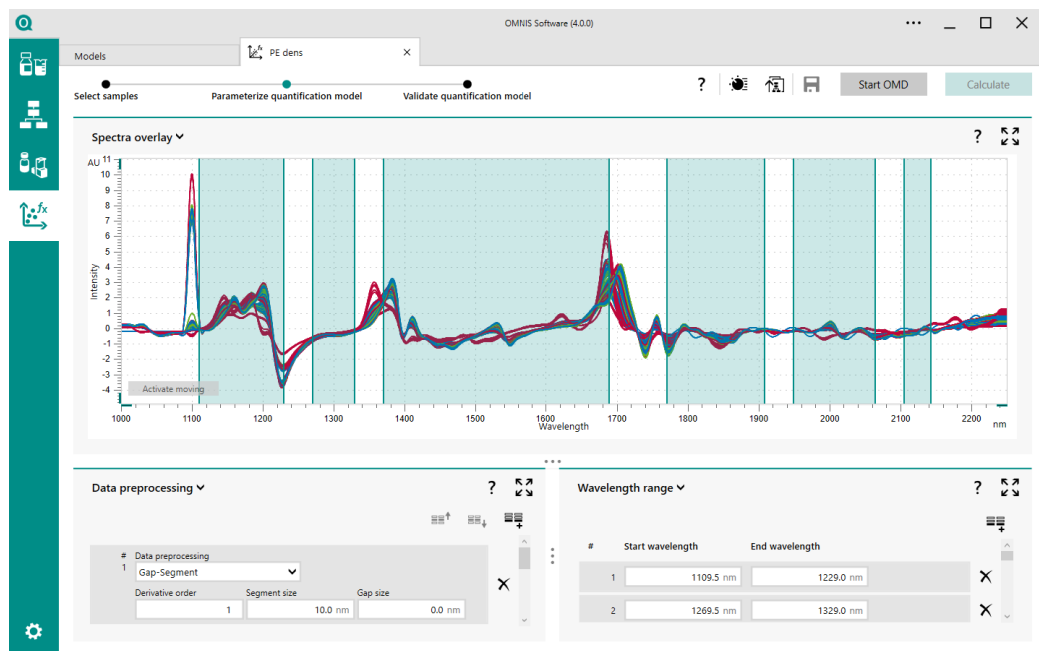


OMNIS NIR (Lab)



OMNIS Spectroscopy step by step

Tutorial

8.0600.8202EN / v7 / 2026-02-17



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OMNIS NIR (Lab)

OMNIS Software version 4.7

Tutorial

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OMNIS Software version 4.7

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 4.7.

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:

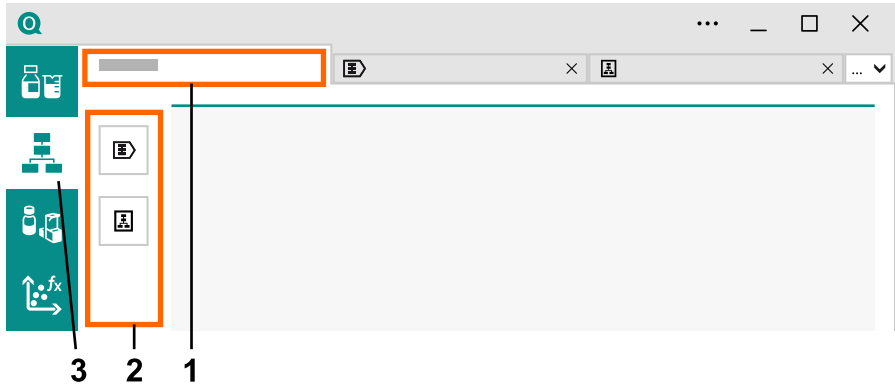
- Instrument functions are available as license packages (see [Metrohm Knowledge Base](#)).

To work through this tutorial, the following function license is required:

- Function license Lab NIR Spectroscopy

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 ► Manual**.



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.

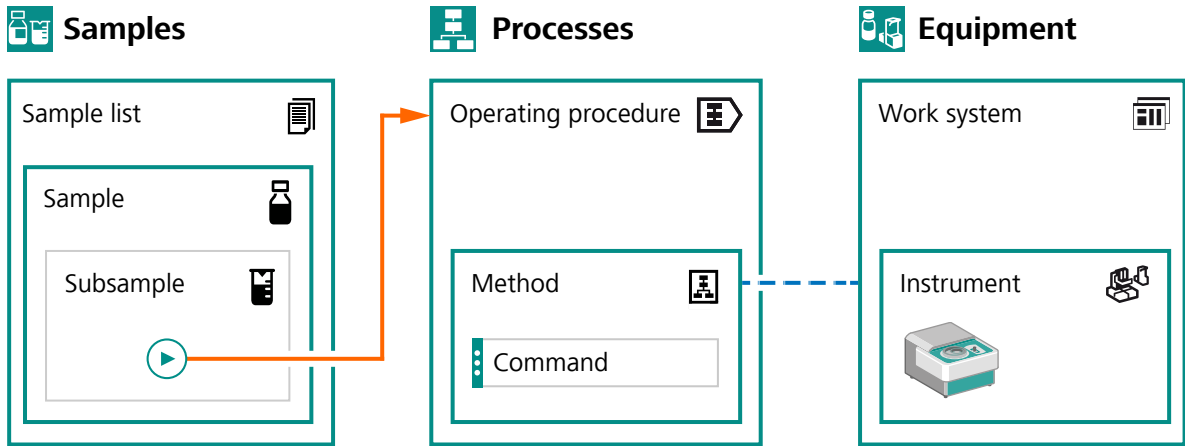


Figure 1 3 work areas with relevant subsections

	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.

- 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.

Processes – Overview

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

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



Processes

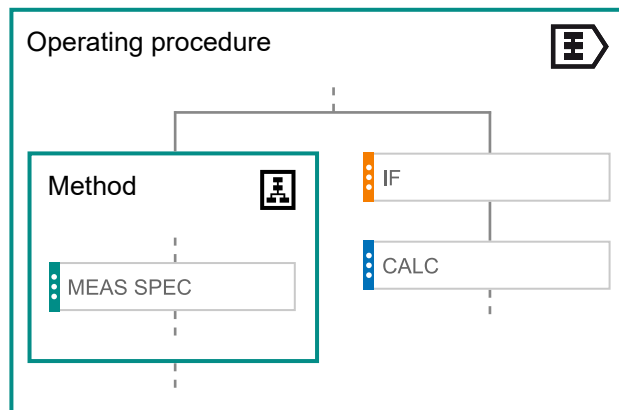


Figure 3 Processes work area

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.

