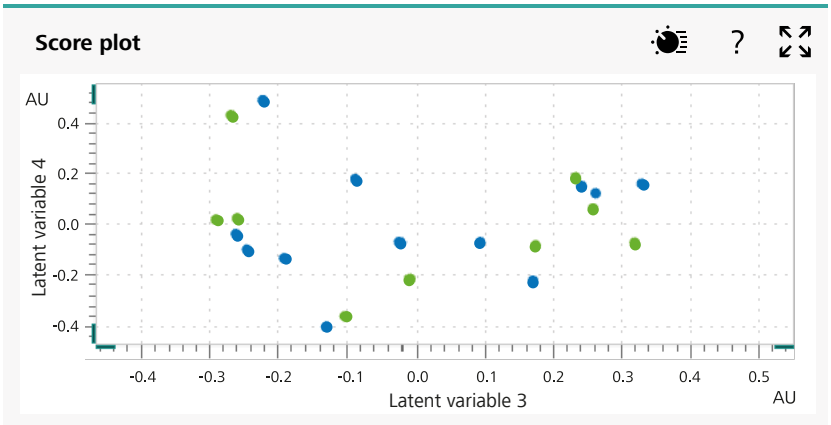
The display of the chart can be adjusted and individual or multiple points can be selected *(see "Handling the charts", chapter 11.3, page 168)*.

The points are lined up like a string of pearls. This is due to the regular intervals between the reference values and the absence of additional sample variations. When this is the case, the question can

be raised as to whether all of the sample variations to be expected in the future have been taken into account.

In the case of higher latent variables, which explain an ever-diminishing portion of the variance, the effect is no longer visible, as the following figure shows.

Example: Score plot of the EtOH spectra for the latent variables 3 and 4:



The validation dataset (green points) takes up approximately the same space within the latent variables shown in the two figures as the calibration dataset (blue points). No potential outliers are to be seen.

7 Excluding or including outliers

- Check potential outliers carefully.
- If samples are assigned to another dataset, assign them all in one step:
  - Select all points to be reassigned in either the influence plot, the correlation plot or the score plot *(see "Selecting multiple points or curves", page 170)*.
  - Right-click on one of the selected items to open the context menu. Select the matching dataset.
  - Click on **[Calculate]** to recalculate the quantification model.
- After the spectra are reassigned, validate the quantification model once again.

8 Saving the model

- Click on or press the **[CTRL]**+**[S]** keys.

Duplicating the quantification model

The quantification model can be duplicated in order to access the current status if required:

1. Save the quantification model.
2. Select the quantification model under **Calibration and evaluation** ▶ **Quantification models**.
3. Duplicate the selected quantification model by clicking on  .
4. Open the duplicated quantification model and continue the optimization.

5.3.4 Parameterizing the quantification model

The **Parameterize quantification model** process step enables automatic or manual optimization of the spectra. Artifacts and nonlinearities are corrected. If carried out correctly, parameterization improves the accuracy and robustness of the model.

The parameterization is applied to:

- all spectra in the calibration dataset
- all spectra in the validation dataset and in the outlier dataset

During prediction in the **Samples** work area, the spectrum of a sample is acquired and analyzed with a model. The parameterization defined in the model is also applied to this spectrum.

2 parameterization options are available:

- Defining the wavelength ranges to be used.
- Apply data preprocessing to bring the spectra into a more suitable form.

Automatic parameterization

Optimize parameterization

Prerequisites:

- In the **Calibration and evaluation** work area, the model is open and at the front.

1 'Parameterize quantification model' process step

- Click on the **Parameterize quantification model** process step in the navigator.

2 Defining data preprocessing and wavelength ranges automatically

- In the **Spectra overlay** area, click on the **[Optimize parameterization]** button.
  Note: Existing data preprocessing and wavelength ranges are overwritten.

3 Manual parameterization (optional)

- If required, the data preprocessing and wavelength ranges can undergo further manual processing.

Manual parameterization

Manual parameterization begins with a visual examination of the spectra in the **Select samples** process step.

Depicting the spectra

Prerequisite:

- In the **Calibration and evaluation** work area, the model is open and at the front.

1 'Select samples' process step

- Click on the **Select samples** process step in the navigator.

In this process step, the spectra can be investigated simultaneously in tabular form and in curve form.

2 Investigating the spectra

- Handling the tables *(see "Handling the tables", chapter 11.2, page 167)*
- Handling the charts *(see "Handling the charts", chapter 11.3, page 168)*

3 'Parameterize quantification model' process step

- Click on the **Parameterize quantification model** process step in the navigator.
- In the **Data preprocessing** area, expand the selection list with ⌄ and select the **Loading plot** area.

In this process step, the spectra can be investigated simultaneously in curve form and in **Loading plot**. The Loading plot shows how the original wavelength variables contribute to the structure of each latent variable.

Additional steps

- Manual wavelength selection *(see "Manual wavelength selection", chapter 5.3.4.1, page 86)*
- Define data preprocessing manually *(see "Defining data preprocessing manually", chapter 5.3.4.2, page 89)*

5.3.4.1 Manual wavelength selection

A wavelength selection can possibly improve the quantification model. Example: If noise is visible at high absorbance values, then the relevant wavelength ranges can be excluded.

The model uses the defined wavelength ranges. If no wavelength ranges are defined, then the model will use all wavelengths.

Displaying the spectra and the loadings

Prerequisites:

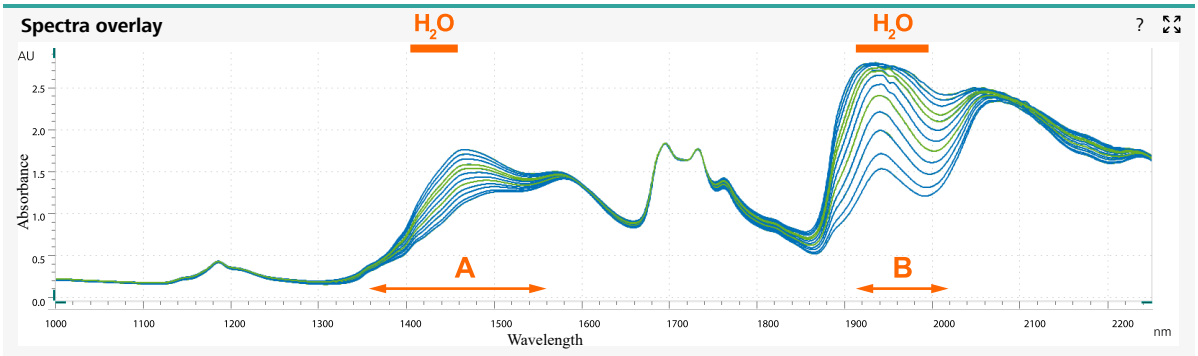
- In the **Calibration and evaluation** work area, the model is open and at the front.

1 'Parameterize quantification model' process step

- Click on the **Parameterize quantification model** process step in the navigator.
- Display the **Spectra overlay**, **Loading plot** and **Wavelength range** areas simultaneously.

Spectra overlay

The generic H_2O absorption bands can be looked up in a chart and used as a rough guide. The H_2O absorption bands extend from 1400 to 1450 nm and from 1900 to 1980 nm.



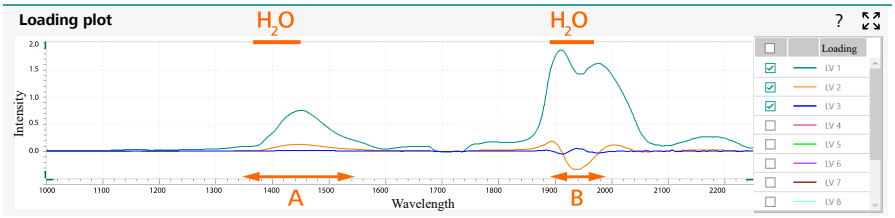
The EtOH spectra show clearly visible variations corresponding to the different H_2O content of the samples (**A** and **B** ranges).

However, there is a difference between the 2 ranges. In contrast to the range **B** (1900–2000 nm), the range **A** (1350–1550 nm) shows uniform

vertical spacing between the lines, corresponding to the uniform spacing of the reference values.

Loading plot

Loadings show how the original wavelength variables contribute to the structure of each latent variable.



The ranges **A** (1350–1550 nm) and **B** (1900–2000 nm) already identified have the highest loadings, especially for the latent variable 1 (green). These ranges therefore contribute the most to the formation of latent variable 1.

It is irrelevant whether the loadings are positive or negative.

Due to the artifacts in the **B** range, it seems sensible to test a model based on the **A** range (1350 to 1550 nm).

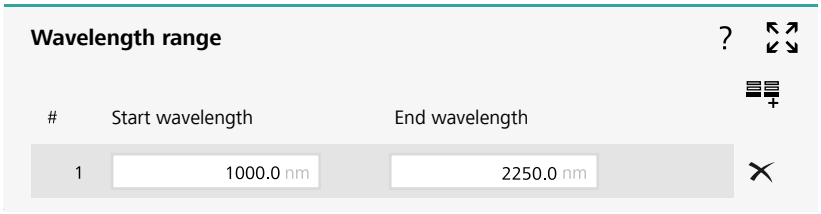
Defining wavelength ranges

Prerequisite:

- In the **Calibration and evaluation** work area, the model is open and at the front.
- The navigator is in the **Parameterize quantification model** process step.

1 Adding the wavelength range

- In the **Wavelength range** area, add a wavelength range by clicking on .



A wavelength range is added. The area initially covers all wavelengths.

2 Defining the wavelength range

Define the wavelength range in one of these ways:

- To define the wavelength range by entering numbers, enter the **Start wavelength** and the **End wavelength** in the corresponding input fields.
- To define the wavelength range in the chart, proceed as follows:
  - Click on **[Activate moving]** in the **Spectra overlay** area.
  - Move the cursor to the left edge of the highlighted area until the cursor is displayed as ↔.
  - Hold down the left mouse button and move the left edge to the corresponding position.
  - Proceed in the same way on the right side of the highlighted area.
  - To move a wavelength range, move the cursor over the area until the cursor is displayed as ⇔. Hold down the left mouse button and move the area to the left or right.
  - Click on **[Deactivate moving]**.

3 Adding additional wavelength ranges

Additional wavelength ranges may be added by clicking on  .

Wavelength ranges may not overlap

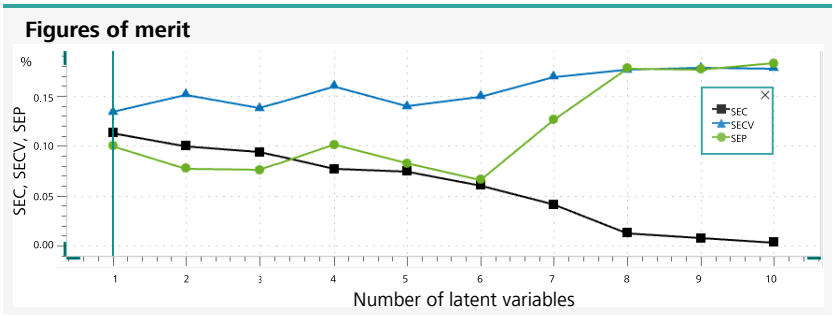
First, a new wavelength range overlaps with the existing wavelength ranges. Adjust the wavelength range so that no overlaps occur.

4 Calculating the model

- Calculate the model by clicking on **[Calculate]**.

5 Validating the model

- In the navigator, click on **Validate quantification model** to switch to the validation process step.
- Validate the model. Compare the figures of merit with the previously created model.



In the case of water in EtOH and with a wavelength range of 1350 to 1550 nm, a single latent variable (instead of 3 latent variables) appears to be sufficient when the entire wavelength range is used. The SECV is similar in both cases: 0.13% and 0.14%.

A single variable instead of 3 variables is a remarkable improvement. Irrelevant variance has been successfully eliminated. The new model with fewer variables is presumably more robust.

6 Saving the model

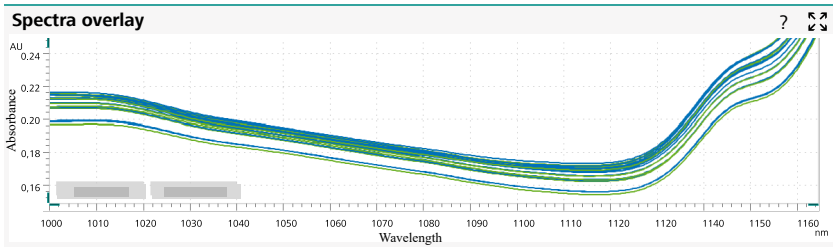
- Click on 🖬 or press the **[CTRL]**+**[S]** keys.

If the newly created wavelength selection is to be taken into account when splitting the dataset or detecting outliers, then the dataset can be split once again.

5.3.4.2 Defining data preprocessing manually

Suitable data preprocessing can improve the quantification model. Example: Baseline shifts do not contain any relevant information for most applications and can be removed.

EtOH spectra



In the case of the EtOH spectra, small baseline shifts between the spectra are visible when zooming into the 1000 to 1200 nm range. These are constant (not wavelength-dependent) baseline shifts.

The baseline shifts are due to the formation of gas bubbles from an intentionally not outgassed sample in a flow cell for demonstration purposes.
A clear liquid does not usually show baseline shifts to this extent.

The baseline shift can be corrected with data preprocessing.

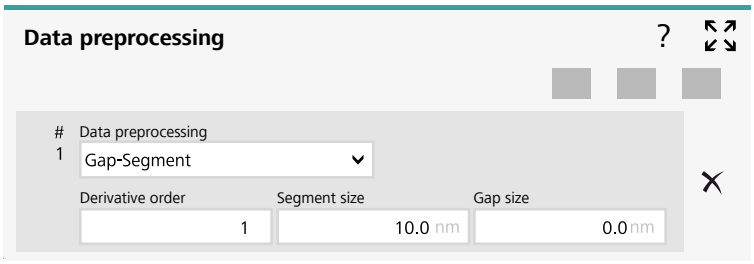
Defining data preprocessing manually

Prerequisites:

- The navigator is in the **Parameterize quantification model** process step.

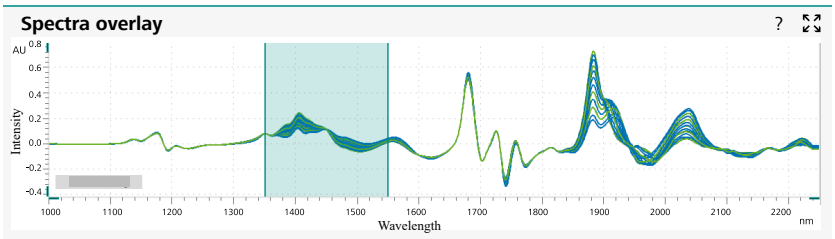
1 Adding the data preprocessing step

- In the **Data preprocessing** area, add a data preprocessing step by clicking on .
- In the **Data preprocessing** field, select the type of data preprocessing and fill in the associated fields.
  Example of gap-segment with a first derivative that removes constant (non-wavelength-dependent) baseline shifts:



The preprocessed spectra are shown immediately in the **Spectra overlay** area.

After the data preprocessing, the spectra look different.



In the case of the EtOH spectra, the constant baseline shifts are removed.

2 Adding additional data preprocessing steps

Additional data preprocessing steps may be added by clicking on .

If multiple data preprocessing steps are used, then the sequence can be decisive. Preferably, gap-segment or Savitzky-Golay is applied prior to SNV, and SNV before Detrend.

Lines can be moved up or down by clicking on and , thus defining the sequence.

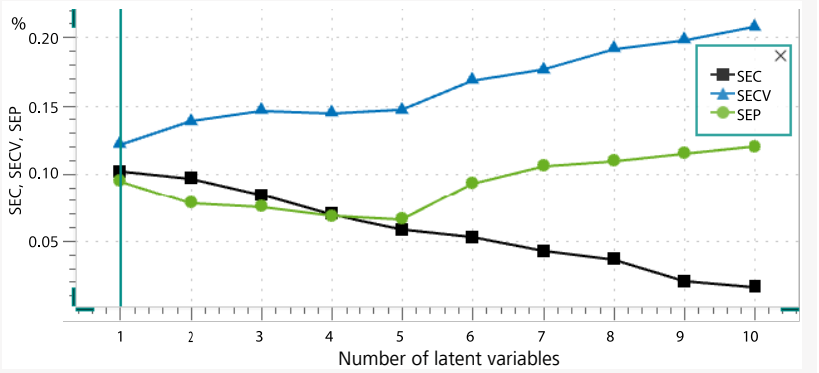
3 Calculating the model

- Calculate the model by clicking on **[Calculate]**.

4 Validating the model

- In the navigator, click on **Validate quantification model** to switch to the validation process step.
- Validate the model. Compare the figures of merit with the previously created models.

Figures of merit



In the case of H_2O in EtOH, with a wavelength range of 1350 to 1550 nm and a gap-segment with derivative order 1, a single latent variable still appears to be sufficient. The SECV has minimally improved to 0.12%.

As the baseline shifts do not contain any relevant information for this application, the data preprocessing can be retained despite the minimal improvement.

5 Saving the model

- Click on or press the **[CTRL]**+**[S]** keys.

If the newly created data preprocessings are to be taken into account when splitting the dataset or detecting outliers, then the dataset can be split once again.

5.4 Publishing a quantification model

The model must first be published before it can be used for determinations. This means that the model can still be developed further without impacting the published version and the determinations carried out with it.

Publish quantification model

Prerequisite:

- The model is calculated and saved.
- The model is open.
- The required number of latent variables is selected.

1 Opening a dialog

- Open the **Publish quantification model** dialog by clicking on .

If the model has been previously published and used in methods, then these methods can be automatically updated by enabling the **Update methods** check box.

Note: Not updated automatically:

- Opened methods
- Signed and published methods
- If the filtering of the data permissions is enabled: Methods without data permissions of the currently logged-in user

2 Nearest Neighbor Distance

If the **Calculate Nearest Neighbor Distance** check box is enabled, then the **Nearest Neighbor Distance (NND)** is available. The model calculates the distance to the nearest calibration sample spectrum for each calibration sample spectrum in the latent variable space. The largest of all distances determined is stored in the **LimitNearestNeighborDistance** (NND limit value) **PREDICT** command variable *(see "Prediction", chapter 2.3.2, page 21)*.

During prediction, the quantification model calculates the distance between the acquired spectrum and the nearest calibration sample spectrum in the same way. This distance is stored in the **Nearest-**

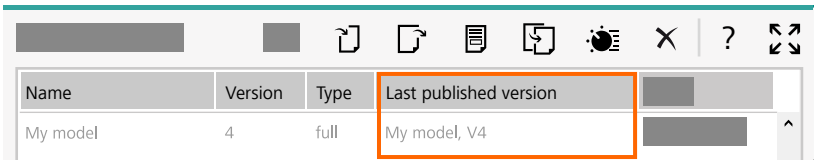
NeighborDistance (NND) **PREDICT** command variable *(see "Prediction", chapter 2.3.2, page 21)*.

The two variables can be compared with one another and monitored using result monitoring *(see "Creating an operating procedure", page 151)*.

3 Publishing

- Publish the model by clicking on **[Publish]**.

The last published version is displayed under **Calibration and evaluation ▶ Quantification models**:



The **PREDICT** command can now access the published version of the model.

Model overview

If one or more quantification models are selected, then the model overview on the right-hand side shows the most important data at a glance (maximum of 5 different models).

If the last saved version does not correspond to the last published version, then the model overview will display both versions.

5.5 Slope/y-intercept correction

The **Slope/y-intercept correction** enables correction of systematic errors in the application of a quantification model. A robust and reliable quantification model is a prerequisite. The errors should be statistically significant, but not too large.

Systematic errors can be seen in the correlation plot as deviations of the regression line from the ideal line (slope = 1, y-intercept = 0).

Random errors can also be seen in the correlation plot. The more scattered the points around the regression line, the higher the random errors. Random errors cannot be corrected with the slope/y-intercept correction.

A slope/y-intercept correction is used in the following cases:

- If a quantification model is revalidated, or monitored with check samples, and it is found that the figures of merit no longer correspond to the requirements, for instance due to environmental changes.
- If a quantification model has been created with imported XDS/DS spectra *(see "Switching from XDS/DS Analyzers (quantification)", chapter 11.6, page 178)*.

2 corrections are available:

- **Bias**: Corrects the bias – the average deviation between the reference values and the predicted values of the samples.
  After the correction, the bias is 0.
- **Slope/y-intercept**: Corrects the slope and the y-intercept of the regression line in the correlation plot.
  After the correction, the slope is equal to 1 (corresponds to a 45° straight line) and the y-intercept is equal to 0.
  Note: After a slope/y-intercept correction, the bias equals 0.