Equipment

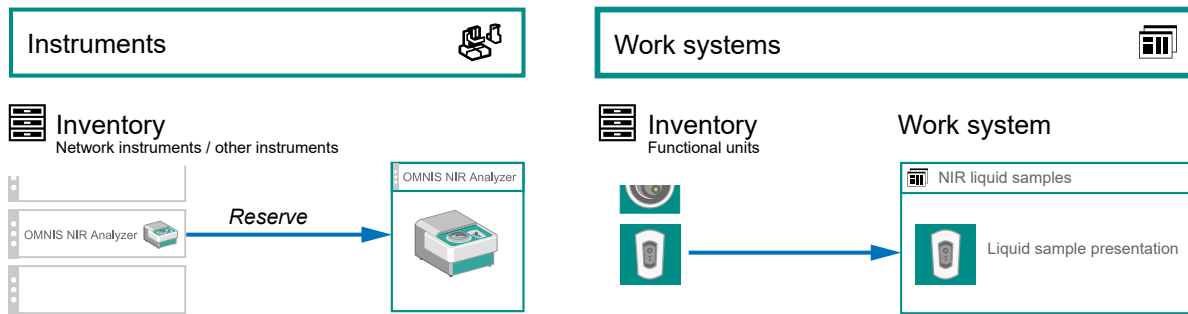


Figure 4 Equipment work area

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

2.2 Practical introduction

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

Work areas

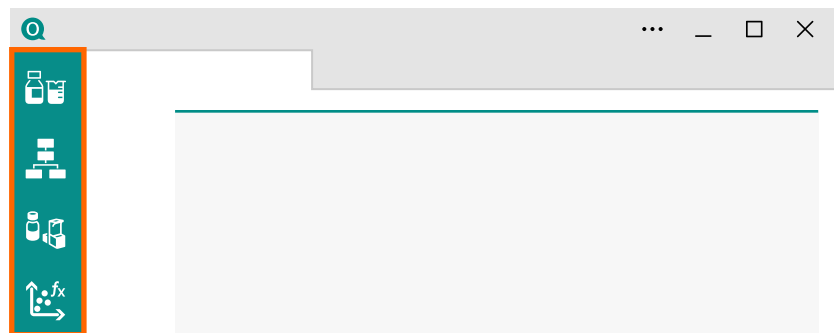
The OMNIS Software divides the user interface into multiple work areas. Work areas can have different subsections.

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.

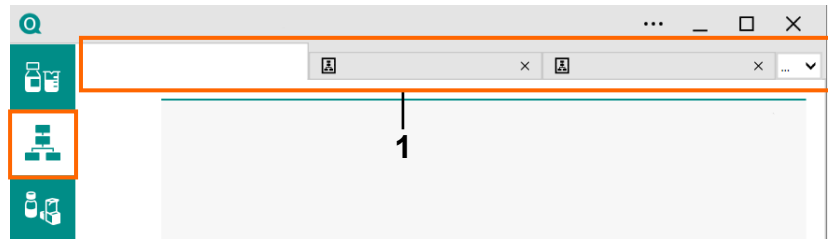


For the above example, open the **Processes** work area by clicking on the corresponding icon .

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.

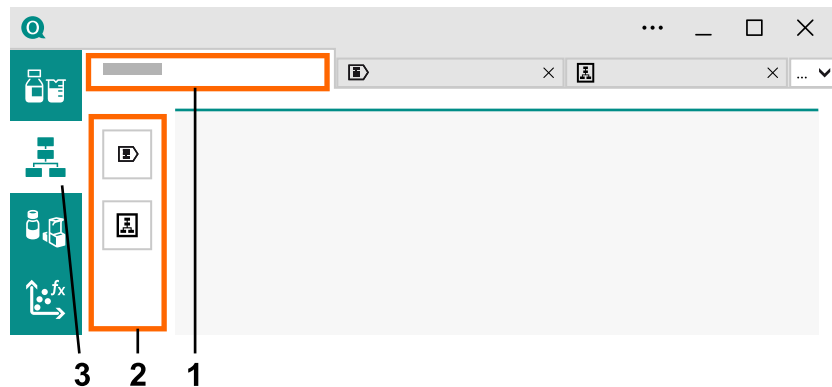
To show tooltips on a touch screen, touch and hold the element.



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

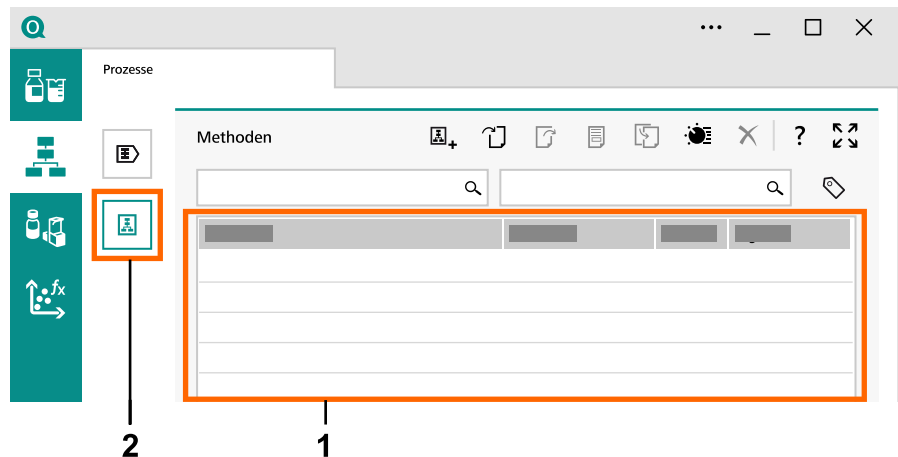
2 Opening the Subsection tab

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).