The bias correction and, in particular, the slope/y-intercept correction should be used with caution.
If the errors are not statistically significant, do not apply any correction. If the errors are statistically significant, investigate them thoroughly. If possible, eliminate the cause of the errors. If there is a legitimate reason that the errors cannot be fixed, then a bias correction or a slope/y-intercept correction may be applied.

Note on number of samples:

- A reliable estimate of the bias needs at least 20 samples.
- A reliable estimate of the slope needs at least 30 samples.

Preparing samples

Typically, the slope/y-intercept correction is carried out with samples used for revalidation of the quantification model, or with check samples used for monitoring the performance of the quantification model.

- Assemble the samples in **Sample lists** or **Search queries**.

- Ensure that each sample includes the following:
  - A reference value for the parameter to be corrected.
  - A spectrum.
  - A calculated value for each of the spectra.

Creating the slope/y-intercept correction

Multiple quantification models (optional)

- If different quantification models are to be corrected, then create a separate slope/y-intercept correction for each model.
- If multiple similar versions of the same quantification model are to be corrected, then a single slope/y-intercept correction will be sufficient.

  The similar versions may differ in the following points:
  - Different names of the quantification model
  - Different names of the reference parameter
  - Different outlier datasets, as long as the calibration dataset is not affected as a result
  - Different validation datasets, as long as the calibration dataset is not affected as a result
  - Different cross-validation parameters

  However, the similar versions must not exhibit any differences that could influence the prediction result:
  - The calibration datasets must be identical.
  - The parameterization must be identical.
  - The number of latent variables must be identical.

1 Creating the new slope/y-intercept correction

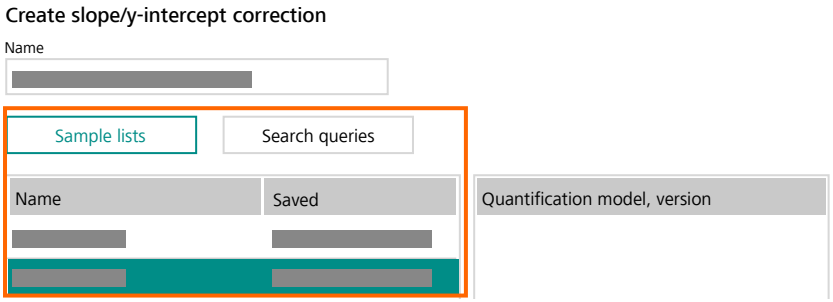
- Under **Calibration and evaluation** ▶ **Slope/y-intercept corrections**, click on  .

2 Naming the slope/y-intercept correction

- Enter a matching name in the **Name** field, e.g., the name of the quantification model to be corrected.

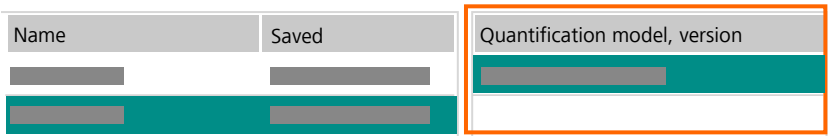
3 Selecting samples

- Select one or more **Sample lists** or **Search queries** from which samples are to be used for the creation of the slope/y-intercept correction.



4 Selecting the quantification model

The **Quantification model** list displays all quantification models that can be corrected with the selected samples.



- In the **Quantification model** list, select the quantification model to be corrected.
  Notice: If the predicted values of the samples used originate from multiple versions of a quantification model, then all of these versions can be selected if necessary.
- Click on **[Continue]**.

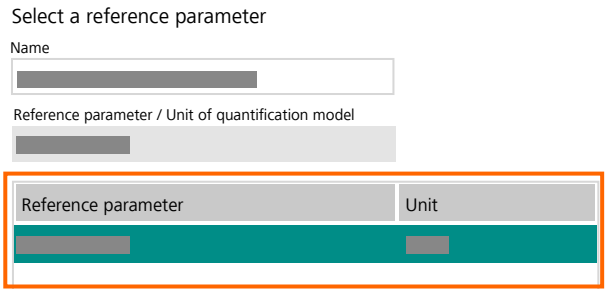
5 Reference parameters of the quantification model

The **Reference parameter/unit of the quantification model** field shows the name and the unit of the reference parameter of the selected quantification model.

- Adjust the number of **Decimal places** if required.

6 Reference parameters of the selected samples

The **Reference parameter** list shows all of the available reference parameters of the selected samples.



- Select the desired reference parameter from this list.

If the reference parameter has several designations in the sample lists or search queries, then all these designations can be selected.

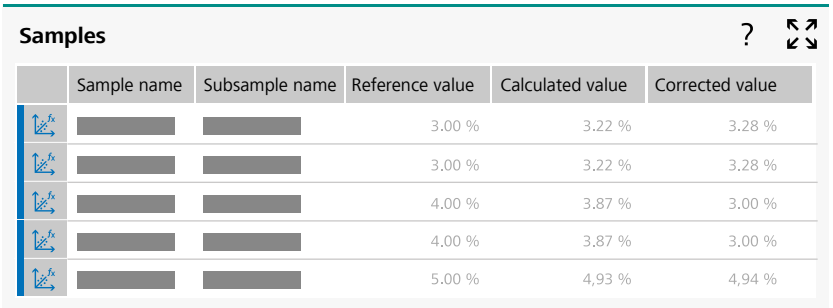
7 Confirming entries

- Create the new slope/y-intercept correction by clicking on **[Create]**.

The correction is calculated from the reference values and the predicted values of the selected samples. All samples that meet the following two conditions will be considered:

- The sample data contains a reference value for the selected reference parameter.
- The sample has a quantification result calculated by the selected quantification model.

A new tab shows the slope/y-intercept correction. The **Samples** area lists the samples that were included.



8 Type of correction

Select the type of correction, **Bias** or **Slope/y-intercept**.

9 Highlighting outliers

- To highlight a spectrum as an outlier, right-click on the spectrum in the **Samples** area and click on **Outlier dataset** to exclude it from the calculation.

The  symbol highlights the spectrum as an outlier. The spectrum is removed from the **Correlation plot** area and from all calculations.

- To remove the outlier marking, right-click on the spectrum once again and click on **Correction dataset**.

The spectrum is highlighted again with  . The spectrum reappears in the **Correlation plot** area and is once again included in all calculations.

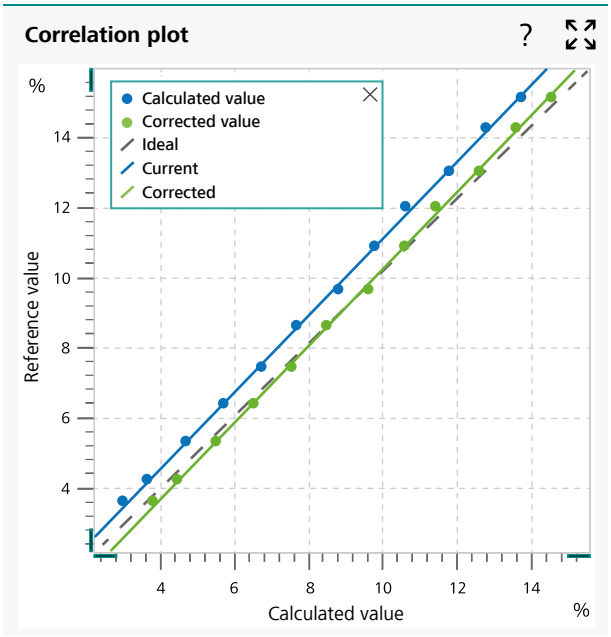
Multiple spectra can be selected and modified at the same time by using either the **[CTRL]** key or the **[SHIFT]** key *(see "Handling the tables", chapter 11.2, page 167)*.

10 Correlation plot

The **Correlation plot** area depicts 2 points for each spectrum with the same reference value on the y-axis, but with different values on the x-axis:

- The blue point shows the **calculated value** predicted by the quantification model on the x-axis.
  - Place the cursor on a point to display its residual, (the difference between calculated value and reference value).
- The green point shows the **corrected value** on the x-axis. This corrected value depends on the selected type of correction (bias or slope/y-intercept).
  - Place the cursor on a point to display its residual, (the difference between corrected value and reference value).

Correlation plot for a bias correction

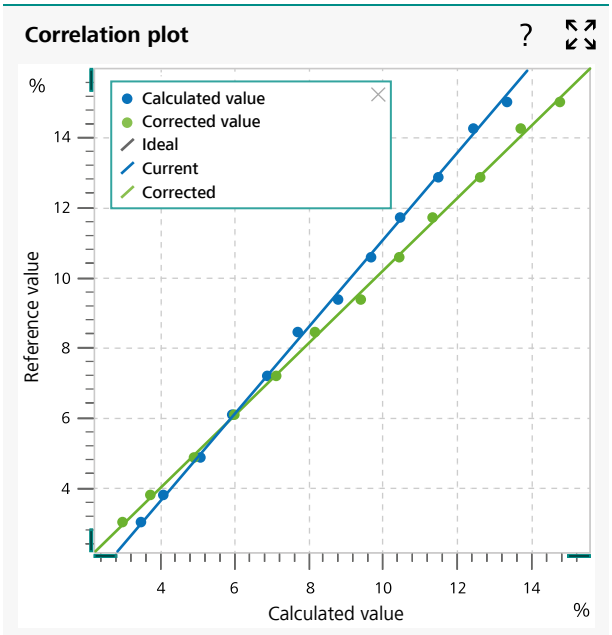


The calculated values (blue points) are corrected for the bias. The corrected regression line (green) intersects the dashed 45° ideal line in such a way that a bias of 0 results.

The example in the above correlation plot results in an SEP improvement from 0.82 to 0.24.

A reliable estimate of the bias needs at least 20 samples.

Correlation plot for a slope/y-intercept correction



The calculated values (blue points) are corrected for the slope and the y-intercept. The corrected values (green points) have an ideal regression line (45°, y-intercept 0).

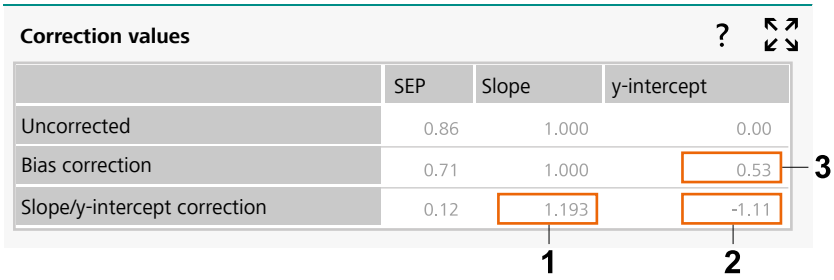
The example in the above correlation plot results in an SEP improvement from 0.86 to 0.12.

A reliable estimate of the slope needs at least 30 samples.

11 Correction values

The SEP and correction values for slope and y-intercept are displayed in the **Correction values** area, depending on the type of correction:

- **SEP** displays the standard error of prediction based on the samples in the correction dataset.
  Note: The formulas for the 3 SEP values take into account in each case the corresponding degrees of freedom. With a small number of samples, the corrected values may therefore be greater than the uncorrected values.
- The multiplicative correction value **Slope** and the additive correction value **y-intercept** convert the calculated values (blue points) into the corrected values (green points):
  Corrected value = Calculated value × Slope + y-intercept



The table contains the following data of the calculated values (blue points):

- The slope of the regression line **(1)**.
- The y-intercept of the regression line **(2)**.
- The bias of the regression line **(3)**.

12 Publishing the slope/y-intercept correction

A published slope/y-intercept correction can neither be opened nor edited retrospectively.

- Check whether the suitable type of correction (bias or slope/y-intercept) has been selected.
- Click on to open the **Publish slope/y-intercept correction** dialog.
- Publish the slope/y-intercept correction by clicking on **[Publish and close]**.

The slope/y-intercept correction is published and saved at the same time. The tab is closed and the slope/y-intercept correction is displayed in the overview list.

The **PREDICT** command can now access the published version of the slope/y-intercept correction.

6 Identification model

An illustration of the runs in the OMNIS Software can be found in the Appendix *(see "Developing a model", page 184)*.

An identification model provides the following, depending on the use:

- An **identification** of an unknown sample (e.g., fructose). The result is a product name.
- A **verification** of the product membership (e.g., fructose) of a sample. The result is yes/no – Verification successful or failed.

6.1 Creating the identification model

Creating the identification model

Prerequisite:

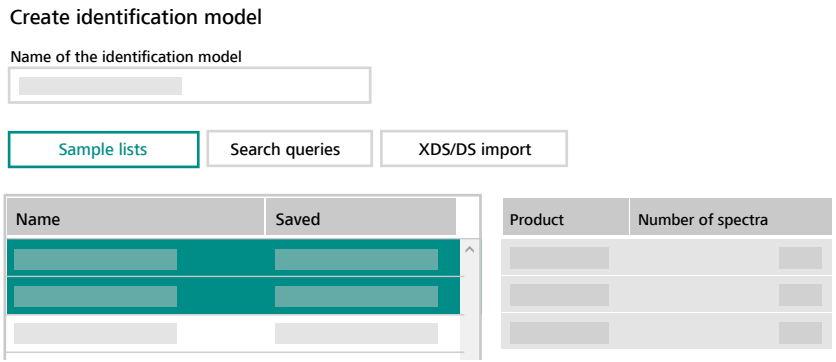
- A dataset with spectra and product names is created *(see "Acquiring spectra", chapter 4.2, page 59)*.

1 Creating and naming the identification model

- Under **Calibration and evaluation** ▶ **Identification models**, click on .
  A new identification model appears in a new tab.
- Enter a matching name in the **Name of the identification model** input field.

2 Selecting the samples

- Display all sample lists by clicking on **Sample lists**.
- Select all of the prepared sample lists.



Samples can also be selected from a search query. Samples from XDS devices and DS devices can also be imported *(see "Switching from XDS/DS Analyzers (quantification)", chapter 11.6, page 178)*.

The sample selection can be adjusted later.

The selection must contain samples with a product parameter. The **Product** column lists the products included.

3 Creating the identification model

- Click on **[Create]**.
- Save the model: Click on or press the **[CTRL]**+**[S]** keys.

6.2 Selecting the samples and splitting the dataset

The tab of the identification model shows a horizontal navigation bar at the top, the **navigator**. The navigator guides you through the next steps of model development.

Depiction of spectra

In the 3 process steps, the individual spectra are displayed in the form of curves, points or table cells.

Selected spectra are highlighted simultaneously in all depictions and in all process steps.

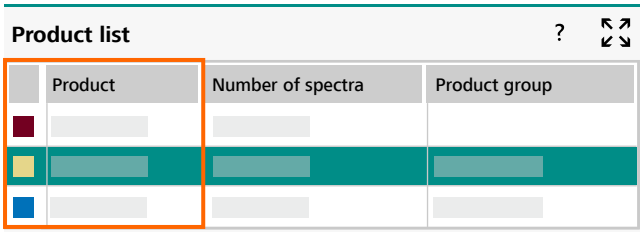
Tables and charts

The handling of tables and charts is described in the appendix:

- Handling the tables *(see "Handling the tables", chapter 11.2, page 167)*
- Handling the charts *(see "Handling the charts", chapter 11.3, page 168)*

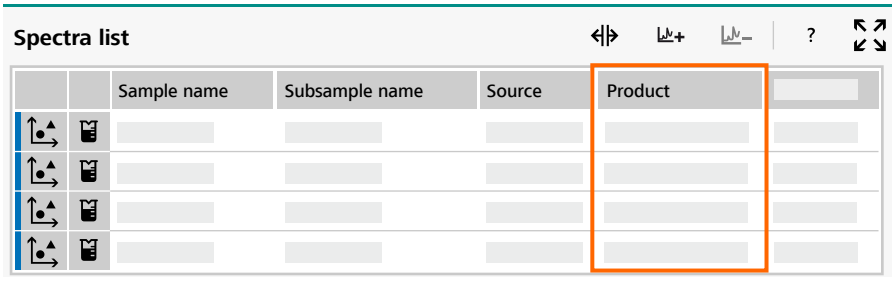
'Select samples' process step

The **Product list** area shows the products of the selected samples:



Each product is identified by a product color. A different color can be selected by clicking on a product color.

As soon as at least one product is selected in the product list, the **Spectra list** area will show all spectra of the selected products:



An input field shows the product membership of the sample (marked orange in the picture).

The following icons symbolize the assignment to the datasets:

	The spectrum is assigned to the calibration dataset.
	The spectrum is assigned to the validation dataset.
	The spectrum is assigned to the outlier dataset.
	Indicates missing or invalid data. Consult the tooltip.

The spectra in the **Spectra list** area also appear in the **Spectra overlay** area and are depicted as follows:

- Spectra in the calibration dataset are marked **blue**, spectra in the validation dataset are marked **green**, and spectra in the outlier dataset are marked **red**.
- If the **Display product colors** toggle switch is activated, then the spectra will be colored according to the product colors.

The **Select samples** process step enables the following:

- **Adjusting the sample selection**
  Add additional spectra or delete spectra.

- **Splitting the dataset**
  Automatic or manual splitting of the dataset:
  - **Calibration dataset**: The model is calculated using the spectra and product memberships of the calibration dataset.
  - **Validation dataset**: The spectra and product memberships of the validation dataset are used solely to validate the model.
  - **Outlier dataset**: The outlier dataset has no influence on the model or its validation. Outliers are shown only in some tables, and then for information purposes.

A model can be developed without a validation dataset, e.g., if only a limited number of samples are available in an initial phase or if the validation is carried out exclusively with an external dataset.

Adjusting the sample selection

Prerequisite:

- In the **Calibration and evaluation** work area, the model is open and at the front *(see "Creating the identification model", chapter 6.1, page 102)*.
- The navigator is in the **Select samples** process step.

1 Adding or deleting spectra

The sample selection and the product membership can be adjusted in the **Spectra list** area at any time:

- To select samples whose spectra should be added to the spectra list, click on .
- To remove spectra from the spectra list, select the spectra and click on .
  Note: The associated samples and their spectra are retained in the database.

2 Changing the product membership

- Select all spectra to which a different product is to be assigned.
- Right-click on the selected spectra and select **Assign product** in the context menu.
  An input window appears:
  - Click in the **New product** field. Either select an existing product or enter a new product name.
  - Assign the product to the selected spectra by clicking on **[Assign]**.

3 Saving the model

- Click on or press the **[CTRL]**+**[S]** keys.

Splitting the dataset automatically

Outlier detection enables the automatic creation of an outlier dataset. The remaining spectra can be automatically split into a calibration dataset and a validation dataset.

If separate samples were collected for calibration and validation, then the samples can be assigned manually.

Prerequisite:

- In the **Calibration and evaluation** work area, the model is open and at the front.
- The navigator is in the **Select samples** process step.

1 Calling up the dataset splitting

- Click on in the **Spectra list** area.

The **Dataset splitting** dialog opens.

2 Determining the outlier dataset

- To assign spectra to the outlier dataset automatically, enable the **Detect outliers** toggle switch. The automatic outlier detection detects spectral outliers based on deviations in the spectra.
  - Adjust the **Significance level** if required. The higher the significance level, the more spectral outliers will be detected. Typical values are 5% or 1%.

3 Determining the validation dataset

The automatic splitting is done in a way which ensures that the calibration dataset and the validation dataset are representative of the population and independent of one another.

- To automatically assign spectra to the validation dataset, activate the **Determine validation dataset** toggle switch.
  - In the **Percentage** field, define the percentage of spectra for the validation dataset, e.g. between 20% and 30%.

4 Defining the options

Define options for dataset splitting:

- **Apply parameterization**: Apply data preprocessing and wavelength selection to the spectra *(see "Parameterizing the identification model", chapter 6.5, page 113)*.
  Note: Subsequent changes to the parameterization will not affect the dataset assignment. Unless the dataset is to be split again.
- **Keep outliers**: Retain existing outliers and do not take them into account in the split. This option can lead to an increase in the size of the outlier dataset, even if the **Significance level** is not changed.
- **Keep validation dataset**: Retain the existing spectra in the validation dataset and do not take it into account in the split. This option leads to an increase in the size of the validation dataset, even if the **Percentage** is not changed.

5 Starting the automatic splitting

- Click on **[Split]**.

The split of the dataset corresponds to the settings made.

6 Saving the model

- Click on or press the **[CTRL]**+**[S]** keys.

Influence plot and score plot

After automatic dataset splitting, the influence plot and score plot charts are available:

- In the **Select samples** process step, click on in one of the areas and select the **Influence plot** or the **Score plot** chart.

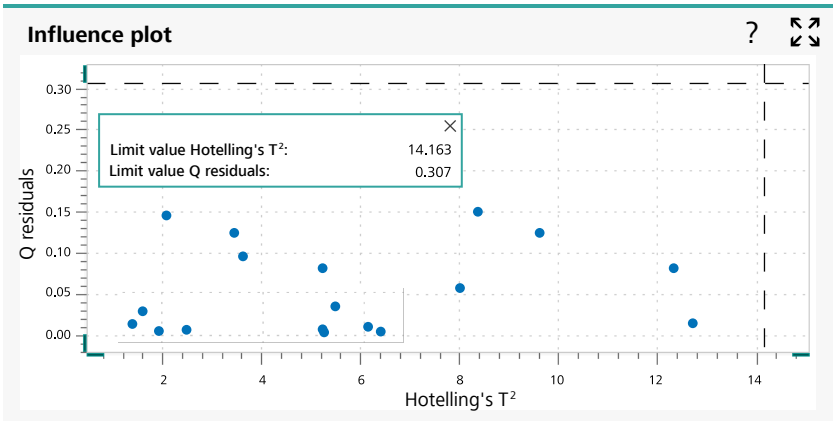
The influence plot and the score plot are based on the **PCA** calculation method (principal component analysis). The number of principal components is chosen in such a way that the explained variance is at least 95%.

The spectra in the following form serve as the starting point for the PCA:

- Spectra without data preprocessing and wavelength selection if the **Apply parameterization** option has been deactivated for automatic dataset splitting.
- Spectra with data preprocessing and wavelength selection if the **Apply parameterization** option has been activated for automatic dataset splitting.
  Note for when this option is activated: If the parameterization is changed, then the influence plot and the score plot will not be available until after a new automatic dataset splitting.

Influence plot

The **Influence plot** describes the characteristic properties of the spectra and helps identify outliers.



Handling the chart

The display of the chart can be adjusted and individual or multiple points can be selected *(see "Handling the charts", chapter 11.3, page 168)*.

Each point represents a spectrum. High values for Hotellings T^2 and Q residuals indicate possible outliers.

Spectra with high values for Hotellings T^2 indicate an extreme composition of the relevant samples.

Spectra with high Q residuals indicate unusual chemical components in the relevant samples.

The dashed lines show the critical values (limit values) for the specified significance level. If no outlier determination was carried out during the automatic dataset splitting, then the significance level is 5%.

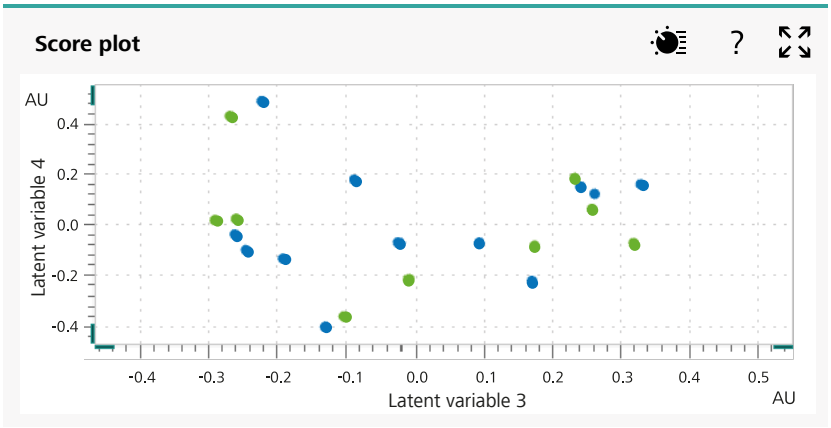
The above figure does not show any potential outliers. All points are within the dashed lines.

Score plot

While the Hotellings T^2 value of a spectrum combines the scores of all principal components in a single value, the score plot enables an even more detailed analysis of the scores.

Each point in the score plot represents a spectrum. The scores for the first two principal components can be read on the x-axis and the y-axis. The scores are normalized, each principal component is assigned the same weight.

Every other pair of principal components can also be displayed under **Properties**.



Splitting the dataset manually (optional)

Prerequisite:

- In the **Calibration and evaluation** work area, the model is open and at the front.
- The navigator is in the **Select samples** process step.

If automatic dataset splitting is carried out before manual splitting, then the **Influence plot** and the **Score plot** will be available.

1 Reassigning the spectra

- Select the spectra in one of the areas.
  Example of selection in the influence plot:
  - Open the **Influence plot** area.
  - In the influence plot, select one or more points *(see "Selecting multiple points or curves", page 170)*.
  - Right-click on one of the selected items to open the context menu. Assign the spectra to a dataset:
    Calibration dataset
    Validation dataset
    Outlier dataset

2 Saving the model

- Click on or press the **[CTRL]**+**[S]** keys.

6.3 Calculating the identification model

A first model may be calculated without parameterization. This provides a comparative benchmark for the validation results. The influence of a subsequent parameterization can be better assessed.

If noise or other artifacts make some wavelengths unusable, then these wavelengths can be excluded from the start *(see "Parameterizing the identification model", chapter 6.5, page 113)*.

Calculating the model

Prerequisite:

- In the **Calibration and evaluation** work area, the identification model is open and at the front.

1 Starting the calculation

- Calculate the model by clicking on **[Calculate]**.

If the **[Calculate]** button is disabled, then the following causes may apply:

- The model has already been calculated and no changes have been made since then.
- One of the process steps contains an incorrect entry. The process step of the affected area is displayed in **red** in the navigator. The field with the incorrect entry is outlined in red.

6.4 Validating the identification model

The process step **Validate identification model** enables validation with the following samples:

- **Samples in the calibration dataset**
  These samples were used to create the model. Correct classification by the model is therefore easier than with other samples.
- **Samples in the validation dataset (if available)**
  These samples are independent of the model. Their validation results are a better benchmark for the identification of unknown samples.

The term **product** is used below, even though it could just as easily be a **product group**. Product groups combine several products and are used for model hierarchies *(see "Model hierarchy", chapter 8, page 136)*.

Identification of a sample

The model assigns a probability to the sample for each product.

The probabilities are independent of one another. The values do not add up to 100%.

The values are to be considered relative to each other, which makes it possible to compare the different products.

The evaluation is carried out with the help of an adjustable **probability threshold** and with qualifications for individual products:

1. For each product whose probability is above the probability threshold, a qualification of the sample is performed using the corresponding qualification model. If the qualification fails, then the probability for the corresponding product is set to zero.
2. Evaluation with the modified probabilities from step 1:
   a. If no probability is above the probability threshold, then the identification fails (identification status **Unidentified**).
   b. If a single probability is above the probability threshold, then the sample is successfully identified and assigned to the corresponding product (identification status **Identified**).
   c. If multiple probabilities are above the probability threshold, then the prediction is ambiguous and the identification has failed (identification status **Ambiguous**).

Validation result of a sample

The OMNIS Software compares the product determined by the model with the expected product. This produces the validation result:

- **Successful**: The identification is successful and matches the expected product.
- **Failed**: No match, no identification or ambiguous identification.

Validation overview area

The **Validation overview** area summarizes the results for the samples of the calibration dataset and the validation dataset (if available).

On the left is an overview of all calibration samples and validation samples:

Total	
Successful %	Correctly classified samples in %
Successful	Number of correctly classified samples
Failed	Number of incorrectly classified samples
Number of spectra	Number of spectra in the calibration dataset and in the validation dataset

On the right is an overview of the individual products and product groups:

Product/product group	Failed	Successful	Successful %
Product A	Number of product A samples that are not classified as product A	Number of correctly classified product A samples	Correctly classified product A samples in %
Product B	Number of product B samples that are not classified as product B	Number of correctly classified product B samples	Correctly classified product B samples in %
Product group C	Number of product group C samples that are not classified as product group C	Number of correctly classified product group C samples	Correctly classified product group C samples in %

Validation results area

The **Validation results** area shows the detailed results of the individual samples. The samples of all products selected in the **Validation overview** area are displayed.

For each sample, the products whose original probabilities are above the probability threshold are displayed. If a 0.0% probability is displayed, then the qualification for the corresponding product failed.

Handling and copying data

- Handling the tables *(see "Handling the tables", chapter 11.2, page 167)*

Optimizing the identification model

The following measures can help improve the identification model.

1 Adjusting the probability threshold

- If many predictions are ambiguous or many 0.0 % probabilities occur, then the probability threshold can be raised.
- If many samples are not identified because the probability threshold was not reached, then the probability threshold can be decreased.

- Execute the following steps to adjust the Probability threshold:
  - Open the properties of the identification model by clicking on  .
  - Select **Parameters** in the selection list.
  - Adjust the **Probability threshold**. The default value is 80%.
  - Recalculate and validate the identification model.

2 Adjusting the parameterization

- Adjust the data preprocessing *(see "Data preprocessing", chapter 6.5.2, page 116)*.
- Adjust the wavelength ranges *(see "Wavelength selection", chapter 6.5.1, page 115)*.

3 Developing the Model hierarchy

A **Model hierarchy** enables the hierarchical structuring of identification models and the quantitative analysis of identified samples *(see "Model hierarchy", chapter 8, page 136)*.

6.5 Parameterizing the identification model

The **Parameterize identification model** process step enables the optimization of spectra. Artifacts and nonlinearities are corrected. If carried out correctly, parameterization improves the accuracy and robustness of the model.

The parameterization is applied to:

- all spectra in the calibration dataset
- all spectra in the validation dataset and in the outlier dataset

During prediction in the **Samples** work area, the spectrum of a sample is acquired and analyzed with a model. The parameterization defined in the model is also applied to this spectrum.

2 parameterization options are available:

- Defining the wavelength ranges to be used.
- Apply data preprocessing to bring the spectra into a more suitable form.

The visual investigation of the spectra begins in the **Select samples** process step.

Depicting the spectra

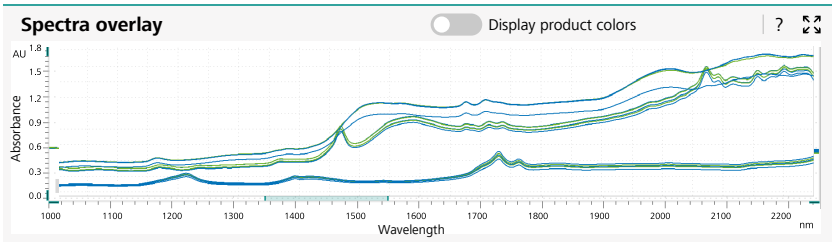
Prerequisite:

- In the **Calibration and evaluation** work area, the model is open and at the front.

1 Selection of the spectra to be investigated

- In the **Select samples** process step, select all products in the **Product list** area whose spectra are to be displayed.

The **Spectra overlay** area shows the spectra of the selected products.



The picture shows spectra of 3 different products. Spectra of different products can be visually easy or difficult to distinguish.

The spectra are depicted as follows:

- Spectra in the calibration dataset are marked **blue**, spectra in the validation dataset are marked **green**, and spectra in the outlier dataset are marked **red**.
- If the Display product colors toggle switch is activated, then the spectra will be colored according to the product colors.

2 Investigating the spectra

- Handling the tables *(see "Handling the tables", chapter 11.2, page 167)*
- Handling the charts *(see "Handling the charts", chapter 11.3, page 168)*

Additional steps

- Wavelength selection *(see "Wavelength selection", chapter 6.5.1, page 115)*
- Defining data preprocessing *(see "Data preprocessing", chapter 6.5.2, page 116)*

6.5.1 Wavelength selection

A wavelength selection can possibly improve the identification model. Example: If noise is visible at high absorbance values, then the relevant wavelength ranges can be excluded.

The model uses the defined wavelength ranges. If no wavelength ranges are defined, then the model will use all wavelengths.

Defining wavelength ranges

Prerequisite:

- In the **Calibration and evaluation** work area, the model is open and at the front.

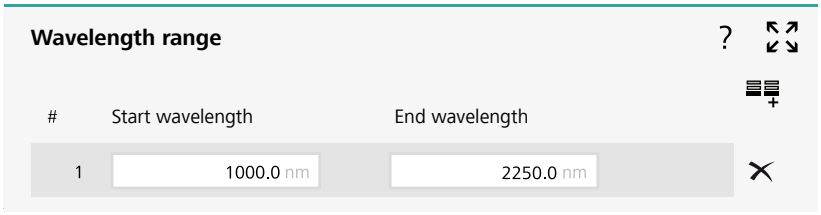
1 Selection of the spectra to be displayed

Select the products whose spectra are to be displayed:

- In the **Select samples** process step, select the products in the **Product list** area.
  or
- In the **Parameterize identification model** process step, select the products in the product list.

2 Adding the wavelength range

- Switch to the **Parameterize identification model** process step in the navigator.
- In the **Wavelength range** area, add a wavelength range by clicking on  .



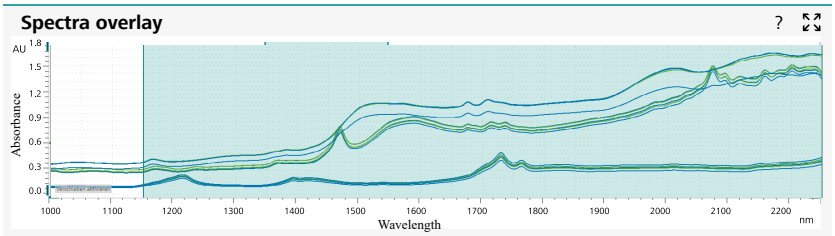
A wavelength range is added. The area initially covers all wavelengths.

3 Defining the wavelength range

Define the wavelength range in one of these ways:

- To define the wavelength range by entering numbers, enter the **Start wavelength** and the **End wavelength** in the corresponding input fields.

- To define the wavelength range in the chart, proceed as follows:
  - Click on **[Activate moving]** in the **Spectra overlay** area.
  - Move the cursor to the left edge of the highlighted area until the cursor is displayed as ↔.
  - Hold down the left mouse button and move the left edge to the corresponding position.
  - Proceed in the same way on the right side of the highlighted area.
  - To move a wavelength range, move the cursor over the area until the cursor is displayed as ⇔. Hold down the left mouse button and move the area to the left or right.
  - Click on **[Deactivate moving]**.



A wavelength range from 1150 to 2250 nm is defined in the picture. This area is used by the model.

4 Adding additional wavelength ranges

Additional wavelength ranges may be added by clicking on ⊞.

Wavelength ranges may not overlap
First, a new wavelength range overlaps with the existing wavelength ranges. Adjust the wavelength range so that no overlaps occur.

5 Saving the model

- Click on 🖫 or press the **[CTRL]**+**[S]** keys.

If the newly created wavelength selection is to be taken into account when splitting the dataset or detecting outliers, then the dataset can be split once again.

6.5.2 Data preprocessing

Suitable data preprocessing can improve the identification model. Example: Baseline shifts do not contain any relevant information for most applications and can be removed.

Defining data preprocessing

Prerequisite:

- In the **Calibration and evaluation** work area, the model is open and at the front.

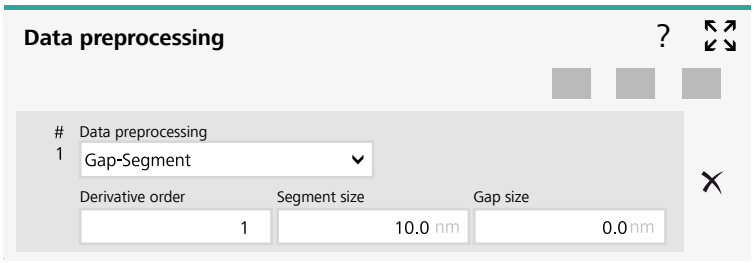
1 Selection of the spectra to be displayed

Select the products whose spectra are to be displayed:

- In the **Select samples** process step, select the products in the **Product list** area.
  or
- In the **Parameterize identification model** process step, select the products in the product list.

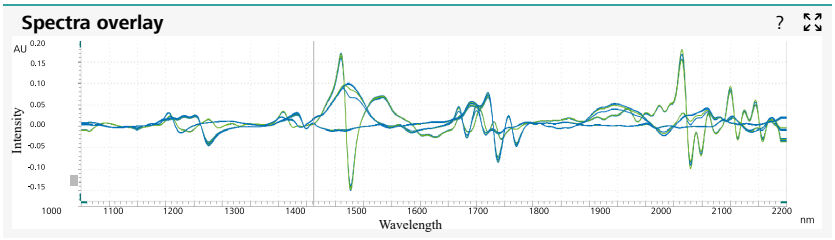
2 Adding the data preprocessing step

- Switch to the **Parameterize identification model** process step in the navigator.
- In the **Data preprocessing** area, add a data preprocessing step by clicking on .
- In the **Data preprocessing** field, select the type of data preprocessing and fill in the associated fields.
  Example of gap-segment with a first derivative that removes constant (non-wavelength-dependent) baseline shifts:



The preprocessed spectra of the products selected in step 1 are shown immediately in the **Spectra overlay** area.

After the data preprocessing, the spectra look different, e.g.



3 Adding additional data preprocessing steps

Additional data preprocessing steps may be added by clicking on .

If multiple data preprocessing steps are used, then the sequence can be decisive. Preferably, gap-segment or Savitzky-Golay is applied prior to SNV, and SNV before Detrend.

Lines can be moved up or down by clicking on and , thus defining the sequence.

4 Saving the model

- Click on or press the **[CTRL]**+**[S]** keys.

If the newly created data preprocessings are to be taken into account when splitting the dataset or detecting outliers, then the dataset can be split once again.

6.6 Publishing the identification model

The model must first be published before it can be used for determinations. This means that the model can still be developed further without impacting the published version and the determinations carried out with it.

Publish identification model

Prerequisite:

- The model is calculated and saved.
- The model is open.

1 Opening a dialog

- Open the **Publish identification model** dialog by clicking on .

If the model has been previously published and used in methods, then these methods can be automatically updated by enabling the **Update methods** check box.

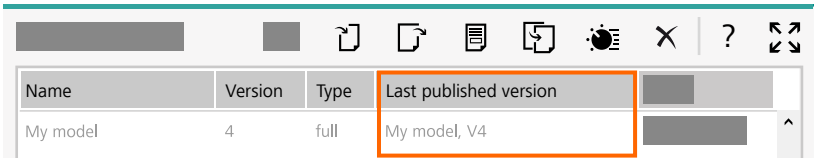
Note: Not updated automatically:

- Opened methods
- Signed and published methods
- If the filtering of the data permissions is enabled: Methods without data permissions of the currently logged-in user

2 Publishing

- Publish the model by clicking on **[Publish]**.

The last published version is displayed under **Calibration and evaluation ▶ Identification models**:



The **PREDICT** command can now access the published version of the model.

7 Qualification model

An illustration of the runs in the OMNIS Software can be found in the Appendix *(see "Developing a model", page 184)*.

A qualification model makes it possible to distinguish one group of samples from other samples. The qualification model is suitable, e.g., for distinguishing usable samples (positive samples) from unusable samples (negative samples).

7.1 Creating a qualification model

Creating a qualification model

Prerequisite:

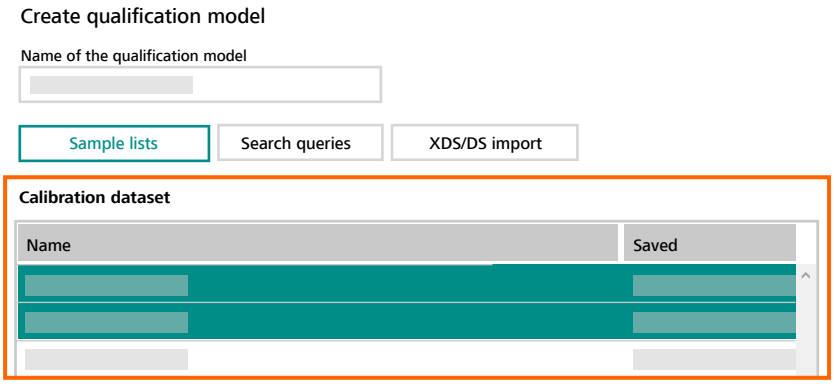
- A dataset with spectra is created *(see "Acquiring spectra", chapter 4.2, page 59)*.

1 Creating and naming a qualification model

- Under **Calibration and evaluation** ▶ **Qualification models**, click on .
  A new qualification model appears in a new tab.
- Enter a matching name in the **Name of the qualification model** input field.

2 Selecting calibration samples

- Display all sample lists by clicking on **Sample lists**.
- Select the prepared sample lists for the calibration dataset.