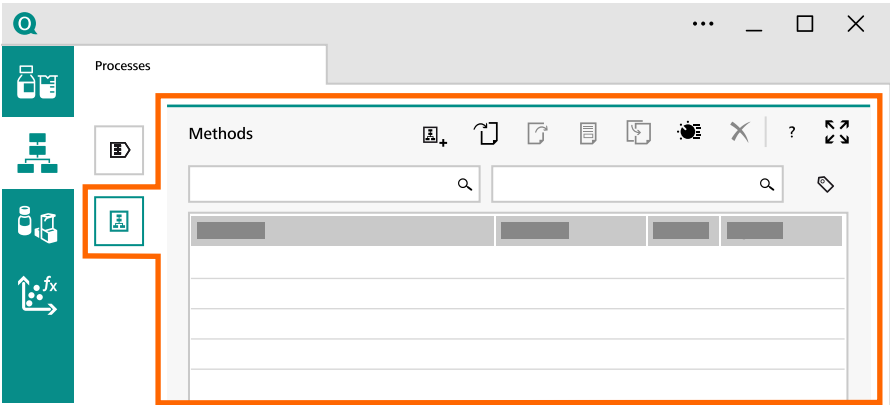
Tabs

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

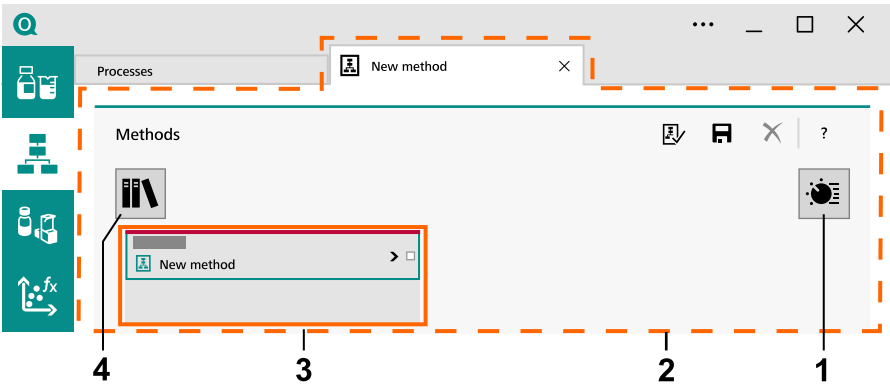
Open **Processes** ► **Methods** as described above.



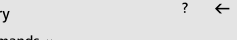
2 Creating a method

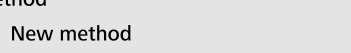
- Click on +.

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.

- 

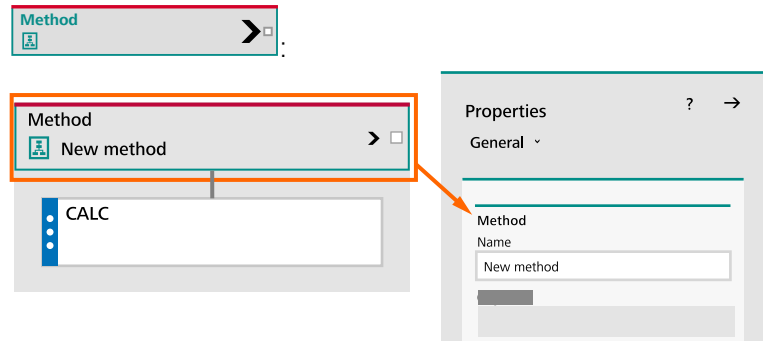


- To close the library window, click on .

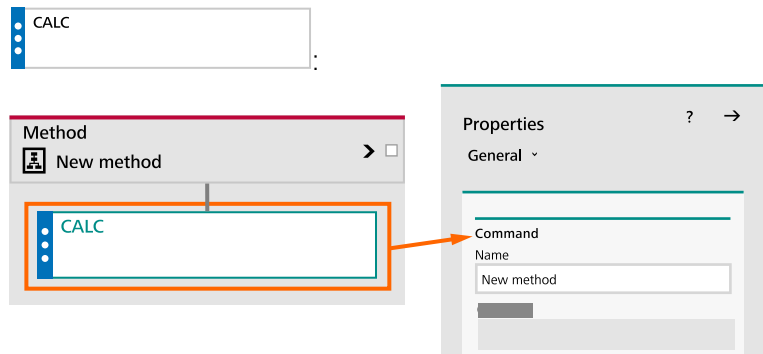
2 Properties window

- 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**.

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.

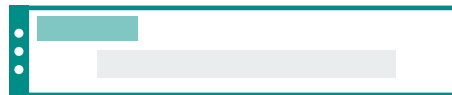
Some commands can also be inserted in operating procedures, e.g., the **IF** command.

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.

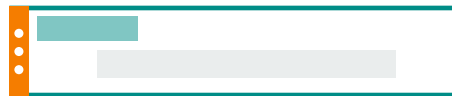
 It is good practice to change the default command name (e.g., Acquire spectrum 1), to a more specific name. The cross-references to the command are automatically adjusted.

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

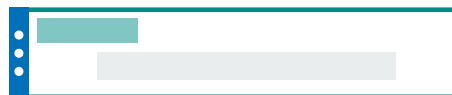
- Measuring commands, calibration commands and titration commands



- 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:

Command name	Description	Resulting command variables (followed by <i>.Command name</i>)
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.

Command name	Description	Resulting command variables (followed by <i>.Command name</i>)
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.

- 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 164*).

 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 181*).

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**.

- i** 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

- i** 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  +.

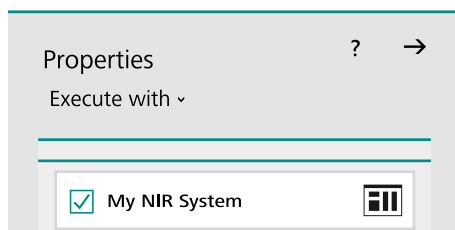
A tab opens with the newly created method and the title **New method**.

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

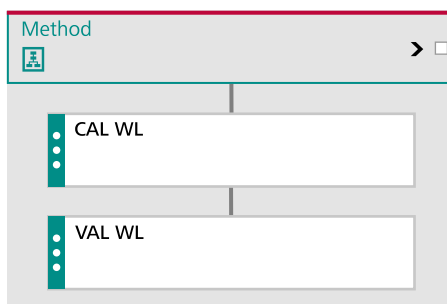
- Under **Properties** ► **Execute with**, select the work system to be used.



- i** 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

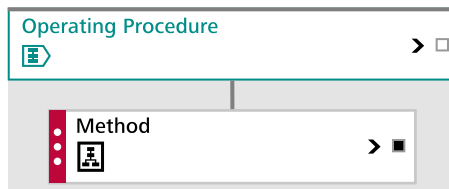
- Open **Processes** ► **Operating procedures** by clicking on , then on .
- Create a new operating procedure by clicking on .

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.

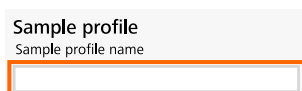
Creating a sample profile

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:

Confirm with **[Enter]**.

Sample list ▾ 

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 165*).

 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 .

 Sample name	 No.	Subsample name
 Sample 1	 1	Subsample 1

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:

Overall status
Successful

 **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)**

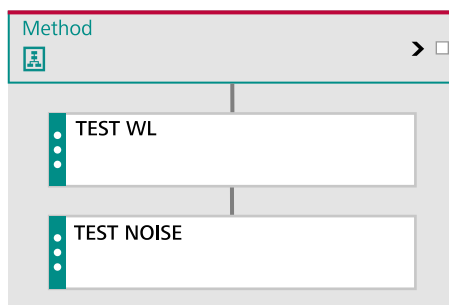
Internal instrument performance tests must be carried out successfully before the corresponding functional unit can be used to acquire spectra.

- 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*).



 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 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**.

Operating procedure	Number of subsamples
1	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**.

Sample list ▾

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 164*).

Executing the instrument performance test

Prerequisite:

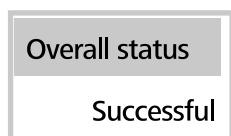
- Start the test by clicking on .

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:



 **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 164*).

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*).

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 164*).

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.

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 182*)
- Recording reference values or the product names (*see "Recording reference values or the product names", page 182*)

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**.

 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 sample vessel to be used for measuring the sample.
 - Select the measuring mode:
 - Single-point measurement** for homogeneous solids. The instrument performs a measurement at a position. The sample holder does not rotate.
 - Multi-point measurement** for heterogeneous solids. The following two modes are available:
 - (a) **Five-point measurement**: The instrument performs measurements at 5 different positions. After each measurement, the sample holder is rotated to the next position.
 - (b) **Continuous measurement**: The instrument measures continuously while the sample holder rotates.

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 (only for disposable vials) or the 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*).

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.

- 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.
 - Deactivate 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.

Sample data

Field name short
Name

Field name long
Enter name

Type of input field
Number

Use as
Input field

Properties input field

Default value

Minimum value
-10000000000

Maximum value
10000000000

Unit

☒ Field editable
☒ Allow empty field
☐ Force entry

Write product names in the text field

Sample data

Field name short

Field name long

Enter name

Type of input field

Text

Use as

Product

Properties input field

Default value

☒ Field editable

☒ Allow empty field

☐ Force entry

Select product names from the list

Sample data

Field name short

Field name long

Enter name

Type of input field

Selection list

Use as

Product

Properties input field

Select elements

List elements

Enter name

Product A

Product B

Product C

Default value

Empty

☐ Allow free text

☒ Allow empty field

☐ Force entry

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**.

Operating procedures / subsamples

Operating procedure	Number of subsamples
1	1

6 Saving the sample profile

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

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

- Select the sample profile already created under **Samples ► Sample profiles**.

- 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



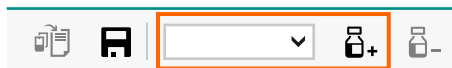
WARNING

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.

- 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.

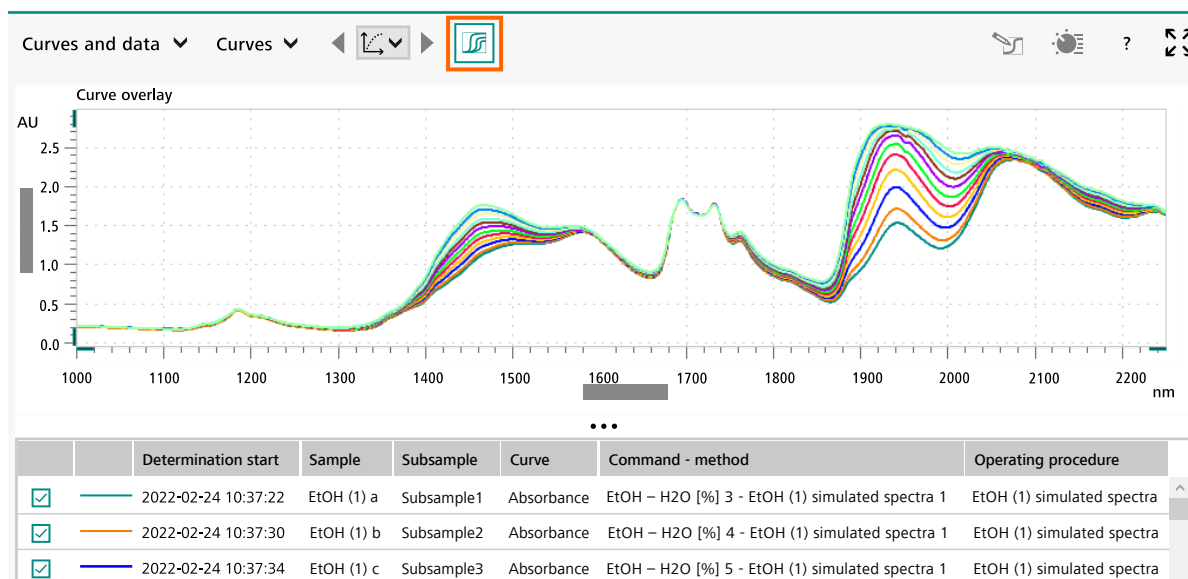
3 Carrying out the determinations

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.



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.

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 183](#)).

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 155](#)).

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.

Create quantification model

Name of the quantification model

Sample lists
Search queries
XDS/DS import

Name	Saved	Reference parameter	Unit
EtOH (1) simulation	2022-02-22 20:38:13	H2O	%
EtOH (2) simulation	2022-02-24 09:42:40		
My sample list	2022-02-17 10:49:44		

 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 177).

 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.

Define reference parameter

Name of the quantification model

Reference parameter	Unit
H2O	%

Name of the reference parameter

H2O

Unit of the reference parameter

%

Decimal places

2

- 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 ² P
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:

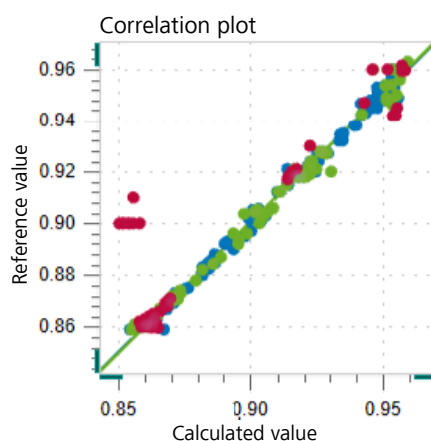
- **SEC**: Standard error of calibration.
- **SECV**: Standard error of cross validation.
- **SEP**: Standard error of prediction. This figure is the best estimate for the prediction error when analyzing unknown samples. The SEP is displayed only if a validation dataset is available.