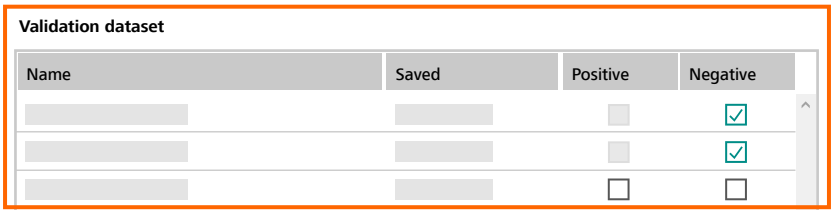


Samples can also be selected from a search query. Samples from XDS devices and DS devices can also be imported *(see "Switching from XDS/DS Analyzers (quantification)", chapter 11.6, page 178)*.

The sample selection can be adjusted later.

3 Selecting validation samples (optional)

- Click on **Add validation datasets**.
- Assign the prepared sample lists for the validation datasets to the positive or negative validation dataset using the corresponding check boxes.



4 Creating a qualification model

- Click on **[Create]**.
- Save the model: Click on or press the **[CTRL]**+**[S]** keys.

7.2 Selecting the samples and splitting the dataset

The tab of the qualification model shows a horizontal navigation bar at the top, the **navigator**. The navigator guides you through the next steps of model development.

Depiction of spectra

In the 3 process steps, the individual spectra are displayed in the form of curves, points or table cells.

Selected spectra are highlighted simultaneously in all depictions and in all process steps.

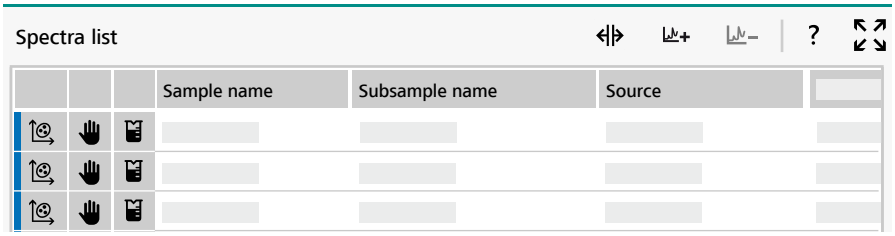
Tables and charts

The handling of tables and charts is described in the appendix:

- Handling the tables *(see "Handling the tables", chapter 11.2, page 167)*
- Handling the charts *(see "Handling the charts", chapter 11.3, page 168)*

'Select samples' process step

The **Calibration dataset** area lists the spectra in the calibration dataset:



If samples have been selected for the validation datasets, then their spectra will appear in the **Validation dataset** area.

The following icons indicate the assignment to the datasets:

	The spectrum is assigned to the calibration dataset.
	The spectrum is assigned to the positive validation dataset.
	The spectrum is assigned to the negative validation dataset.
	The spectrum was manually assigned to the dataset.
	The spectrum was automatically assigned to the dataset.
	The spectrum was acquired in the OMNIS Software.
	The spectrum has been imported from an external file.

In the **Spectra overlay** area, the spectra in the calibration dataset are marked **blue**, the spectra in the positive validation dataset are marked **green**, and the spectra in the negative validation dataset are marked **red**.

The **Select samples** process step enables the following:

- **Adjusting the sample selection**
  Add additional spectra or delete spectra.

- **Splitting the dataset**
  Automatic or manual splitting of the dataset:
  - **Calibration dataset**: The model is calculated using the spectra of the calibration dataset.
  - **Validation dataset**: The spectra of the validation datasets are used exclusively to validate the model.

A model can be developed without a validation dataset, e.g., if only a limited number of samples are available in an initial phase or if the validation is carried out exclusively with an external dataset.

Adjusting the sample selection

Prerequisite:

- In the **Calibration and evaluation** work area, the model is open and at the front *(see "Creating a qualification model", chapter 7.1, page 120)*.
- The navigator is in the **Select samples** process step.

1 Adding or deleting spectra

- Add spectra: In the **Calibration dataset** area or in the **Validation dataset** area, click on +.
- Remove spectra: Select the spectra and click on –.
  Note: The associated samples and their spectra are retained in the database.

2 Saving the model

- Click on or press the **[CTRL]**+**[S]** keys.

Splitting the dataset automatically

The splitting includes all spectra of the calibration dataset and of both validation datasets. The splitting offers the following options:

- Automatic creation of a negative validation dataset (optional)
  The spectra for the negative validation dataset are determined by the outlier detection (spectral outliers).
- Automatic creation of a positive validation dataset (optional)
  The remaining spectra can be automatically split into the calibration dataset and the positive validation dataset.

The samples can also be assigned manually at any time.

Prerequisite:

- In the **Calibration and evaluation** work area, the model is open and at the front.
- The navigator is in the **Select samples** process step.

1 Calling up the dataset splitting

- Click on  in the **Calibration dataset** area.

The **Dataset splitting** dialog opens.

2 Determining the negative validation dataset (optional)

- To automatically assign spectral outliers to the negative validation dataset, activate the **Determine negative spectra** toggle switch.
  - Adjust the **Significance level** if required. The higher the significance level, the more spectral outliers will be detected. Typical values are 5% or 1%.

The determination of negative spectra should be used with caution. A high significance level can improve the reliability of positive results. However, more positive samples can also be overlooked and falsely classified as negative.

The spectra in the negative validation dataset determined must be examined to determine whether they are actually outliers. The influence plot and the score plot are helpful for this.

3 Determining the positive validation dataset (optional)

The automatic splitting is done in a way which ensures that the calibration dataset and the positive validation dataset are representative of the population and independent of one another.

- To automatically assign spectra to the positive validation dataset, activate the **Determine positive spectra** toggle switch.
  - In the **Percentage** field, define the percentage of spectra for the positive validation dataset, e.g. between 20% and 30%.

4 Defining the options

Define options for dataset splitting:

- **Apply parameterization**: Apply data preprocessing and wavelength selection to the spectra *(see "Parameterizing the qualification model", chapter 7.5, page 130)*.
  Note: Subsequent changes to the parameterization will not affect the dataset assignment. Unless the dataset is to be split again.

- **Keep negative spectra**: Retain the existing spectra in the negative validation dataset and do not take it into account in the split. This option can lead to an increase in the size of the negative validation dataset, even if the **Significance level** is not changed.
- **Keep positive spectra**: Retain the existing spectra in the positive validation dataset and do not take it into account in the split. This option leads to an increase in the size of the positive validation dataset, even if the **Percentage** is not changed.

If separate spectra have been added for the validation datasets, then the options **Keep negative spectra** and **Keep positive spectra** should be activated. Otherwise, all spectra will be merged and split again, which can lead to undesirable results.

5 Starting the automatic splitting

- Click on **[Split]**.

The split of the dataset corresponds to the settings made.

6 Saving the model

- Click on or press the **[CTRL]**+**[S]** keys.

Influence plot and score plot

After automatic dataset splitting, the influence plot and score plot charts are available:

- In the **Select samples** process step, click on in one of the areas and select the **Influence plot** or the **Score plot** chart.

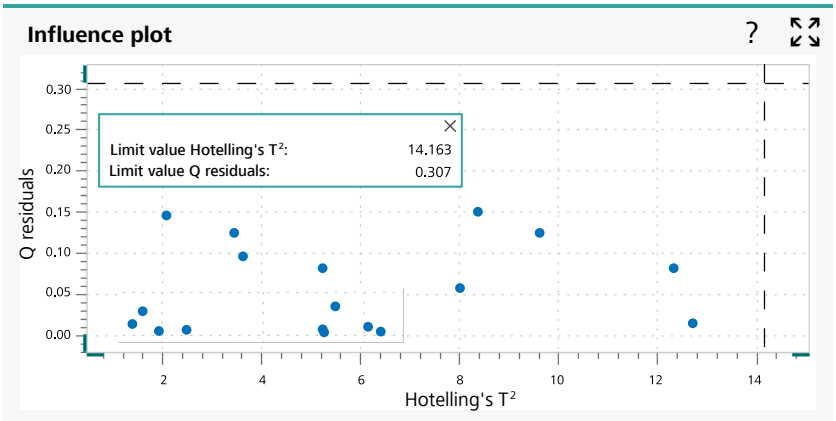
The influence plot and the score plot are based on the **PCA** calculation method (principal component analysis). The number of principal components is chosen in such a way that the explained variance is at least 95%.

The spectra in the following form serve as the starting point for the PCA:

- Spectra without data preprocessing and wavelength selection if the **Apply parameterization** option has been deactivated for automatic dataset splitting.
- Spectra with data preprocessing and wavelength selection if the **Apply parameterization** option has been activated for automatic dataset splitting.
  Note for when this option is activated: If the parameterization is changed, then the influence plot and the score plot will not be available until after a new automatic dataset splitting.

Influence plot

The **Influence plot** describes the characteristic properties of the spectra and helps identify spectral outliers for the negative validation dataset.



Handling the chart

The display of the chart can be adjusted and individual or multiple points can be selected *(see "Handling the charts", chapter 11.3, page 168)*.

Each point represents a spectrum. High values for Hotellings T^2 and Q residuals indicate possible outliers.

Spectra with high values for Hotellings T^2 indicate an extreme composition of the relevant samples.

Spectra with high Q residuals indicate unusual chemical components in the relevant samples.

The dashed lines show the critical values (limit values) for the specified significance level. If no negative spectra were determined during the automatic dataset splitting, then the significance level is 5%.

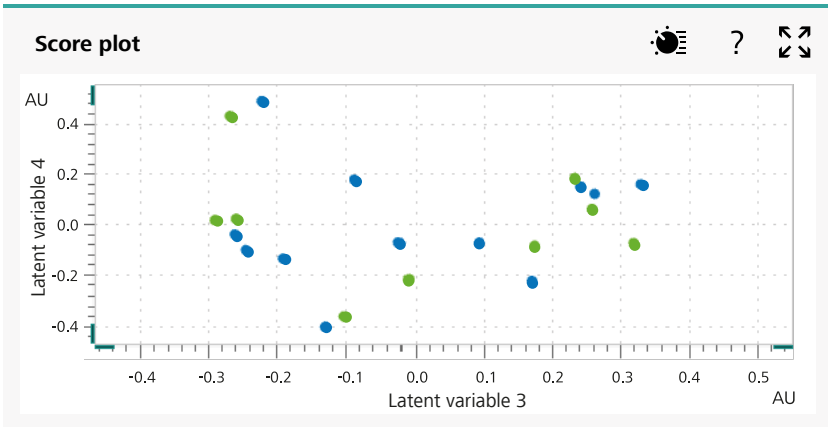
The above figure does not show any potential negative spectra. All points are within the dashed lines.

Score plot

While the Hotellings T^2 value of a spectrum combines the scores of all principal components in a single value, the score plot enables an even more detailed analysis of the scores.

Each point in the score plot represents a spectrum. The scores for the first two principal components can be read on the x-axis and the y-axis. The scores are normalized, each principal component is assigned the same weight.

Every other pair of principal components can also be displayed under **Properties**.



Splitting the dataset manually (optional)

Prerequisite:

- In the **Calibration and evaluation** work area, the model is open and at the front.
- The navigator is in the **Select samples** process step.

If automatic dataset splitting is carried out before manual splitting, then the **Influence plot** and the **Score plot** will be available.

1 Reassigning the spectra

- Select the spectra in one of the areas.
  Example of selection in the influence plot:
  - Open the **Influence plot** area.
  - In the influence plot, select one or more points *(see "Selecting multiple points or curves", page 170)*.
  - Right-click on one of the selected items to open the context menu. Assign the spectra to a dataset:
    Positive validation dataset
    Negative validation dataset
    Calibration dataset

2 Saving the model

- Click on or press the **[CTRL]**+**[S]** keys.

7.3 Calculating a qualification model

A first model may be calculated without parameterization. This provides a comparative benchmark for the validation results. The influence of a subsequent parameterization can be better assessed.

If noise or other artifacts make some wavelengths unusable, then these wavelengths can be excluded from the start *(see "Parameterizing the qualification model", chapter 7.5, page 130)*.

Calculating the model

Prerequisite:

- In the **Calibration and evaluation** work area, the qualification model is open and at the front.

1 Starting the calculation

- Calculate the model by clicking on **[Calculate]**.

If the **[Calculate]** button is disabled, then the following causes may apply:

- The model has already been calculated and no changes have been made since then.
- One of the process steps contains an incorrect entry. The process step of the affected area is displayed in **red** in the navigator. The field with the incorrect entry is outlined in red.

7.4 Validating a qualification model

The **Validate qualification model** process step enables validation with the following samples:

- **Samples in the calibration dataset**
  These samples were used to create the model. Correct classification by the model is therefore easier than with other samples.
- **Samples in the positive and in the negative validation dataset (if available)**
  These samples are independent of the model. Their validation results are a better benchmark for the qualification of unknown samples.

Validation result of a sample

The qualification model determines a result (positive or negative) for each sample. A positive result is expected for the samples in the calibration

dataset and in the positive validation dataset. A negative result is expected for the samples in the negative validation dataset. The OMNIS Software compares the result determined by the model with the expected result. This produces the validation result:

- **Successful** The result determined by the model matches the expected result.
- **Failed** The result determined by the model does not match the expected result.

Validation overview area

The **Validation overview** area summarizes the results for the samples of the calibration dataset and the validation datasets (if available).

On the left is an overview of all calibration samples and validation samples:

Total	
Successful %	Correctly predicted samples in %
Successful	Number of correctly predicted samples
Failed	Number of incorrectly predicted samples
Number of spectra	Number of spectra in the calibration dataset and in both validation datasets

On the right-hand side is an overview of the individual datasets.

Validation results area

The **Validation results** area shows the detailed results of the individual samples. The samples of all datasets selected in the **Validation overview** area are displayed.

Optimizing a qualification model

The following measures can help improve the qualification model:

- Adjust the data preprocessing *(see "Data preprocessing", chapter 7.5.2, page 132)*.
- Adjust the wavelength ranges *(see "Wavelength selection", chapter 7.5.1, page 131)*.

If the dataset is split again, then the qualification model can be adapted to the requirements by determining negative spectra:

- A higher significance level can increase the reliability of positive results. This means: Fewer false positive results, but more positive samples are overlooked.

- A lower significance level (or omitting the determination of negative spectra) can mean that fewer positive samples are overlooked. This means: Fewer false negative results, but more negative samples will be falsely classified as positive.

7.5 Parameterizing the qualification model

The **Parameterize qualification model** process step enables the optimization of spectra. Artifacts and nonlinearities are corrected. If carried out correctly, parameterization improves the accuracy and robustness of the model.

The parameterization is applied to:

- all spectra in the calibration dataset
- all spectra in both validation datasets

During prediction in the **Samples** work area, the spectrum of a sample is acquired and analyzed with a model. The parameterization defined in the model is also applied to this spectrum.

2 parameterization options are available:

- Defining the wavelength ranges to be used.
- Apply data preprocessing to bring the spectra into a more suitable form.

The visual investigation of the spectra begins in the **Select samples** process step.

Depicting the spectra

Prerequisite:

- In the **Calibration and evaluation** work area, the model is open and at the front.

1 'Select samples' process step

- Click on the **Select samples** process step in the navigator.

In this process step, the spectra can be investigated simultaneously in tabular form and in curve form. If automatic dataset splitting was carried out, then the influence plot and the score plot are also available.

2 Investigating the spectra

- Handling the tables *(see "Handling the tables", chapter 11.2, page 167)*

- Handling the charts *(see "Handling the charts", chapter 11.3, page 168)*

Additional steps

- Wavelength selection *(see "Wavelength selection", chapter 7.5.1, page 131)*
- Defining data preprocessing *(see "Data preprocessing", chapter 7.5.2, page 132)*

7.5.1 Wavelength selection

A wavelength selection can improve the qualification model. Example: If noise is visible at high absorbance values, then the relevant wavelength ranges can be excluded.

The model uses the defined wavelength ranges. If no wavelength ranges are defined, then the model will use all wavelengths.

Defining wavelength ranges

Prerequisite:

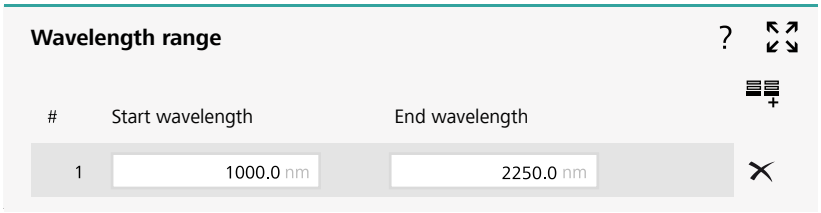
- In the **Calibration and evaluation** work area, the model is open and at the front.

1 'Parameterize qualification model' process step

- Click on **Parameterize qualification model** in the navigator.

2 Adding the wavelength range

- In the **Wavelength range** area, add a wavelength range by clicking on  .



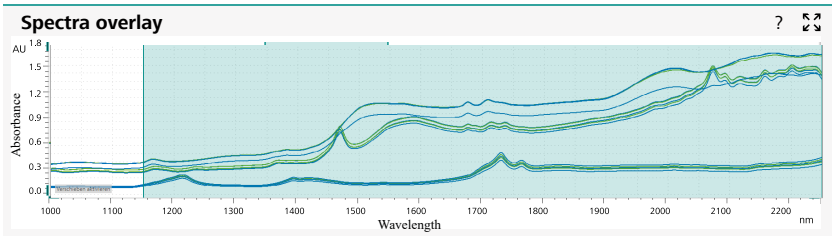
A wavelength range is added. The area initially covers all wavelengths.

3 Defining the wavelength range

Define the wavelength range in one of these ways:

- To define the wavelength range by entering numbers, enter the **Start wavelength** and the **End wavelength** in the corresponding input fields.

- To define the wavelength range in the chart, proceed as follows:
  - Click on **[Activate moving]** in the **Spectra overlay** area.
  - Move the cursor to the left edge of the highlighted area until the cursor is displayed as ↔.
  - Hold down the left mouse button and move the left edge to the corresponding position.
  - Proceed in the same way on the right side of the highlighted area.
  - To move a wavelength range, move the cursor over the area until the cursor is displayed as ⇔. Hold down the left mouse button and move the area to the left or right.
  - Click on **[Deactivate moving]**.



A wavelength range from 1150 to 2250 nm is defined in the picture. This area is used by the model.

4 Adding additional wavelength ranges

Additional wavelength ranges may be added by clicking on ⊞.

Wavelength ranges may not overlap
First, a new wavelength range overlaps with the existing wavelength ranges. Adjust the wavelength range so that no overlaps occur.

5 Saving the model

- Click on 🖫 or press the **[CTRL]**+**[S]** keys.

If the newly created wavelength selection is to be taken into account when splitting the dataset, then the dataset can be split once again.

7.5.2 Data preprocessing

Suitable data preprocessing can improve the qualification model. Example: Baseline shifts do not contain any relevant information for most applications and can be removed.

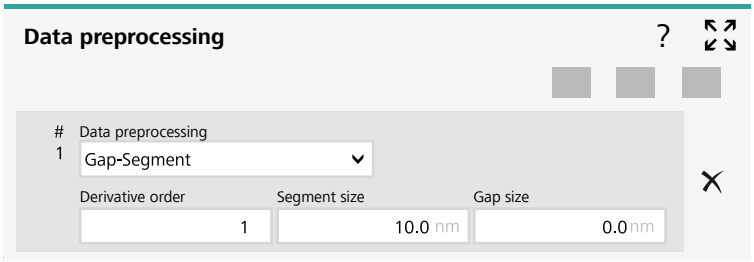
Defining data preprocessing

Prerequisite:

- In the **Calibration and evaluation** work area, the model is open and at the front.
- The navigator is in the **Parameterize qualification model** process step.

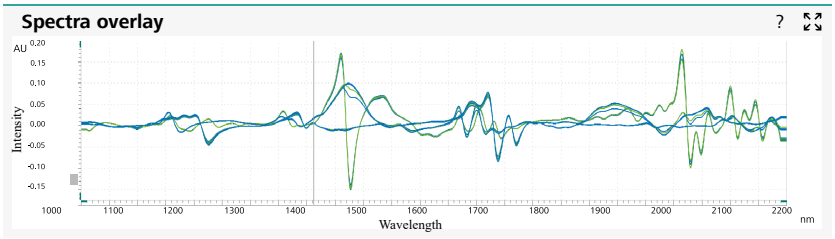
1 Adding the data preprocessing step

- In the **Data preprocessing** area, add a data preprocessing step by clicking on .
- In the **Data preprocessing** field, select the type of data preprocessing and fill in the associated fields.
  Example of gap-segment with a first derivative that removes constant (non-wavelength-dependent) baseline shifts:



The preprocessed spectra of the products selected in step 1 are shown immediately in the **Spectra overlay** area.

After the data preprocessing, the spectra look different, e.g.



2 Adding additional data preprocessing steps

Additional data preprocessing steps may be added by clicking on .

If multiple data preprocessing steps are used, then the sequence can be decisive. Preferably, gap-segment or Savitzky-Golay is applied prior to SNV, and SNV before Detrend.

Lines can be moved up or down by clicking on and , thus defining the sequence.

3 Saving the model

- Click on or press the **[CTRL]**+**[S]** keys.

If the newly created data preprocessings are to be taken into account when splitting the dataset, then the dataset can be split once again.

7.6 Publishing a qualification model

The model must first be published before it can be used for determinations. This means that the model can still be developed further without impacting the published version and the determinations carried out with it.

Publish qualification model

Prerequisite:

- The model is calculated and saved.
- The model is open.

1 Opening a dialog

- Open the **Publish qualification model** dialog by clicking on .

If the model has been previously published and used in methods, then these methods can be automatically updated by enabling the **Update methods** check box.

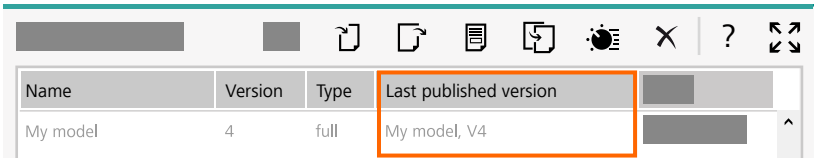
Note: Not updated automatically:

- Opened methods
- Signed and published methods
- If the filtering of the data permissions is enabled: Methods without data permissions of the currently logged-in user

2 Publishing

- Publish the model by clicking on **[Publish]**.

The last published version is displayed under **Calibration and evaluation ▶ Qualification models**:



The **PREDICT** command can now access the published version of the model.

8 Model hierarchy

A model hierarchy enables the following:

- **Structure identification models hierarchically**
  If an identification model has difficulty distinguishing between similar products, then this distinction can be outsourced to submodels optimized for this purpose.
  Example: An identification model with 4 different products can hardly distinguish between the similar products of fructose and glucose. If fructose and glucose are combined into one product group, "sugar", then the main model is able to differentiate between sugar and the other two products. If a sample is identified as sugar, then a submodel takes over the classification between fructose and glucose.
  If necessary, additional hierarchy levels may be added.
- **Link quantification models with products**
  Identified samples can be analyzed quantitatively. A quantification model is linked to the corresponding product for each parameter of interest. A slope/y-intercept correction can be applied if required.
- **Structure quantification models hierarchically**
  In some cases, a hierarchical structure of quantification models can achieve a better predictive ability than a single quantification model. In this process, subordinate quantification models are optimized in each case for a part of the reference value range of the superordinate model.
  Example: If the superordinate quantification model yields a result of < 5, then a subordinate quantification model for values of < 5 will be used to determine the final result. Analogous for results of ≥ 5.
- **Model hierarchy from quantification models**
  In a model hierarchy, one or more quantification models can be listed below one another, with or without subordinate quantification models. In the method, a single **PREDICT** command is then sufficient to predict all parameters of interest.

A model hierarchy provides the following, depending on the use:

- An **identification** of an unknown sample (e.g., fructose). The result is a product name.
  - Optionally, one or more **quantifications**, depending on the product determined.
- A **verification** of the product membership (e.g., fructose) of a sample. The result is yes or no – Verification successful or failed.
  - Optionally, one or more **quantifications**, depending on the product determined and regardless of the verification result.
- One or more **quantifications**.

8.1 Developing the model hierarchy

8.1.1 Developing models

First, the identification models and/or quantification models to be used in the model hierarchy must be developed.

Developing identification models

If the model hierarchy is to contain identification models, develop them as follows.

1 Main model

- Develop an identification model for all existing products *(see "Identification model", chapter 6, page 102)*.
- If individual products are difficult to distinguish from one another, then these products can be combined in a single product group:
  - In the **Select samples** process step, define a common name in the **Product group** column in the **Product list** area for the products to be combined. In the following example, products **C1** and **C2** together form product group **C**:

Product list

Product	Number of spectra	Product group
A		
B		
C1		C
C2		C

The model then treats product group **C** as a single product. The model no longer classifies between A/B/C1/C2, but only between A/B/C.

Additional product groups can be created if required.

2 Submodels

Develop an identification model for each product group of the main model *(see "Identification model", chapter 6, page 102)*.

In the example above, a submodel is developed with all samples associated with the products **C1** and **C2**. The same samples should be used for this as in the main model for product group **C**.

Right-click on the **Product list** to create submodels for the defined product groups.

3 Additional hierarchy levels

If a submodel contains more than 2 products, then multiple products can be combined into a product group if required. A separate identification model is again developed for this product group. Additional hierarchy levels arise in this way.

Developing quantification models

If the model hierarchy is to contain quantification models, then develop them as follows.

1 Quantification models for each parameter of interest

Develop the corresponding quantification models for all quantitative parameters of interest *(see "Quantification model", chapter 5, page 65)*.

2 Subordinate quantification models

If subordinate quantification models are required for a quantification model, then develop the subordinate quantification models.

8.1.2 Inserting models in a model hierarchy

Prerequisite: All models to be used in the model hierarchy have been created *(see "Developing models", chapter 8.1.1, page 137)* and published.

The next steps are to create the model hierarchy and insert the models into the model hierarchy:

- Insert the published main model or the published quantification models.
- Link published submodels with product groups.
- Link published quantification models with products.
- Link published subordinate quantification models with superordinate quantification models.

Creating the model hierarchy

1 Generating the model hierarchy

- Under **Calibration and evaluation** ▶ **Model hierarchies**, click on .
  A new model hierarchy appears in a new tab.

2 Naming the model hierarchy

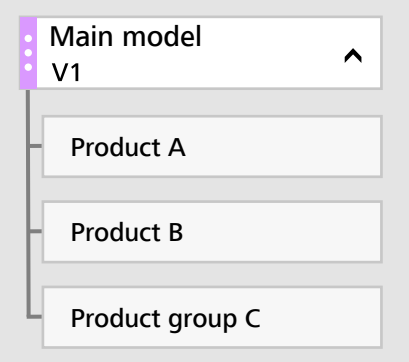
- Open the **Properties** window by clicking on in the top toolbar.
- Enter the desired name in the **Name** field under **Properties** ▶ **General**.

Inserting identification models in a model hierarchy

If the model hierarchy is to contain identification models, insert them as follows.

1 Adding the main model

- The **Edit model hierarchy** process step contains the model hierarchy editor. The model hierarchy editor is still empty at first.
- Click on to open the **Library** window.
- Under **Library** ▶ **Identification models**, use drag and drop to pull the main model to the right and insert it into the model hierarchy editor.



The vertical arrow is used to collapse and expand the products.

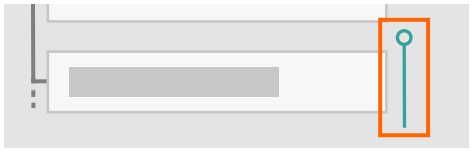
If a model cannot be found in the library:

- Ensure that the model is published.
- In the library, update the view by clicking on .

2 Linking submodels with product groups

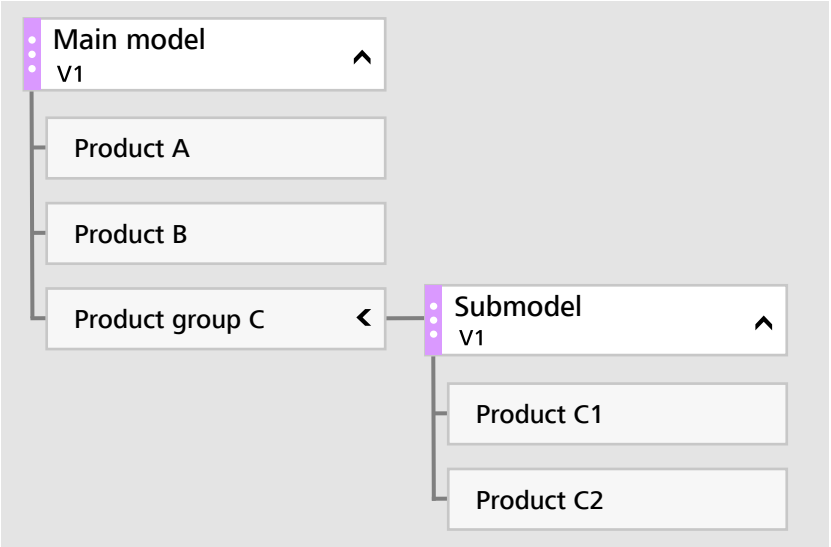
If product groups have been defined in the main model:

- Use drag and drop to insert the submodel next to the associated product group under **Library** ▶ **Identification models**. A vertical green line shows the insertion position:



- **Additional submodels and hierarchy levels**
  Link additional submodels to the respectively associated product group in the same way.

Example: A submodel is linked with a product group.



The horizontal arrow is used to collapse and expand the submodel.

Submodels can be linked to product groups or products.

3 Versioning models

The models contained in the model hierarchy remain unchanged, even if a new version is published for a model. If necessary, remove

the corresponding model from the model hierarchy and insert a newer version.

4 Saving the model hierarchy

- Click on or press the **[CTRL]**+**[S]** keys.

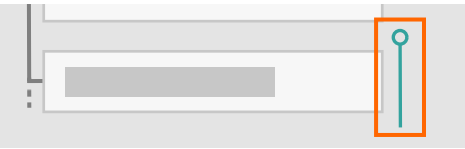
Inserting quantification models in a model hierarchy

If the model hierarchy is to contain quantification models, then insert them as follows.

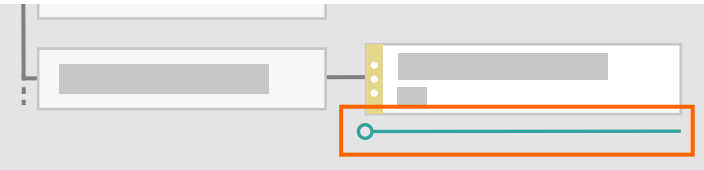
1 Model hierarchy with identification models

If the model hierarchy also contains identification models, then the quantification models must be linked to products:

- Use drag and drop to insert the quantification model next to the associated product under **Library** ▶ **Quantification models**. A vertical green line shows the insertion position:

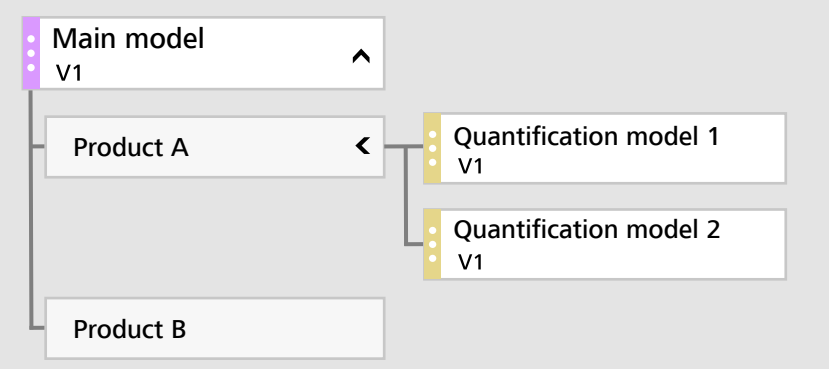


- If multiple quantitative parameters of interest are to be predicted for the same product:
  - Insert the other quantification models one below the other using drag and drop. A horizontal green line shows the insertion position:



- If quantitative analyses are planned for additional products, then link the corresponding quantification models in the same way.

Example: 2 quantification models are linked to a product.

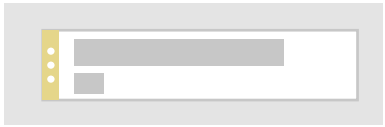


Quantification models can be linked to products or product groups.

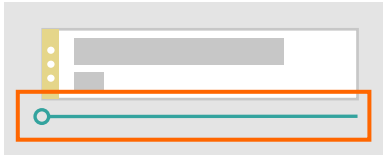
2 Model hierarchy without identification models

If the model hierarchy is to contain only quantification models:

- The **Edit model hierarchy** process step contains the model hierarchy editor. The model hierarchy editor is still empty at first.
- Click on to open the **Library** window.
- Under **Library** ▶ **Quantification models**, use drag and drop to pull the first quantification model to the right and insert it into the model hierarchy editor.



- If the model hierarchy is to predict multiple parameters of interest, then insert the additional quantification models one below the other using drag and drop. A horizontal green line shows the insertion position:



If a model cannot be found in the library:

- Ensure that the model is published.
- In the library, update the view by clicking on .

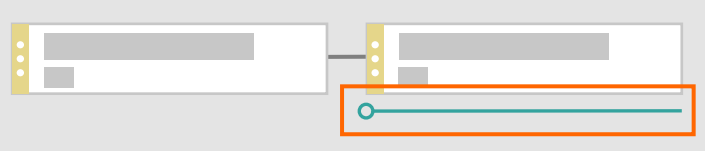
3 Subordinate quantification models

To insert subordinate quantification models into the model hierarchy, proceed as follows:

- Use drag and drop to insert the subordinate quantification model next to the superordinate quantification model under **Library** ▶ **Quantification models**. A vertical green line shows the insertion position:

- In the **Add condition** dialog, specify the condition under which the subordinate quantification model is to be applied. The condition must include part of the predicted values of the superordinate quantification model., e.g., < 5.
  Once the condition is added, the subordinate quantification model is linked to the superordinate quantification model.

- Insert the other subordinate quantification models one below the other using drag and drop. A horizontal green line shows the insertion position:



Notice: If a subordinate quantification model is not required for a number range, then the superordinate model can be used as a substitute and linked to itself.
Prerequisites: The conditions of the subordinate quantification models must meet the following prerequisites:
  - The conditions must cover the entire range of rational numbers.
  - The conditions must not overlap.

  Example with correct conditions:
  - Condition for model A1 (single value): < 5
  - Condition for model A2 (interval): ≥ 5 and < 10
  - Condition for model A3 (single value): ≥ 10

  If the prerequisites are not met, then the model hierarchy can still be saved but not published.
  Check conditions:
  - Click on **Validate internally**.
  - If no error message appears, then the prerequisites are met.
  - Otherwise, the error message will inform you where and why the prerequisites have been violated.

  View or edit conditions:
  - Select a subordinate quantification model.
  - Open the **Properties** window by clicking on .
  - Open the Condition subsection.
  - Select the relevant subordinate quantification models, one after the other, to display the respective condition. Adjust the condition if required.
- If subordinate quantification models are planned for further quantification models, then link these in the same way.

4 Slope/y-intercept corrections

Modify all quantification models that require a slope/y-intercept correction as follows:

- Select the quantification model.
- Open the **Properties** window by clicking on .

- Define the slope/y-intercept correction under **Properties** ▶ **Parameters**.

The  symbol indicates that the quantification model is linked with a slope/y-intercept correction. The corrected result is used when checking conditions for possible subordinate quantification models.

5 Versioning models and slope/y-intercept corrections

The models and slope/y-intercept corrections contained in the model hierarchy remain unchanged, even if a new version is published for the model or for the slope/y-intercept correction. If necessary, remove the corresponding model or slope/y-intercept correction from the model hierarchy and insert a newer version.

6 Saving the model hierarchy

- Click on  or press the **[CTRL]**+**[S]** keys.

8.2 Validating the model hierarchy

Model hierarchies with quantification models

No quantification models are evaluated during the validation of model hierarchies. However, for quantification models with subordinate quantification models, a check is made to determine whether the prerequisites are met:

- The conditions must cover the entire range of rational numbers.
- The conditions must not overlap.

Check conditions

- Click on **Validate internally**.
- If no error message appears, then the prerequisites are met.
- Otherwise, the error message will inform you where and why the prerequisites have not been met.

Internal and external validation

For model hierarchies with identification models, the **Validate model hierarchy** process step offers 2 different validations:

- **Internal validation**
  The internal validation uses the calibration dataset and, if available, the validation dataset of the main model.

- **External validation**
  The external validation uses a separate, external dataset. Samples are collected and measured on a different day for the external dataset, possibly by a different person and with a different instrument.

Validating the model hierarchy

Prerequisite:

- In the **Calibration and evaluation** work area, the model hierarchy is created, open and at the front *(see "Developing the model hierarchy", chapter 8.1, page 137)*.
- If externally validated, then a corresponding dataset with spectra and product names will be available *(see "Acquiring spectra", chapter 4.2, page 59)*.

1 Switching to the 'validation' process step

- In the navigator, click on **Validate model hierarchy** to switch to the validation process step.

2 Performing the internal or external validation

Internal validation

- Click on **Validate internally**.

The internal validation uses only spectra of the main model (calibration dataset and, if available, validation dataset). Outliers and additional spectra in submodels are not included.

External validation

- Click on **Validate externally**.
- Select samples from Sample lists or Search queries.
  The selection must contain samples with a product parameter.
  The **Product** column lists the products included.
- Click on **[Validate]**.

3 Checking the validation results

- In the **Validate model hierarchy** process step, check the **Validation overview** area. The Validation overview summarizes the results in the same way as for a single model *(see "Validation overview area", page 111)*.
  The model hierarchy is viewed as a single, large model. The validation result of a spectrum is either successful or failed.

- Check the results of individual spectra:
  - In the **Validation overview** area, select all of the products whose individual spectra are to be displayed.
  - The **Validation results** area lists the spectra of the selected products.
    The **Model hierarchy result** shows the final result of the model hierarchy in each case.
    The step-by-step evaluation for each hierarchy level is then displayed. The **Result of level 1** shows the result of the main model. This is followed by the results of the corresponding submodels for all other hierarchy levels.

8.3 Publishing the model hierarchy

The model hierarchy must first be published before it can be used for determinations.

Publish model hierarchy

Prerequisite:

- The model hierarchy is saved.
- The validation is optional. Metrohm recommends carrying out a validation.
- The model hierarchy is opened.

1 Opening a dialog

- Open the **Publish model hierarchy** dialog by clicking on  .

If the model hierarchy has previously been published and used in methods, then these methods can be automatically updated by enabling the **Update methods** check box.

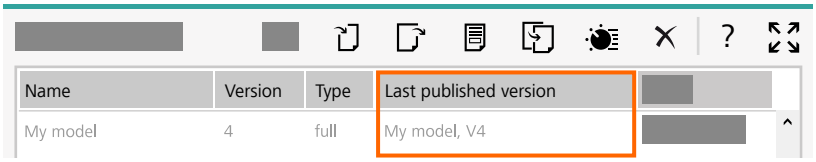
Note: Not updated automatically:

- Opened methods
- Signed and published methods
- If the filtering of the data permissions is enabled: Methods without data permissions of the currently logged-in user

2 Publishing

- Publish the model hierarchy by clicking on **[Publish]**.

The last published version is displayed under **Calibration and evaluation ▶ Model hierarchies**:



The **PREDICT** command can now access the published version of the model hierarchy.

9 Prediction

For the prediction, a model is applied to the spectrum of an unknown sample. Depending on the model, the following can be predicted:

- Parameter of interest (quantification)
- Product membership or verification result (identification)
- Qualification result (qualification)

The samples for prediction must be treated and measured in the same way as the samples used to create the model.

An illustration of the runs in the OMNIS Software can be found in the Appendix *(see "Prediction", page 185)*.

9.1 Preparing the prediction

To prepare the prediction, create a method, an operating procedure, a sample profile, and a sample list as follows. The method will contain a **PREDICT** command, which produces a connection to the model.

Setting up the method

1 Applying and naming the method

The spectra must be acquired with the same settings as the spectra for model development. The simplest way is to apply the method used for the model development *(see "Preparing the spectra acquisition", chapter 4.1, page 49)*.

- Under **Processes** ▶ **Methods**, select the method that was used for the model development.
- Duplicate the selected method by clicking on .
- Open the duplicated method by double-clicking on the method name.
- Open the **Properties** window by clicking on .
- Enter a matching name in the **Name** field under **Properties** ▶ **General**.

2 Inserting the PREDICT command

PREDICT creates a prediction for the acquired spectrum.

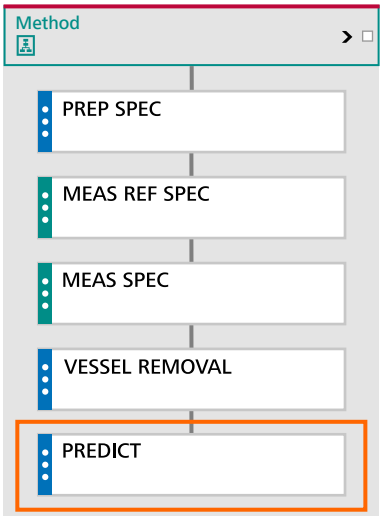
- Open the **Library** window by clicking on .

- Under **Library** ▶ **Commands**, search for the **PREDICT** command and use drag and drop to insert it into the method.