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.



Each point represents a sample:

- 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.

- 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.



4



- 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*.

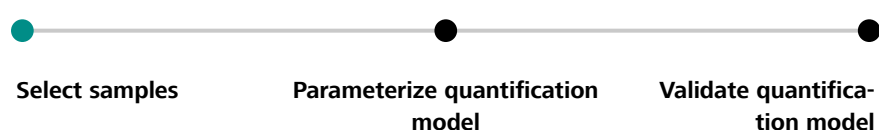
- Select one or more models. For a multiple selection, use the **[SHIFT]** key or the **[CTRL]** key.

- 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 166*)
- Handling the charts (*see "Handling the charts", chapter 11.3, page 167*)

Select samples process step

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

Spectra list

		Sample name	Subsample name	Source	H2O	

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.

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.

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.

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

The **Dataset splitting** dialog opens.

- 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%.

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%.

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:

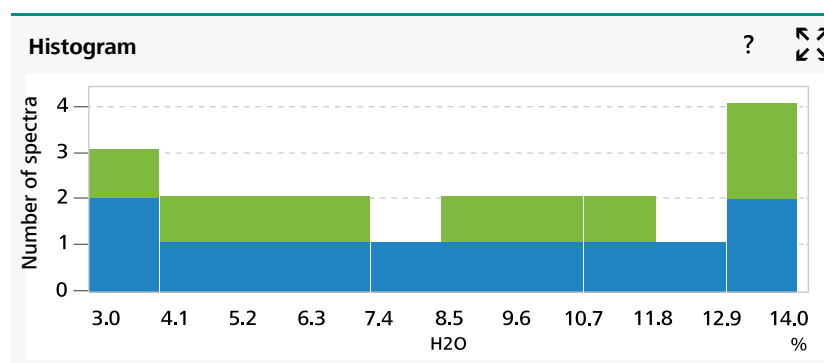
- Optionally, put the spectra in a suitable order. Sort the overview list by clicking on a column header.
- Select multiple spectra by using the **[CTRL]** key or the **[SHIFT]** key.
- Right-click on the selection to open the context menu. Assign the selected spectra.

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.

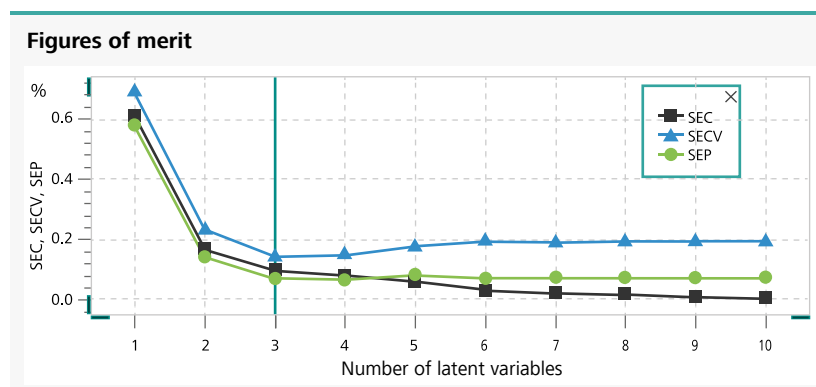
The charts **Loading plot** and **Score plot** can also be displayed by clicking on .

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).



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**.

- The number of selected latent variables is indicated by ✓ in the table.

4

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:

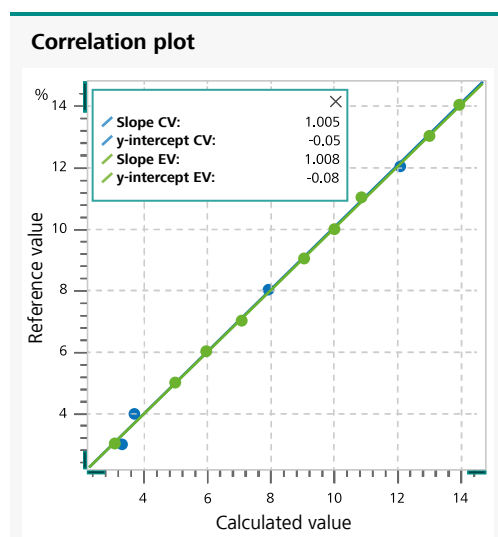
Reference value	Value for the reference parameter
Calculated value	Result of the quantification model
Residual	Difference between calculated value and reference value

 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 167*).

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:

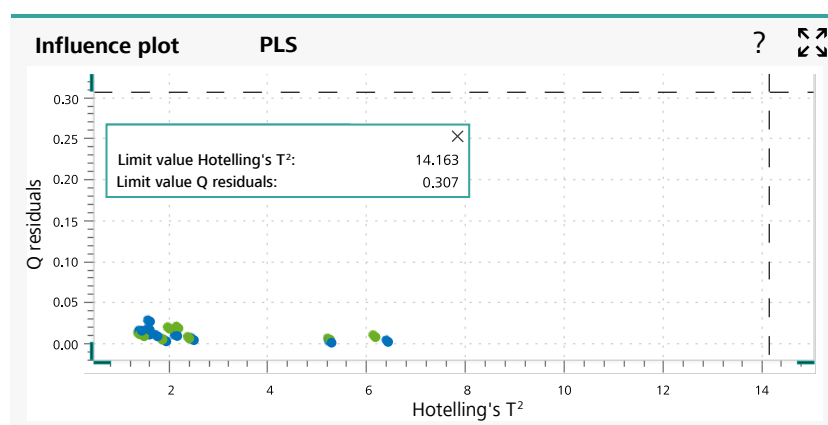
- **PLS** (Partial least squares regression)
PLS uses the relevant information from both the spectra and the reference values. PLS is the basis of the quantification model.
- **PCA** (Principal Component Analysis)
PCA extracts the relevant information from the spectra.

i 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



i 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 167*).