Observe the correct sequence of the commands:

Liquid samples

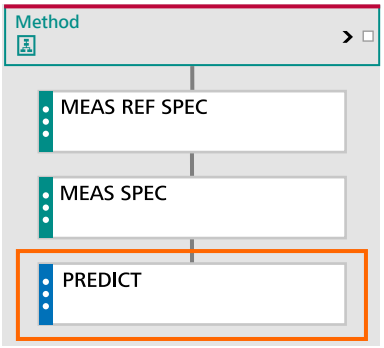
Basic structure



The **PREDICT** command can also be placed before or next to the **VESSEL REMOVAL** command.

Solid samples

Basic structure



3 Configuring the PREDICT command parameters

- Select the PREDICT command.
- Open the **Properties** window by clicking on .

- Define the command parameters under **Properties** ▶ **Parameters**:
  - **Referencing a spectrum**
    Expand the **Name of the measuring command** list. Select the name of the **MEAS SPEC** command that acquires the spectrum to be evaluated.
  - **Referencing a model**
    Select the **Structure** of the model: **Single model** or **Model hierarchy**.
  - If the **Single model** structure was selected, then select the **Model type**: Quantification model, identification model or qualification model.
  - Select the published model or the published model hierarchy.
    Quantification: If necessary, select a slope/y-intercept correction.
    Verification: If the identification model or the model hierarchy is to be used for verification, then switch on the **Use for the verification** option.

4 Multiple parameters of interest (quantification)

If more than one parameter of interest must be predicted for each sample *(see "Multiple parameters of interest (quantification)", chapter 9.1.1, page 156)*, then proceed as follows:

- For each parameter of interest, insert a **PREDICT** command.
  Note: A model hierarchy requires only one **PREDICT** command, regardless of the number of quantification models it contains.
- For each **PREDICT** command, define the command parameters as above. All **PREDICT** commands reference the same spectrum, but a different quantification model for each parameter of interest.

5 Saving a method

- Validate the method by clicking on  .
- Save the method by clicking on  or pressing the **[CTRL]**+**[S]** keys.

Creating an operating procedure

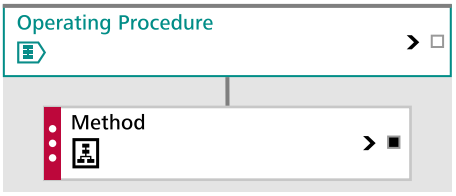
1 Creating and naming the operating procedure

- Under **Processes** ▶ **Operating procedures**, click on  .
  The new operating procedure appears in a new tab.

- Open the **Properties** window by clicking on .
- Enter a matching name in the **Name** field under **Properties** ▶ **General**.

2 Inserting the method

- Open the **Library** window by clicking on .
- Use drag and drop to insert the created method from **Library** ▶ **Methods** into the operating procedure.



3 Defining the result monitoring (optional)

Result monitoring can be used for **quantification**. Example: Monitoring that the analysis result is within certain limits, e.g. within the reference value range of the calibration samples.

As a rule, no result monitoring is used for **identification**. If required, however, the '**IdentificationProbability.Final.***CommandName*' command variable can be monitored in the same way.

- Click on 

.
- Click on to open the **Properties** window.
- Select the **Properties** ▶ **Result monitoring** subsection.
- Click on **[Monitor results]**.

- Click on + to add a new result monitoring:
  - Open the dialog for the variable by clicking on (x).
  - Select the variable of the **PREDICT** command for the predicted value. For quantification, for example: '**Predicted.Quantification.Result.***Command name*'
  - If a model hierarchy variable with index has been selected, then adjust the index in the top input field as required, e.g.: '**Predicted.Quantification{2}.Result.***Command name*' *(see "Model hierarchy – Index for quantification models", chapter 11.4.1, page 176)*
  - Click on **[Apply]** to apply the selected variable.
  - Define the limits of the quantification model in the **Lower warning limit**, **Upper warning limit**, **Lower control limit**, and **Upper control limit** fields. The limits should not exceed the reference value range of the calibration samples.
    Note: For the warning limits, select a smaller area that lies within the intervention limits.
    Identification: Select the value 100 for both upper limits when monitoring the '**IdentificationProbability.Final.***Command name*' command variable.
  - As an option, actions can be defined that are triggered when the limits are violated. At least one **optional run Execute on limit** must be defined in the operating procedure in order for an action to be able to be selected.
  - Close the area by clicking on →.

4 Displaying the results directly in the sample list (optional)

If the prediction results are to be displayed directly in the sample list, then a field for subsample data can be defined *(see "PREDICT command variables", chapter 11.4, page 171)*.

5 Saving the operating procedure

- Click on 🖫 or press the **[CTRL]**+**[S]** keys.

Creating a sample profile

A sample profile simplifies the creation of multiple similar samples.

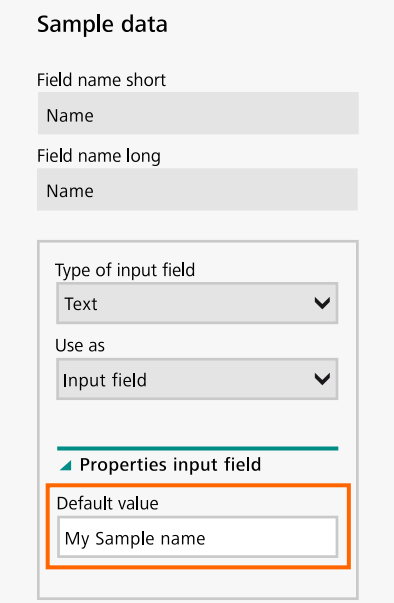
1 Applying and naming the sample profile

Apply the sample profile used for model development *(see "Preparing the spectra acquisition", chapter 4.1, page 49)*.

- Select the sample profile that was used for the model development under **Samples** ▶ **Sample profiles**.
- Duplicate the selected sample profile by clicking on .
- Open the duplicated sample profile by double-clicking on the sample profile name.
- Enter a matching name in the **Name of the sample profile** field.

2 Input field for the sample name

Adjust the default value for the sample name if required.



3 Reference parameters / product parameters

The sample profile contains sample data for the reference parameter (quantification) or for the product parameter (identification, verification).

- **Quantification and identification**: The sample data is not absolutely required for the prediction. The input field can be deleted or used for check samples. Check samples are used to monitor the model and the instrument and to confirm that the system is suitable for further analysis.

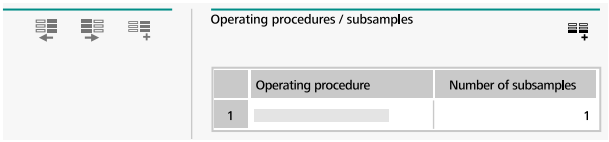
- **Verification**: The product to which the sample is verified is defined in the sample data:
  - **Type of input field**: **Selection list**
  - The product input field must be created for use as a product: **Use as**: **Product**
  - The list elements with the product names should already exist.
  - **Default value**: **Empty**
  - Enable the **Allow empty field** and **Force entry** check boxes.
- **Qualification**: No specific sample data is required for the prediction.

4 Adding additional sample data (optional)

- In the **Sample data** area, add an input field by clicking on  if required.

5 Defining the operating procedure and the number of subsamples

- In the **Operating procedures / subsamples** area, select the created operating procedure.
- Define the number of subsamples: **1**



6 Saving the sample profile

- Click on  or press the **[CTRL]**+**[S]** keys.

Creating a sample list

1 Creating and naming the sample list

- Under **Samples** ▶ **Sample lists**, click on  .
  A new tab opens.
- Enter a matching name in the **Name** field.

2 Adding samples

- In the selection list to the left of the icon, select the created sample profile.

Subsequently added samples are created according to the specifications in the selected sample profile.

- Add a new sample to the sample list by clicking on . Add as many samples as needed.

  Each line of the sample list contains a sample marked with the icon. The sample data follows to the right. This is followed by the subsample marked with and the subsample data.
  The samples are created according to the specifications in the selected sample profile. Each sample contains 1 subsample which uses the defined operating procedure.

	Sample name	My reference parameter		No.	Subsample name	Operating procedure
	Sample 1	%		1	Subsample 1	
	Sample 2	%		2	Subsample 2	
	Sample 3	%		3	Subsample 3	

Figure 6 Sample list (example for quantification)

- Edit the sample names and subsample names as needed.
- Verification: If the product for which the samples are being verified is already known:
  - Select the product in the product input field.

3 Saving the sample list

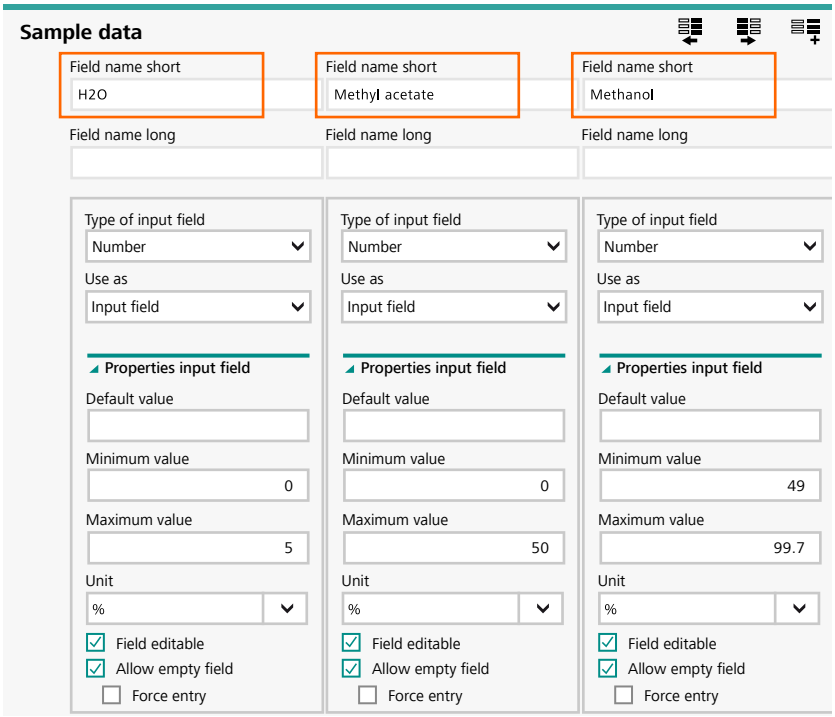
- Click on or press the **[CTRL]**+**[S]** keys.

9.1.1 Multiple parameters of interest (quantification)

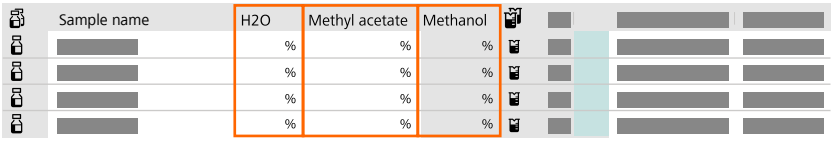
To predict multiple quantitative parameters of interest for each sample, use the following modifications during model development and prediction preparation.

Samples for the development of quantification models

- **Preparing the spectra acquisition**
  In the sample profile, add a separate input field for each reference parameter.



  The sample list contains an input field for each reference parameter.



- **Acquiring the spectra**
  Acquire the spectra as usual.

Developing quantification models

- Create a separate quantification model for each parameter of interest.

Preparing the prediction

Version 1: With model hierarchy

This version requires the creation of a model hierarchy that contains all quantification models. If necessary, the model hierarchy also offers the option of achieving better predictive ability with the help of subordinate quantification models.

Only a single **PREDICT** command is required in the method:

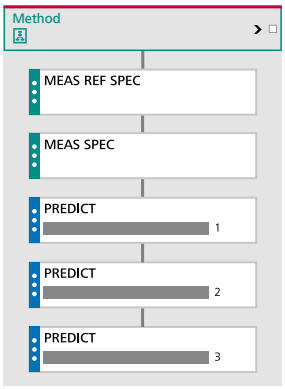
- Create model hierarchy *(see "Inserting models in a model hierarchy", chapter 8.1.2, page 138)*.

- Insert quantification models in the model hierarchy *(see "Inserting models in a model hierarchy", chapter 8.1.2, page 138)*.
- Reference the model hierarchy in the method in the PREDICT command.

Version 2: Multiple PREDICT commands

This version requires multiple **PREDICT** commands in the method. Each command references a quantification model:

- For each parameter of interest, insert a **PREDICT** command in the method:



- Name each **PREDICT** command after one of the parameters of interest.
- Reference the matching quantification model in each **PREDICT** command.
- In each **PREDICT** command, reference the same spectrum, i.e., the same **MEAS SPEC** command.

9.2 Starting the prediction

WARNING

Flammable substances on a hot surface

Risk of fire and burns if flammable substances are spilled. Samples, sample vials, sample holders and the sample presentation can reach temperatures of up to 85 °C.

- Avoid sources of ignition.
- Use protective grounding.
- Use exhaust equipment.
- Clean up spilled liquids and solids immediately.

CAUTION

Volume expansion of the sample due to heating

Injuries and damage to health due to overflowing or breakage of the sample vessel or due to the stopper being blown off.

- Fill the sample vessel only up to the minimum filling height of 2 cm. The liquid can expand in the remaining volume of air.
  Alternatively, use stoppers a with capillary bore.
- Press the stopper in gently so that the sample vessel is not damaged.

CAUTION

Hot sample vials

Burns to the skin due to contact with hot surfaces or hot liquids. Samples, sample vials, sample holders and the sample presentation can reach temperatures of up to 85 °C.

- Wear personal protective equipment and heat-resistant protective gloves.
- Clean up spilled liquids and solids immediately.

Starting the prediction

Prerequisites:

- The prediction is prepared *(see "Preparing the prediction", chapter 9.1, page 149)*.
- The spectrometer is reserved *(see "Reserving and releasing the instruments", chapter 2.4, page 27)*.
- The correct sample holder is inserted. The sample holder must be matched to the sample vessel to be used.

1 Opening a sample list

- If the sample list has been closed, in **Samples** ▶ **Sample lists** open the sample list with a double-click.

Verification

For a verification, the product for which the sample is verified must be defined in the sample data. The product input field must be created for use as a product. Not case-sensitive.

Verification will always fail if the name of a product group that is linked to another identification model is entered in the product input field, verification will always fail. The same applies to a product that is linked to another identification model.

2 Adding additional samples (optional)

If additional samples are needed:

- In the selection list to the left of the icon, select the created sample profile.

Newly added samples are created according to the specifications in the selected sample profile.

- Add new samples to the sample list by clicking on .
- Edit the sample names and subsample names as needed.
- Verification: In the product input field, select the product to which the sample is to be verified.
- Save the sample list: Click on or press the **[CTRL]**+**[S]** keys.

3 Carrying out the determinations

NOTICE

Damage to the temperature sensor during temperature control on the sample vessel

If the sample vessel is removed while the sensor is in direct contact with the sample vessel, then the sensor may be damaged.

- Do not remove the sample vessel until the measurement is complete and the temperature sensor has been moved away from the sample vessel.

- Select the subsample that should be analyzed, using any of the following ways:
  - Select the subsample by clicking on the icon.
  - For analysis purposes, it is sufficient to select a single cell of the subsample.
- Prepare the corresponding physical sample.
  Insert the sample vessel into the sample holder.
- Start the determination by clicking on . A number on the button indicates how many subsamples will be executed.
- The operating procedure assigned to the subsample is started. Follow any instructions that may appear in the **Curves and data** ▶ **Live data** area. If the temperature is controlled at the sample vessel, then do not remove the sample vessel until prompted.
  As soon as the analysis is successfully completed, the status of the subsample will be displayed as .

- Carry out the determinations for all further samples the same way.

The target temperature must not be more than 5.0 K below the ambient temperature.

If the processes are suitable for series determinations, then multiple subsamples can be selected simultaneously. Alternatively, starts all executable subsamples in the sample list.

- Liquid samples: The **VESSEL REMOVAL** command enables series determinations.
- Solid samples: User actions must be provided for carrying out series determinations (e.g., with the **WAIT** command).

Prediction results

The prediction results of the selected samples can be found in the **Results** ▶ **Predictions** area *(see "Prediction results", chapter 9.3, page 161)*.

9.3 Prediction results

Display prediction results:

- Select one or more subsamples.
- The prediction results for all selected and already analyzed subsamples can be found under **Samples** ▶ **Sample list** ▶ **Results** ▶ **Predictions**.

Example for quantification, **Overview** subsection:

Results **Predictions** **Overview** ?

Sample information			Quantification results
No.	Sample name	Subsample name	H2O / %
1	[redacted]	[redacted]	4.7
2	[redacted]	[redacted]	6.1
3	[redacted]	[redacted]	8.0

Multiple subsections are available:

- The **Overview** subsection shows the final results for each subsample.
- The **Detail view** subsection shows detailed prediction results for each subsample. If different models or model hierarchies have been used, then their results are presented in separate tables. A subsample may appear in multiple tables. If necessary, the respective model properties can be displayed.
- Quantification: Evaluated subsamples can be evaluated using other quantification models in the **Reprocessing** subsection.

- Model hierarchy: Evaluated subsamples can be evaluated with a different model hierarchy in the **Reprocessing** subsection.

Prediction results can also be displayed in the subsample data if required *(see "PREDICT command variables", chapter 11.4, page 171)*.

Depending on the model, the results are displayed as follows:

	Results in Samples ▶ Sample list ▶ Results ▶ Predictions	
	Overview subsection	**Detail view subsection**
Quantification	Quantification result (including Slope/y-intercept correction)	▪ **Calculated value**: Prediction result without Slope/y-intercept correction ▪ **Corrected value**: Prediction result with Slope/y-intercept correction **Note**: If no slope/y-intercept correction was applied, then this corrected value is identical to the calculated value. ▪ **Hotellings T²** and **Q residuals** are displayed if the corresponding limit value is exceeded.
Identification	Identification result: ▪ If the identification is successful: Name of the determined product. ▪ If identification has failed: Status of the identification (**Unidentified** or **Ambiguous**).	Additionally: ▪ Probability of the determined product
Verification	▪ Identification result ▪ Verification result: **Successful** or **Failed**	Additionally: ▪ Expected product ▪ Probability of the determined product
Qualification	Qualification result: **Successful** or **Failed**	Additionally: ▪ Grouping by models ▪ Model properties
Model hierarchy	The final results for identification, quantification, and verification are listed side by side. Note: No quantification can be performed if the identification fails.	The detailed prediction results for all models used are listed side by side, each with an indication of the hierarchy level. For subordinate quantification models, the condition defined for the execution of the model is also specified.

Status warning for subsamples

- Check the subsamples in the sample list. In case of errors or warnings, a status warning appears:

- Affected prediction results are marked with the ■ status icon. Hover the cursor over one of the icons to display the causes. Causes can include:
  - Error in the run test before determination of the subsample.
  - Identification: The identification of the sample has failed (Unidentified or Ambiguous).
  - Verification: Verification of the sample has failed.
  - Qualification: The qualification of the sample has failed.
  - Quantification: The acquired spectrum is a spectral outlier (Hotellings T^2 outlier or Q residuals outlier).
  - Quantification: Exceeding the limits defined in the result monitoring (see the following point).

Quantification: Warning limits and intervention limits

If result monitoring is defined in the operating procedure *(see "Creating an operating procedure", page 151)*, then the monitoring status can be checked as follows:

- Open the **Results** ▶ **Predictions** ▶ **Monitoring** area.
- Select the subsample whose monitoring status is to be checked.
  Note: If several samples are selected, then the status of the last sample clicked on is displayed.

Depending on the value of the result, one of the following status icons will appear:

●	The value lies within the defined **warning limits**.
▲	The value lies outside of the defined **warning limits**, but within the defined **intervention limits**.
■	The value lies outside of the defined **intervention limits**.

Visual inspection of the spectra (optional)

- Open the **Curves and data** ▶ **Curves** area.
- Display a single spectrum:
  - Select the corresponding subsample in the sample list (marked with the  icon).
- Displaying multiple spectra:
  - Enable the curve overlay by clicking on  .
  - Select multiple subsamples in the sample list by using the **[CTRL]** key or the **[SHIFT]** key.

- Check the spectra *(see "Handling the charts", chapter 11.3, page 168)*.

10 Test and maintenance intervals

10.1 Instrument performance tests

The instrument performance tests must be performed regularly.

Task	OMNIS command	Recommended execution interval	Result
Wavelength test	**TEST WL**	Unregulated industry: Every 1 to 2 weeks (internal measuring mode) Regulated industry: ▪ Daily: Internal measuring mode ▪ Weekly: External measuring mode	Wavelength accuracy and precision are within the stipulated tolerance.
Noise test	**TEST NOISE**	Unregulated industry: Every 1 to 2 weeks (internal measuring mode) Regulated industry: ▪ Daily: Internal measuring mode ▪ Weekly: Low flux test and high flux test	The noise is within the stipulated tolerance.
Photometric linearity	**TEST PHOTOMETRIC LINEARITY**	Regulated industry: Weekly	The photometric linearity is within the stipulated tolerance.

If a test fails:

- For liquid sample presentation: Check the sampling windows for contamination and clean if necessary.
- Check the operating hours of the lamp module. Replace the lamp if necessary.

- Repeat the instrument performance tests.
  - If the wavelength test fails, repeat the wavelength calibration. If the wavelength test fails again, contact your regional Metrohm service representative.
  - If the noise test fails, contact your regional Metrohm service representative.
  - If the photometric linearity test fails, contact your regional Metrohm service representative.

10.2 Wavelength calibration

After certain actions, a wavelength calibration for the instrument must be carried out in the OMNIS Software. *(see "Starting the wavelength calibration", chapter 3.2.2, page 37)*

Task	OMNIS command	Recommended execution interval	Result
Wavelength calibration	**CAL WL** and **VAL WL**	After replacement of hardware components. After prolonged transport of the instrument.	The x-axis of the spectrum is calibrated.

10.3 Instrument maintenance

Instrument maintenance must be performed regularly.

Task	Execution interval	Result
Maintenance by the regional Metrohm service representative	Annually. If necessary, more often.	The instrument continues to correspond to the technical specifications. Filter mats are checked and replaced if necessary. The internal wavelength standard is recertified.

Recertify external reference standards

If reference standards are used for external instrument performance tests, then these standards must be recertified periodically.

- Observe the next recommended calibration date on the certificate.

11 Appendix

11.1 Reports

Several options are available in the OMNIS Software for the automatic or manual generation of reports (see *Metrohm Knowledge Base*).

The reports typically show the Windows user name, which is different from the OMNIS user name.

11.2 Handling the tables

Tables in models and model hierarchies can be handled as described below. Some of the techniques described can also be used for other tables.

Maximizing and minimizing the area

- Maximize the subsection that contains the table by clicking on ⛶.
- Minimize the subsection by clicking on ⛶.

Changing the column width

1. Position the cursor in the title bar between the first and second column.
   The cursor changes to a ⇔.
2. Hold down the left mouse button and move to the left or right.
3. As soon as the desired width for the first column is reached, release the mouse button.
4. Adjust the other columns one after the other in the same way.

Sorting table lines

1. Click on a column header to sort the lines by this column.
2. If necessary, reverse the sort order by clicking on the column header again.

Selecting one or more table lines

New selection:

- Click on a single line.
  or
- In the overview list of models, you can also move over several lines at once by holding down the left mouse button.

Modify selection:

- Hold down the **[CTRL]** key and click on a single line.
  The selection of the line is inverted. The selection of the remaining lines remains unchanged.
  or
- Hold down the **[SHIFT]** key and click on a line.
  All lines from the last line clicked on without the **[SHIFT]** key to the current line are selected. The remaining lines are deselected.
  or
- Hold down the **[CTRL]**+**[SHIFT]** key combination and click on a line.
  All lines from the last line clicked on to the current line are selected. The selection of the remaining lines remains unchanged.
  or
- Select all lines with **[CTRL]**+**[A]**.

Copying the table to the Windows clipboard

Copy the entire table:

1. Right-click in the table.
2. Select **[Copy table]** in the context menu.

Copy one or more table lines:

1. Select the desired lines.
2. Copy the selected lines with the **[CTRL]**+**[C]** key combination.

The table or the table lines can now be inserted into any file.

11.3 Handling the charts

Charts in models and model hierarchies can be handled as described below. Some of the techniques described can also be used for other charts, e.g. for the spectra in the sample list.

Maximizing and minimizing the area

- Maximize the subsection that contains the chart by clicking on ⛶.
- Minimize the subsection by clicking on ⛶.

Showing and hiding the details window

Some charts contain a details window or a legend. The window can be hidden and shown:

1. Right-click in the chart.
2. Select **[Show/hide details window]** in the context menu.

Zooming

Zooming with the mouse wheel:

1. Position the cursor in the chart.

2. Rotate the mouse wheel forward to zoom in and backward to zoom out.
    a. Zoom vertically only: Press the **[CTRL]** key at the same time.
    b. Zoom horizontally only: Press the **[CTRL]** key at the same time.

Zooming with the mouse button:

- With the left mouse button held down, drag a rectangle over an area, starting from the bottom left or top left corner.
  or
- Hold down **[CTRL]**+**[SHIFT]** and press the left mouse button to zoom in and the right mouse button to zoom out.
  – Zoom vertically only: Hold down the **[CTRL]** key and press the left mouse button to zoom in and the right mouse button to zoom out.
  – Zoom horizontally only: Hold down the **[SHIFT]** key and press the left mouse button to zoom in and the right mouse button to zoom out.

Zoom with the zooming elements:

1. Position the cursor on one of the green zooming elements at the start or end of the x-axis or y-axis.
   The cursor changes to a ⇔ or ⇕.
2. Move along the axis or beyond the axis with the left mouse button pressed down.
3. As soon as the desired view is reached, release the mouse button.

Moving the displayed area

Move in any direction:

1. Position the cursor in the chart.
2. Move the displayed area in any direction by holding the right mouse button pressed down.
3. As soon as the desired view is reached, release the mouse button.

- For touch screens: Press and hold. Then move the displayed area.

Move vertically or horizontally:

1. Position the cursor on the number area of the x-axis or y-axis.
2. Move the displayed area using one of the following methods:
    a. Rotate the mouse wheel.

    a. Move along the axis with the left mouse button pressed down.

Resetting the chart to the default view

- Right-click in the chart. Select **Reset view** in the context menu.
  or
- With the left mouse button held down, drag a rectangle over an area from right to left.

Selecting multiple points or curves

- Click on the points or curves one after the other while holding the **[CTRL]** key pressed down.
  The selection of the point or curve is inverted in each case. The selection of the remaining points or curves remains unchanged.

A mode for multiple selection is available for the **Influence plot**, **Score plot** and **Correlation plot** charts:

- **Enabling multiple selection**
  Click on **Enable multiple selection**.
  Some of the above functions for adjusting the chart are replaced by the following functions.
  - **New selection**
    With the left mouse button held down, drag a rectangle over an area.
    All points within the area are selected. The points outside of the area are deselected.
  - **Expand selection**
    With the **[SHIFT]** key held down, click on individual points or drag a rectangle over an area.
    The corresponding points are selected. The selection of the remaining points remains unchanged.
  - **Reduce selection**
    With the **[ALT]** key held down, click on individual points or drag a rectangle over an area.
    The corresponding points are deselected. The selection of the remaining points remains unchanged.
  - **Invert selection**
    With the **[CTRL]** key held down, click on individual points or drag a rectangle over an area.
    The selection of the corresponding points is inverted. The selection of the remaining points remains unchanged.
  - **Uncheck selection**
    Click in the empty area of the chart.
- **Disable multiple selection**
  Click on **Disable multiple selection** to be able to use the standard functions for adjusting the chart again.

Copying the chart to the Windows clipboard

1. Right-click in the chart.
2. Select **[Copy chart]** in the context menu.

The chart can now be inserted into any file.

Chart in sample list

Curve overlay

Display multiple spectra together in a sample list:

- Open **Curves and data** ▶ **Curves** in the sample list.
- Enable the curve overlay by clicking on . If the icon is not displayed, then enlarge the area by dragging the divider.
- Select multiple subsamples in the sample list by using the **[CTRL]** key or the **[SHIFT]** key.

  To view all of the spectra in the sample list, click on or .

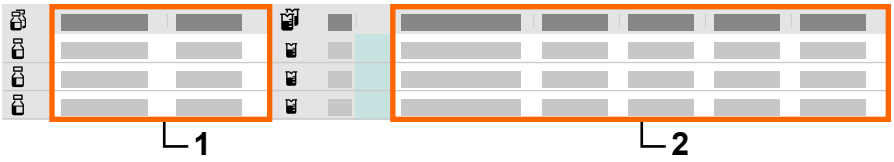
11.4 PREDICT command variables

The OMNIS Software offers different categories of variables, for instance sample data, subsample data, method variables, command variables or system variables.

The software creates some variables automatically. Additional variables can be created if needed. When using variables, their data type must be considered (**Number**, **Text** or **Date/Time**).

The variables can either be used for further calculations, output as a result in reports or entered as a condition, e.g., in the **IF** command.

The sample data **(1)** and the subsample data **(2)** appear in the sample list in the **Samples** work area:



To create **sample data**, edit the sample profile.
To create **subsample data**, edit the operating procedure.

Displaying the prediction results as subsample data

As an example, the following **PREDICT** command variables are to be displayed in the subsample data *(see "Spectra acquisition", chapter 2.3.1, page 19)*:

- Quantification: **Predicted.Quantification.Result.***Command name*
  Predicted value for the parameter of interest.

- Identification: **Product.Identification.Result.***Command name*
  Determined product or determined product group of the identified sample. If the identification has failed, then the variable will remain empty.

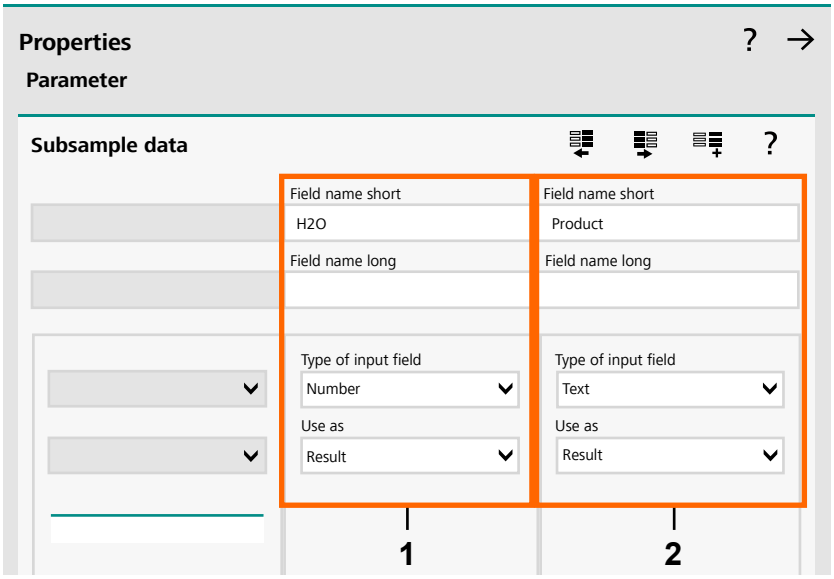
Prerequisite:

A method and an operating procedure have been created to prepare the prediction *(see "Preparing the prediction", chapter 9.1, page 149)*.

1 Creating the subsample data

- Open the corresponding operating procedure.
- Click on Operating procedure.
- Open **Properties** ▶ **Parameters**.
- Click on to create a subsample data field.
  - Enter a matching name for the predicted result or the determined product as **Field name short**.
  - **Quantification**: To create a numeric data field as **Type of input field**, select the **Number** option.
    Identification: To create an alphanumeric data field as **Type of input field**, select the **Text** option.
  - The data field should be filled with the result of a **CALC** command. Therefore, select the **Result** option under **Use as**.
    Note: Input fields for a **Result** cannot be edited manually.

Example for quantification (**1**) and for identification (**2**):



- Save the operating procedure: Click on or press the **[CTRL]**+**[S]** keys.

The variable name for the created subsample data depends on the selected **Field name short**:
'*Field name short*.**CurrentSubsampleData**'

Quantification: If multiple parameters of interest are predicted for each sample, then create separate multiple subsample data for the predicted results.

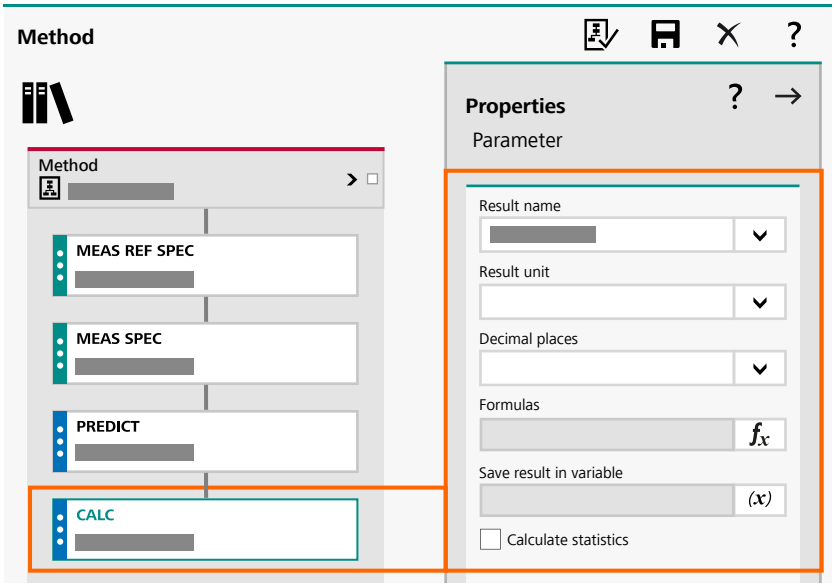
2 Inserting a CALC command for the predicted result

Use the result of a **CALC** command to fill the subsample data that has been set up:

- Open the corresponding method.
- Insert a **CALC** command.
  Arrange the **CALC** command under the **PREDICT** command.

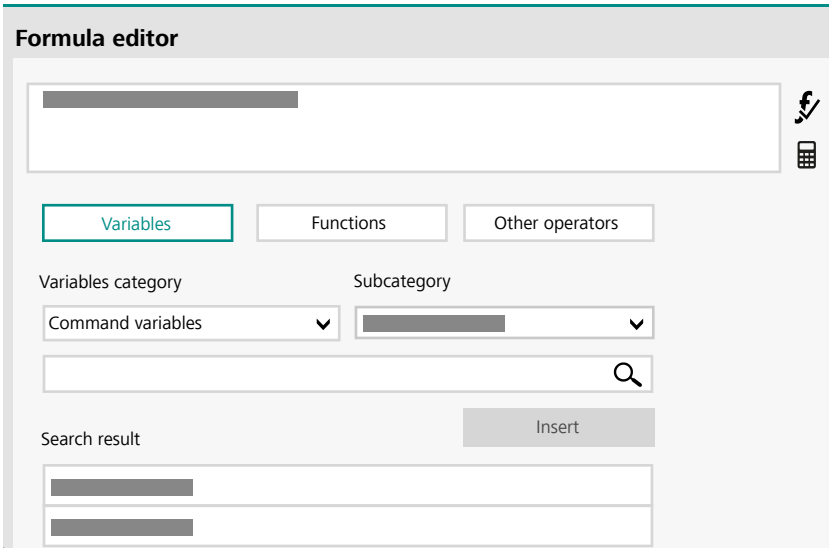
Calculating the value to be displayed

- Select the **CALC** command and open **Properties** ▶ **Parameters**.
  - Enter a matching name for the predicted result or the determined product as **Result name**.
  - Quantification: Enter the **Result unit** and the number of **Decimal places** needed.



Calculate statistics operates on multiple subsamples within a sample. Disable **Calculate statistics**.

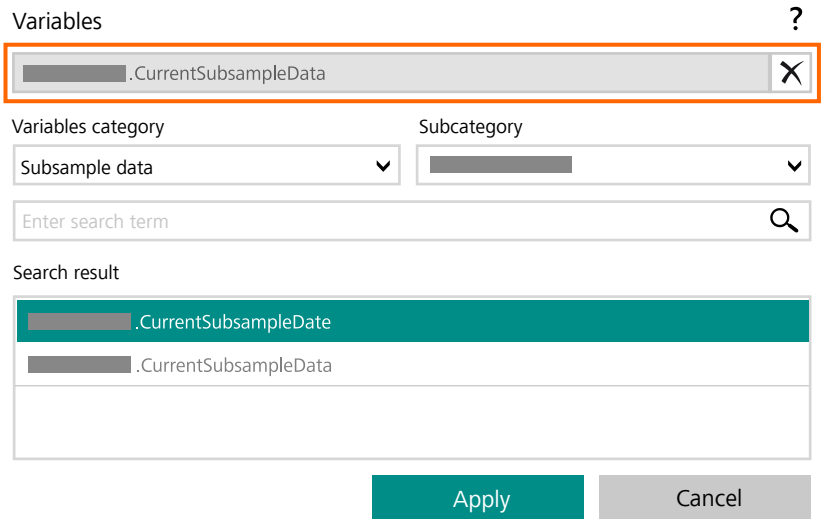
- In the **Formula** field, click on f_x to open the formula editor.



- Create a formula by using the **Command variables** variables category. For the result view, the formula consists of only a single **PREDICT** command variable:
  - Identification:
    Product.Identification.Result.*Command name*
  - Quantification:
    Predicted.Quantification.Result.*Command name*
    If a model hierarchy variable with index has been selected, then adjust the index in the top input field as required, e.g.:
    '**Predicted.Quantification{2}.Result.***Command name*'
    (see "Model hierarchy – Index for quantification models", chapter 11.4.1, page 176)
- Check the validity of the entered formula by clicking on .
- **Note**: The prediction result is not generated until the runtime of a determination. Calculating the formula by clicking on will therefore display the result **Invalid**.
- Close the formula editor by clicking on →.

Saving the calculated value in the subsample data

- In the **Save result in variable** field, click on (x).
- Select the variables category **Subsample data**.
- As subcategory, select the corresponding operating procedure.
  The Search result shows the subsample data defined in the selected operating procedure.
- Select the newly created variable.
  The selected variable is inserted into the **Variables** field.



- Click on **Apply**.
- Save the method: Click on or press the **[CTRL]+[S]** keys.

Quantification: If multiple parameters of interest are predicted for each sample, then insert a **CALC** command for each predicted result.

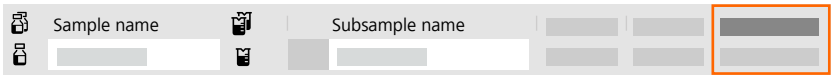
Analyzing the samples

1 Opening a sample list

- If the sample list has been closed, in **Samples** ▶ **Sample lists** open the sample list with a double-click.

2

A field appears in the sample list for all samples that have not yet been analyzed for the subsample data created:



After the prediction, the field displays the result.

Identification: If the identification has failed, then the result field will remain empty.

Additional variables

Additional variables can be displayed in the subsample data in the same way as above *(see "Prediction", chapter 2.3.2, page 21)*.

11.4.1 Model hierarchy – Index for quantification models

A model hierarchy can contain multiple quantification models. Index numbers are used so that the **PREDICT** command variables can differentiate between the quantification models.

The first quantification model that is linked to a specific product or specific product group is assigned Index 1. If additional quantification models are linked to the same product or product group, then the index will be incremented. Incrementing is based on the order of the quantification models in the model hierarchy.

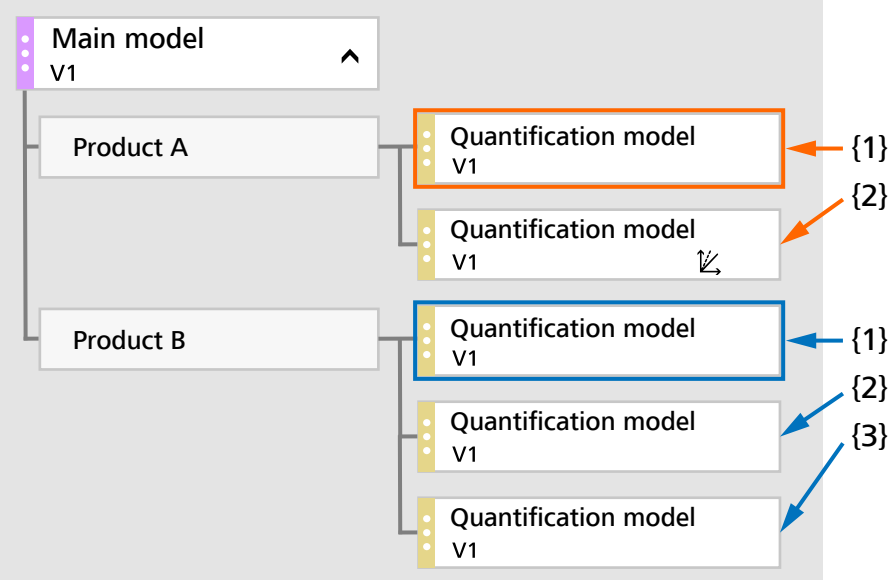


Figure 7 Index numbers for quantification models in a model hierarchy

The **PREDICT** command variables reference the quantification model via the index number.