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

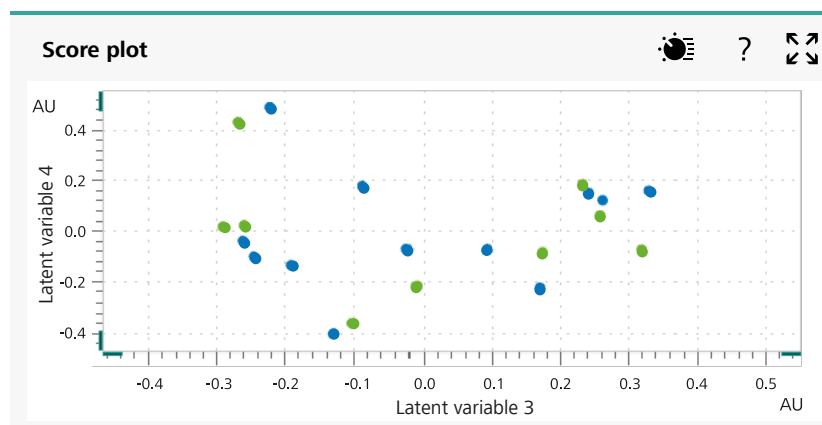
Spectra with high values for Hotellings T^2 indicate an extreme composition of the relevant samples. These samples have a major influence on the model. If the reference value of such a sample is incorrect, then the prediction of similar samples can lead to incorrect results.

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

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 169](#)).
 - 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 166*)
- Handling the charts (*see "Handling the charts", chapter 11.3, page 167*)

3 'Parameterize quantification model' process step

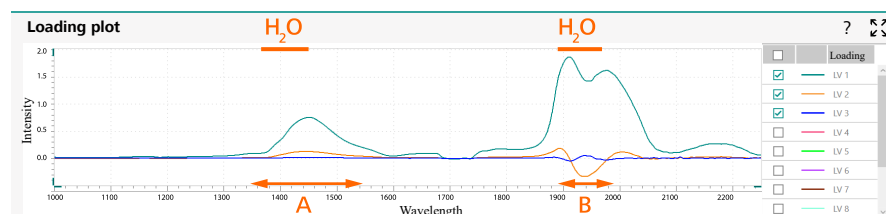
- Click on the **Parameterize quantification model** process step in the navigator.
- In the **Data preprocessing** area, expand the selection list with ▼ and select the **Loading plot** area.

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.

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.

i 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

Wavelength range			?	
#	Start wavelength	End wavelength		
1	1000.0 nm	2250.0 nm		

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

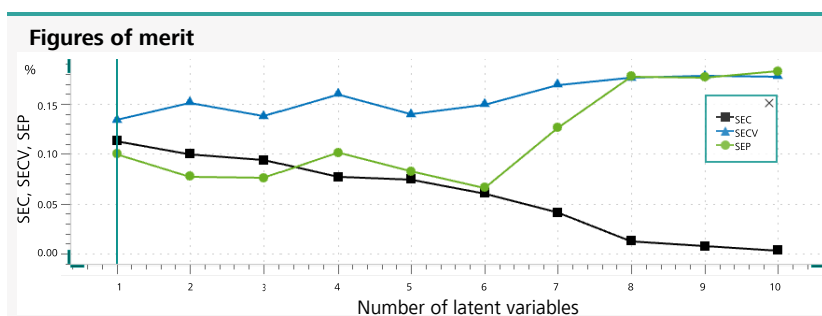
First, a new wavelength range overlaps with the existing wavelength ranges. Adjust the wavelength range so that no overlaps occur.

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

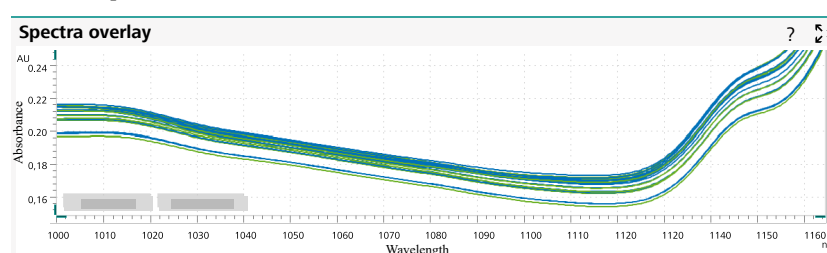
- Click on  or press the **[CTRL]+[S]** keys.

 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.

2 Adding additional data preprocessing steps

Additional data preprocessing steps may be added by clicking on



i 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.

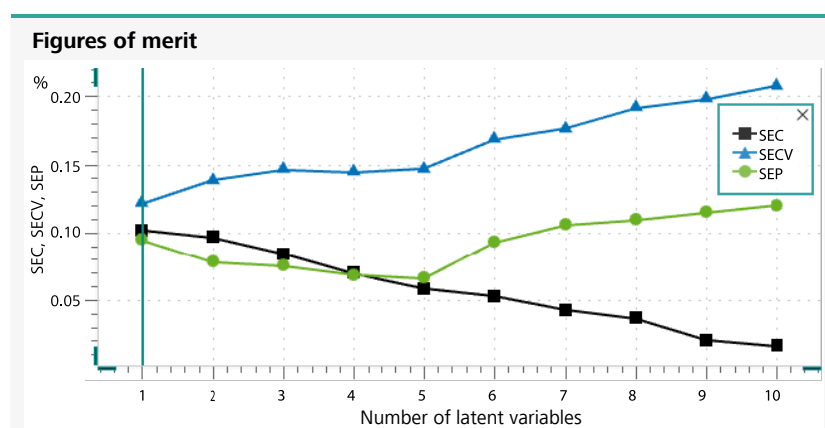
Lines can be moved up or down by clicking on  and , thus defining the sequence.

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.



In the case of H₂O 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.

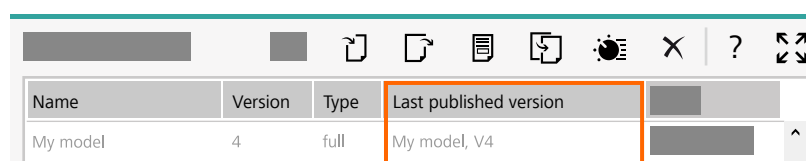
NeighborDistance (NND) **PREDICT** command variable (see "Prediction", chapter 2.3.2, page 21).

The two variables can be compared with one another and monitored using result monitoring (see "Creating an operating procedure", page 150).

3 Publishing

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

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



Name	Version	Type	Last published version
My model	4	full	My model, V4

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 177*).

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.

6 Identification model

i An illustration of the runs in the OMNIS Software can be found in the Appendix ([see "Developing a model", page 183](#)).