Example: '**Predicted.Quantification{1}.Result**.*Command name*'

If a sample is identified as product A for the prediction, then all quantification models linked to product A will be used. In this case, the above command variable references the quantification model linked to product A with Index 1 (framed in orange in the image).

If a sample is identified as product B, then the above command variable references the quantification model linked to product B with Index 1 (framed in blue in the image).

If the command variable for the quantification model framed in blue is to be used in a different way than for the quantification model framed in orange, then a **IF** command can restrict processing to a specific product membership.

In a model hierarchy without identification models, the top quantification model is assigned the Index 1. For the other quantification models, the index is incremented sequentially.

Subordinate quantification models

No index number is assigned to the subordinate quantification models. The **PREDICT** command variable must reference the superordinate quantification model. The variable, on the other hand, provides the values of the subordinate model used for each subsample.

11.5 Exporting and importing models

Models can be exported and imported for use in another OMNIS installation.

Exporting models

1 Opening the export dialog

- Open one of the following subsections in the **Calibration and evaluation** work area:
  - **Quantification models**
  - **Slope/y-intercept corrections**
  - **Identification models**
  - **Qualification models**
  - **Model hierarchies**
- Click on .

An export dialog opens.

2 Creating export files

- Select all models to be exported.
- If only published **type full** models have been selected, then the **Export type** can be defined:
  - A **type full** model is fully functional. After importing, the model can be edited and saved.
  - If a model has been published, then the latest published version can be exported as **type light**. This model is usable for predictions only.
- Adjust the target folder as needed.

The following special characters or character strings may not be used: > < : " / \ | * ? and CON, PRN, AUX, NUL, COM1–COM9, LPT1–LPT9.

- Click on **[Export]** to create the export files.

The models are exported to the chosen folder.

Importing models

1 Opening the import dialog

- Open one of the following subsections in the **Calibration and evaluation** work area:
  - **Quantification models**
  - **Slope/y-intercept corrections**
  - **Identification models**
  - **Qualification models**
  - **Model hierarchies**
- Click on  .

2 Opening files

- Select the folder and all *.opmo files or *.osic files to be imported.
- Click on **[Open]**.

The models are imported into the OMNIS Software.

11.6 Switching from XDS/DS Analyzers (quantification)

When switching from a DS2500 or XDS Analyzer, the spectra and reference parameters used to create a quantification model for XDS/DS Analyzers can be imported into the OMNIS Software. A quantification model can be developed using these data.

In a second step, a slope/y-intercept correction is created. Spectra acquired with the OMNIS Software are used for this purpose. The reference values for these spectra must be known.

Fewer samples are used for the slope/y-intercept correction than for the development of a quantification model:

- A reliable estimate of the bias needs at least 20 samples.
- A reliable estimate of the slope needs at least 30 samples.

Developing the quantification model

Prerequisite:

- The spectra file (.da) containing the XDS/DS spectra is available.

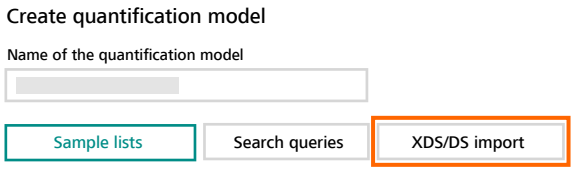
- In the same folder, the reference parameter file (.cn) containing the reference values is available.

1 Creating and naming the quantification model

- Under **Calibration and evaluation** ▶ **Quantification models**, click on .
- Enter a matching name in the **Name of the quantification model** input field.

2 Importing the spectra

- Click on **XDS/DS import**.



- Click on .
- Select the spectra file (.da) that is to be imported.
- Click on **[Open]** to confirm the selection.
- Click on **[Continue]**.

3 Selecting the reference parameter

- Select the reference parameter from the **Reference parameter / unit** list.
- In the **Unit of reference parameter** input field, select the unit of the reference parameter.

All spectra that have the selected designations of the reference parameter are added to the quantification model.

4 Automatic or manual model development

Several options are available for model development:

- Automatic model development with the **OMNIS Model Developer (OMD)**: Click on **[Start OMD]**.
  Continue with *chapter 5.2, Automatic model development – OMD, page 67*.
- Manual model development: Click on **[Create]**.
  Continue with *chapter 5.3, Manual model development, page 70*.

5 Automatic or manual model development

Continue with manual model development if the manual adjustment of the sample selection is to take place first, followed by the automatic development of the model.

- **Automatic model development**: Click on **[Start OMD]**.
  Continue with *chapter 5.2, Automatic model development – OMD, page 67*.
- **Manual model development**: Click on **[Create]**.
  Continue with *chapter 5.3, Manual model development, page 70*.

6 Publishing the model

- Publish the quantification model *(see "Publishing a quantification model", chapter 5.4, page 92)*.

Samples for the slope/y-intercept correction

Each sample for the slope/y-intercept correction needs:

- A reference value for the parameter to be corrected.
- A spectrum.
- A calculated value for each of the spectra.

1 Collecting samples

- Collect the physical samples for the slope/y-intercept correction as if they were samples for a quantification model development *(see "Preparing the model development", chapter 4, page 48)*. However, a smaller number of samples is sufficient.

2 Preparing the spectra acquisition and the prediction

- Create a method, an operating procedure, a sample profile, and a sample list as though the samples were samples for a quantification model development *(see "Preparing the spectra acquisition", chapter 4.1, page 49)*. However:
- **Method**
  In the method, add a **PREDICT** command after the **MEAS SPEC** command and, if available, after the **VESSEL REMOVAL** command.
  State the following as **PREDICT** command parameters:
  - **Name of the measuring command**
  - The **Quantification model** developed with the imported spectra.

3 Acquiring the spectra and predicting the parameters of interest

- Acquire the spectra and predict the parameter of interest as if the samples were samples for a quantification model development *(see "Acquiring spectra", chapter 4.2, page 59)*.

Afterwards, each sample has a spectrum, a reference value, and a calculated value.

If there are multiple parameters of interest, then each sample has a spectrum and, for each parameter of interest, a reference value and a predicted value.

Creating the slope/y-intercept correction

- Create a slope/y-intercept correction. Spectra acquired with the OMNIS Software are used for this purpose. The reference values for these spectra must be known. *(see "Slope/y-intercept correction", chapter 5.5, page 93)*

Prediction

1 Preparing the prediction

- Prepare the processes needed for prediction *(see "Preparing the prediction", chapter 9.1, page 149)*.
  While doing so, state the quantification model and the corresponding slope/y-intercept correction in the **PREDICT** command.

2 Carrying out the prediction

- Carry out the prediction *(see "Starting the prediction", chapter 9.2, page 158)*.

The carrying out of the analysis should be monitored with check samples. Check samples are measured using both the spectroscopic method and the reference method. The results of both methods can be compared.

11.7 Workflows for the OMNIS NIR Analyzer

To analyze samples with an OMNIS NIR Analyzer, the OMNIS Software performs the following tasks:

1. Preparing the instrument:
   a. Instrument configuration
   b. Wavelength calibration and validation
   c. Instrument performance tests
2. Acquiring spectra of the calibration samples
3. Recording reference values of the calibration samples
4. Developing models
5. Predicting the parameters of interest
6. Instrument performance test: repeat as needed.

The following sections illustrate the corresponding workflows in the OMNIS Software.

Preparing the instrument

Before spectra can be acquired, the instrument must be prepared. Among other things, a wavelength calibration must be carried out.

As an example, the following *Figure 8* illustrates a wavelength calibration. The method is illustrated by a single command.

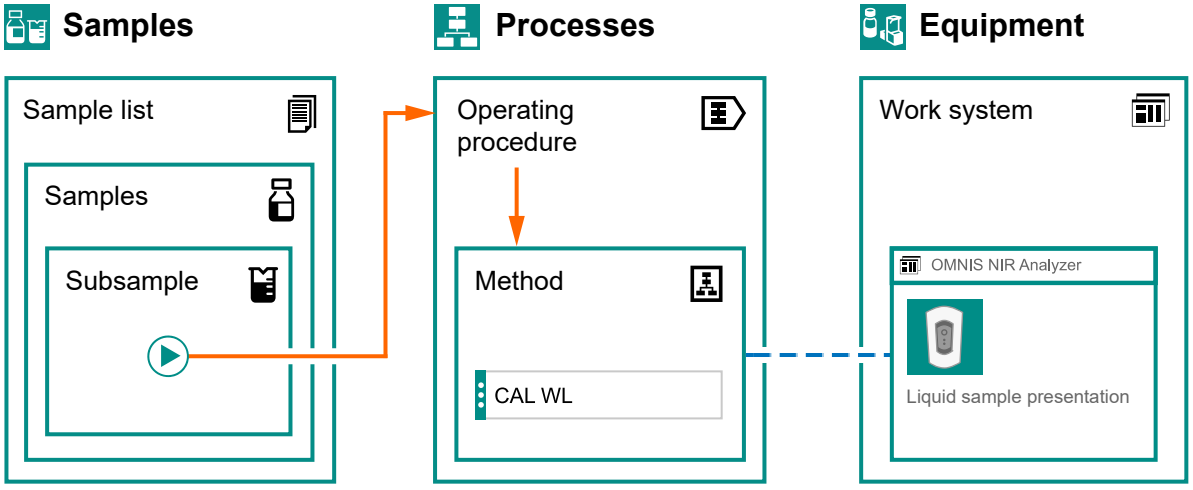


Figure 8 Instrument setup

Links in the OMNIS Software.

- - - - -	The method has a work system assigned to it.

The **Samples** work area contains a sample list with a sample. The sample contains a subsample. The subsample has an operating procedure assigned to it.

Normally, samples are used to analyze real samples. In this case, however, the sample is used to carry out a wavelength calibration. The following steps will be performed as soon as the corresponding subsample is started:

1. The subsample starts the assigned operating procedure.
2. The operating procedure executes the method contained in it.
3. The method runs the **CAL WL** command. The command is executed with the work system that is assigned to the method.
   The work system contains a functional unit of the OMNIS NIR Analyzer instrument. This instrument carries out a wavelength calibration. The calibration data is saved to the instrument.

Recording reference values or the product names

To create a model, a reference value (quantification) or a product name (identification) must be recorded for each calibration sample and validation sample. For the qualification, it must be known whether the individual samples are to be classified as positive or negative.

Example for quantification

The following *Figure 9* indicates the recording of a reference value for the parameter of interest, for instance the water content of a sample.

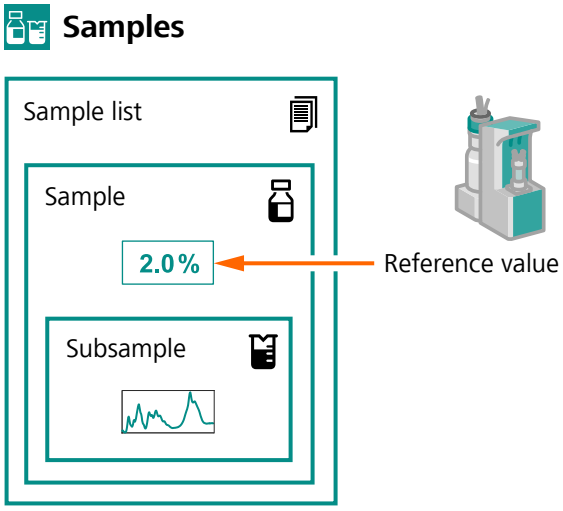


Figure 9 Record a reference value

The parameter of interest is measured with a reference method, e.g. via titration. The measured value serves as reference value.

The reference value is entered into the corresponding input field for each sample in the sample list.

Acquiring spectra of the calibration samples

To create a model, a spectrum must be acquired for each calibration sample and validation sample.

The following *Figure 10* shows a schematic depiction of the acquisition of a spectrum.

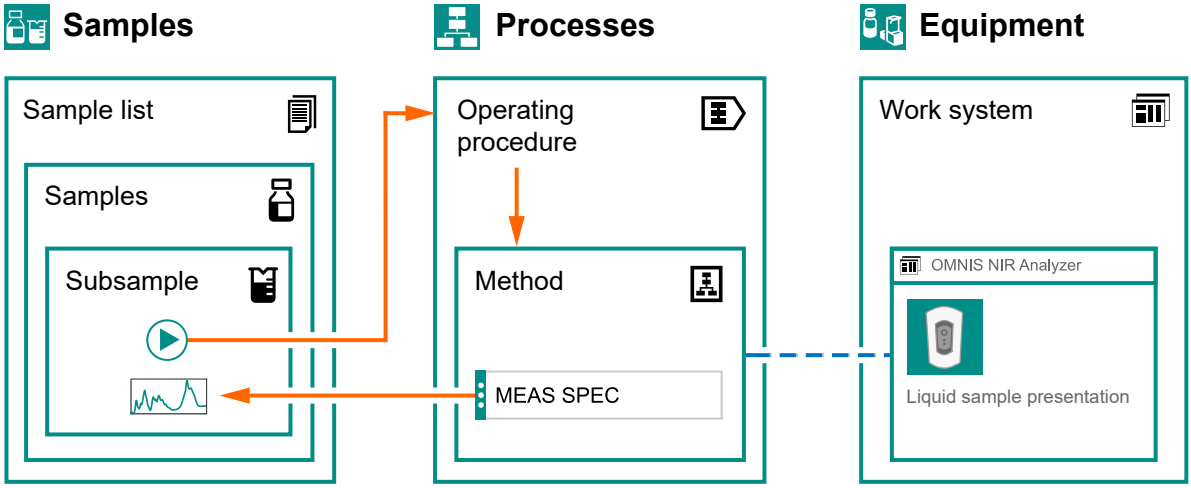


Figure 10 Acquiring a spectrum of a calibration sample

→	Links in the OMNIS Software.
- - - -	The method has a work system assigned to it.

The **Samples** work area contains a sample list with a calibration samples. Each sample contains a subsample. Each of the subsamples has an operating procedure assigned to it.

As soon as a subsample is started, the following steps are performed:

1. The subsample starts the assigned operating procedure.
2. The operating procedure executes the method contained in it.
   The method runs the **MEAS SPEC** command. The command is executed with the work system that is assigned to the method.
   The work system contains a functional unit (e.g., **Liquid Sample Presentation**). The functional unit acquires a spectrum and transmits it to the OMNIS Software. The OMNIS Software saves the spectrum in the subsample data.

Developing a model

Quantification: A quantification model is created from the spectra and reference values of the calibration samples; either automatically with the **OMD** (OMNIS Model Developer) or manually.

Identification: An identification model is created from the spectra and product names of the calibration samples.

Qualification: A qualification model is created from the spectra of the calibration samples.

Example for quantification

The following *Figure 11* shows the steps for the manual development of a quantification model.

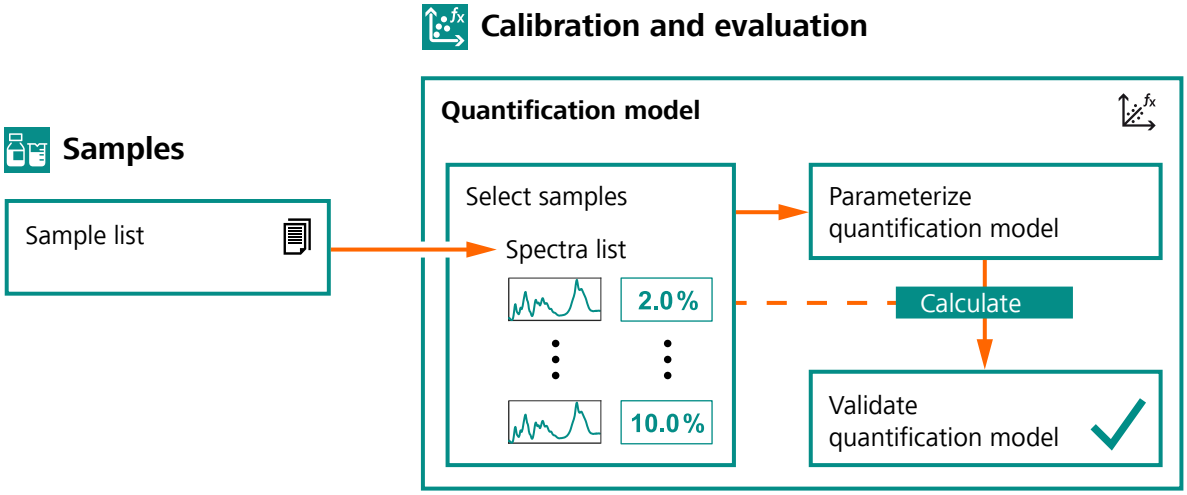


Figure 11 Developing a model (example for quantification)

First of all, the samples including reference values are transferred to a spectra list. The model is then developed in 3 process steps:

1. The **Select samples** process step enables the detection of outliers, the definition of validation spectra and the specification of a cross-validation method.
2. Data preprocessing can be applied to the spectra and wavelength ranges can be defined in the **Parameterize quantification model** process step.
3. Once a model has been calculated, the **Validate quantification model** process step is used to assess whether the model corresponds to the requirements.
   The previous steps can be adjusted to optimize the model. If an suitable model has been found, it can be **published**. The published model can then use unknown samples for the prediction.

Prediction

For the prediction, a model is applied to the spectrum of an unknown sample. Depending on the model, the following can be predicted:

- Parameter of interest (quantification)
- Product membership or verification result (identification)
- Qualification result (qualification)

Example for quantification: The following *Figure 12* illustrates the prediction of a parameter of interest.

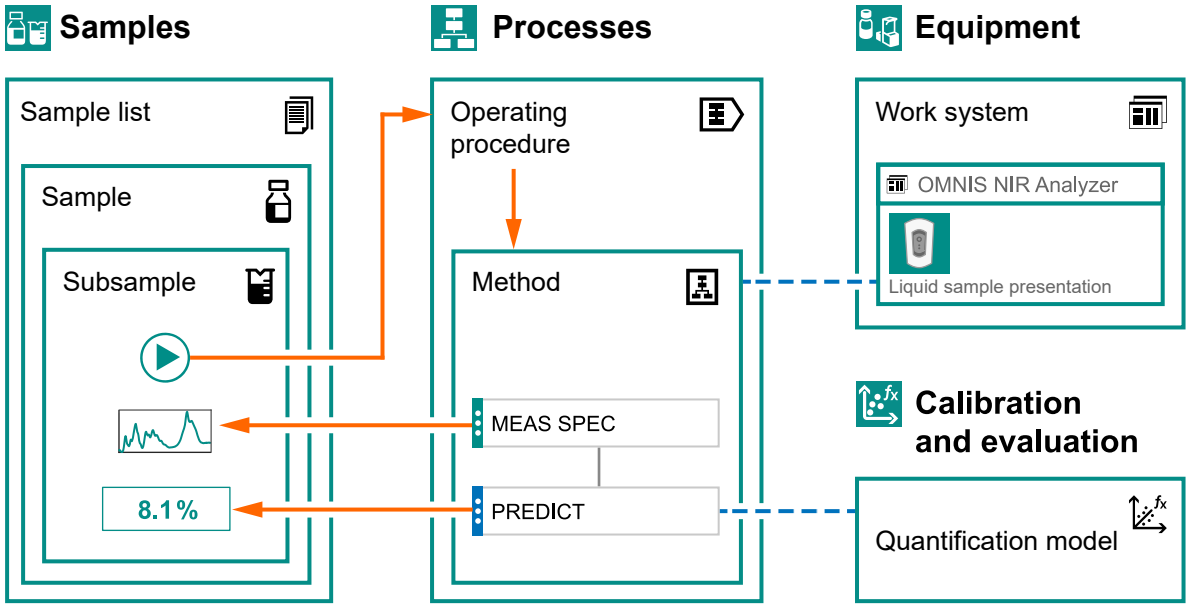


Figure 12 Predicting parameters of interest (example for quantification)

→	Links in the OMNIS Software.
- - - -	The method has a work system assigned to it. The **PREDICT** command specifies a model.

The **Samples** work area contains a sample list with samples that are ready for analysis. Each sample contains a subsample. Each of the subsamples has an operating procedure assigned to it.

As soon as a subsample is started, the following steps are performed:

1. The subsample starts the assigned operating procedure.
2. The operating procedure executes the method contained in it. The method contains at least 2 commands:
   a. **Measuring command**
   The **MEAS SPEC** command acquires a spectrum of the sample. The command is executed with the work system that is assigned to the method.
   The work system contains a functional unit (e.g., **Liquid Sample Presentation**). The instrument uses this to acquire a spectrum and transmit it to the OMNIS Software.
   b. **Prediction**
   A measuring command and a model are selected in the **PREDICT** command. The model evaluates the spectrum that was acquired with the measuring command. This results in a prediction, e.g., a water content of 8.1%.