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.

Create identification model

Name of the identification model

Sample lists
Search queries
XDS/DS import

Name	Saved	Product	Number of spectra

Product list			?	
	Product	Number of spectra	Product group	

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:

Spectra list						?	
		Sample name	Subsample name	Source	Product		

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:

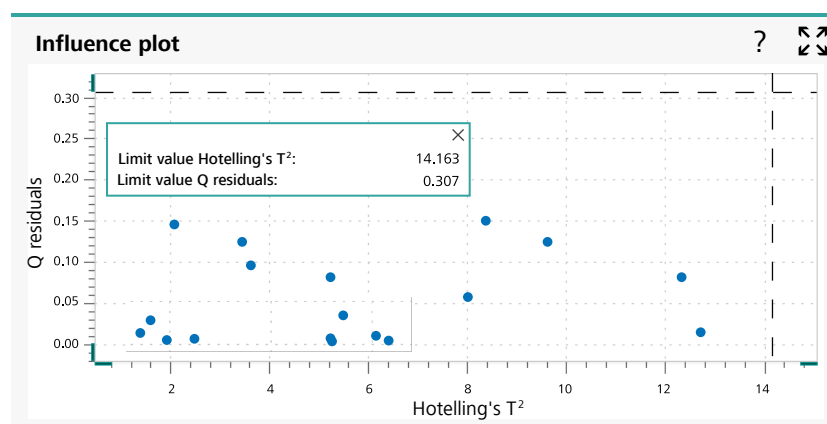
- Spectra in the calibration dataset are marked **blue**, spectra in the validation dataset are marked **green**, and spectra in the outlier dataset are marked **red**.
- If the **Display product colors** toggle switch is activated, then the spectra will be colored according to the product colors.

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

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

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 167*).

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.

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.

i 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 112).

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]**.

i 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.

i 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 135).

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.

i 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 166*)

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 115*).
- Adjust the wavelength ranges (*see "Wavelength selection", chapter 6.5.1, page 114*).

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 135*).

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

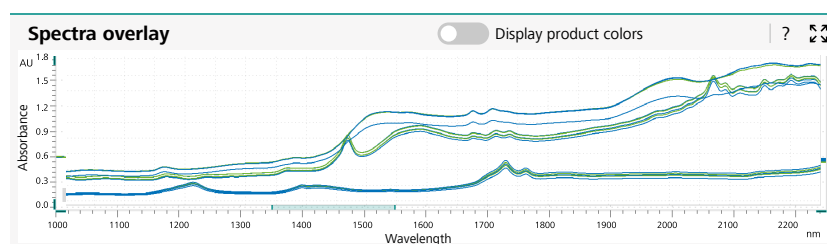
Prerequisite:

- 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 166*)
- Handling the charts (*see "Handling the charts", chapter 11.3, page 167*)

Additional steps

- Wavelength selection (*see "Wavelength selection", chapter 6.5.1, page 114*)
- Defining data preprocessing (*see "Data preprocessing", chapter 6.5.2, page 115*)

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 .

7 Qualification model

i An illustration of the runs in the OMNIS Software can be found in the Appendix ([see "Developing a model", page 183](#)).

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.

Create qualification model

Name of the qualification model

Sample lists

Search queries

XDS/DS import

Calibration dataset

Name	Saved

 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 177*).

 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.

Validation dataset			
Name	Saved	Positive	Negative
		☐	☒
		☐	☒
		☐	☐

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 166*)
- Handling the charts (*see "Handling the charts", chapter 11.3, page 167*)

'Select samples' process step

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

Spectra list						
			Sample name	Subsample name	Source	

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.

- 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

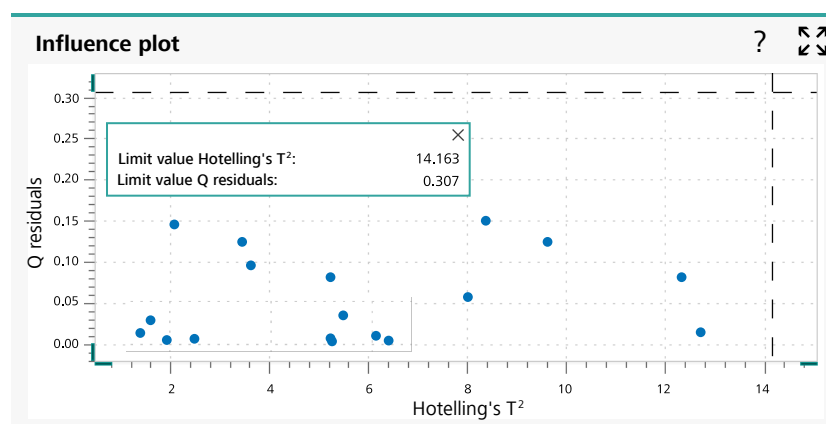
Define options for dataset splitting:

- **Apply parameterization**: Apply data preprocessing and wavelength selection to the spectra (*see "Parameterizing the qualification model", chapter 7.5, page 129*).

Note: Subsequent changes to the parameterization will not affect the dataset assignment. Unless the dataset is to be split again.

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 167](#)).

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.

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 129*).

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

 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]**.

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:

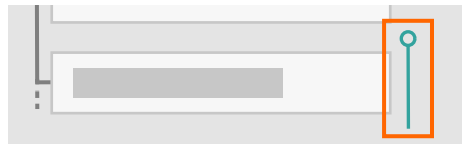
- An **identification** of an unknown sample (e.g., fructose). The result is a product name.
 - Optionally, one or more **quantifications**, depending on the product determined.
- A **verification** of the product membership (e.g., fructose) of a sample. The result is yes or no – Verification successful or failed.
 - Optionally, one or more **quantifications**, depending on the product determined and regardless of the verification result.
- One or more **quantifications**.

- i** 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

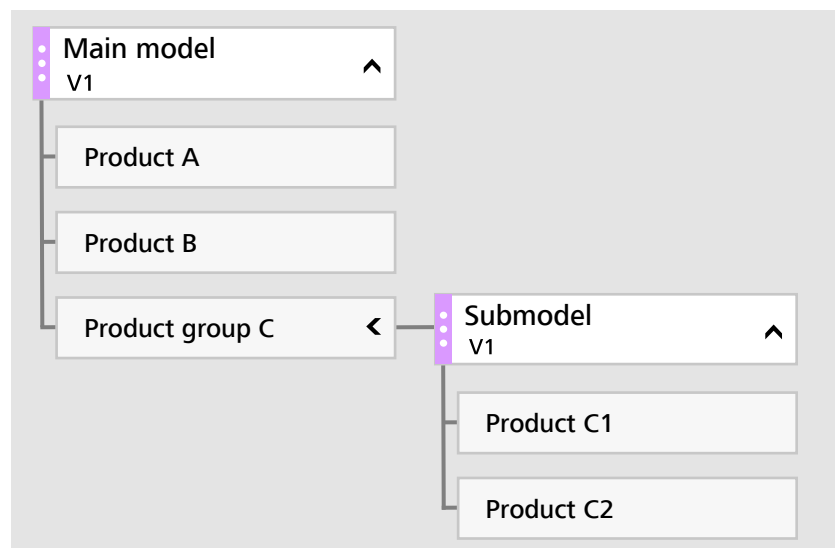
If product groups have been defined in the main model:

- 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.

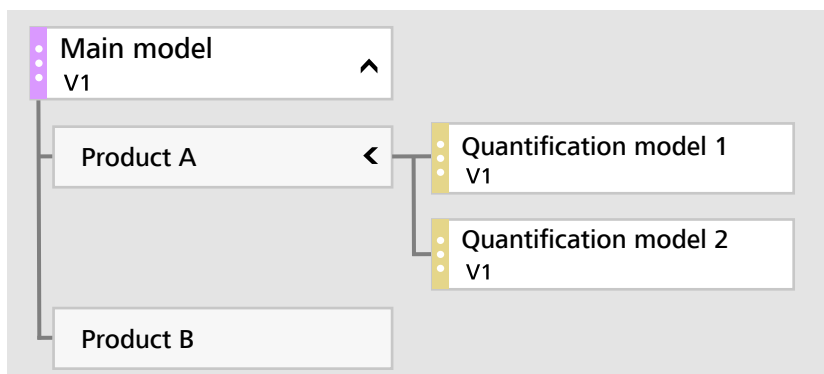


- i** The horizontal arrow is used to collapse and expand the submodel.

- i** 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



i Quantification models can be linked to products or product groups.

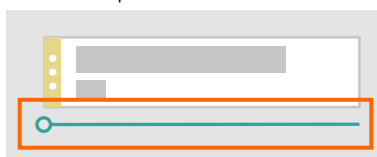
2 Model hierarchy without identification models

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:



- i** 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:

- 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 136*).
- 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 110*).
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**:

Name	Version	Type	Last published version
My model	4	full	My model, V4

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 184*).

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.

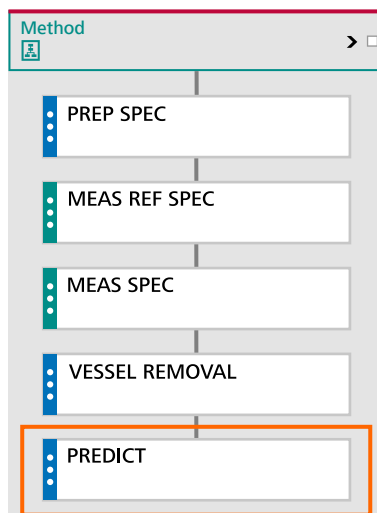
- Open the **Library** window by clicking on .

- 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:

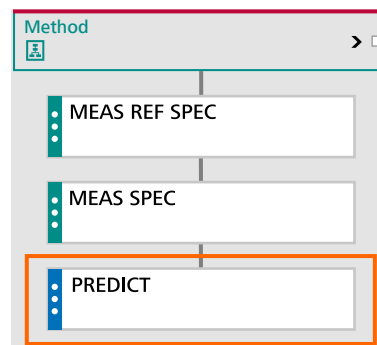
Liquid samples

Basic structure



Solid samples

Basic structure



The **PREDICT** command can also be placed before or next to the **VESSEL REMOVAL** command.

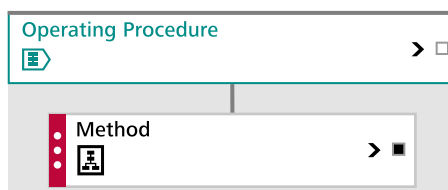
3 Configuring the PREDICT command parameters

- Select the PREDICT command.
- Open the **Properties** window by clicking on .

- 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  **Operating procedure** .
- 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 175)
 - 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 170*).

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.

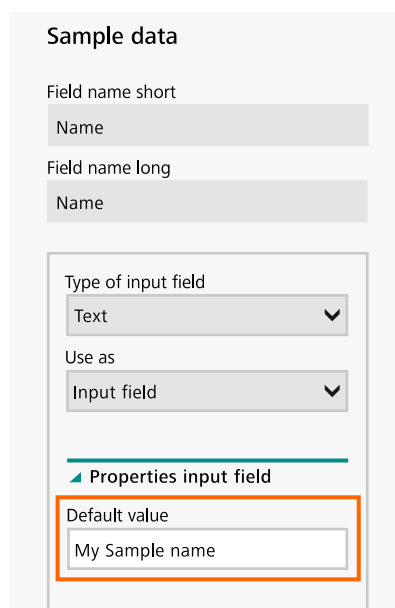
1 Applying and naming the sample profile

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.



Sample data

Field name short
Name

Field name long
Name

Type of input field
Text ▼

Use as
Input field ▼

▲ Properties input field

Default value
My Sample name

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 sub-samples

- In the **Operating procedures / subsamples** area, select the created operating procedure.
- Define the number of subsamples: **1**







Operating procedures / subsamples

	Operating procedure	Number of subsamples
1		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.

Sample list ▾

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.

Sample data

Field name short	Field name short	Field name short
H2O	Methyl acetate	Methanol
Field name long	Field name long	Field name long

Type of input field

Number

Use as

Input field

Properties input field

Default value

Minimum value

Maximum value

Unit

%

☒ Field editable

☒ Allow empty field

☐ Force entry

Type of input field

Number

Use as

Input field

Properties input field

Default value

Minimum value

Maximum value

Unit

%

☒ Field editable

☒ Allow empty field

☐ Force entry

Type of input field

Number

Use as

Input field

Properties input field

Default value

Minimum value

Maximum value

Unit

%

☒ Field editable

☒ Allow empty field

☐ Force entry

The sample list contains an input field for each reference parameter.

Sample name	H2O	Methyl acetate	Methanol
	%	%	%
	%	%	%
	%	%	%
	%	%	%

- **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:

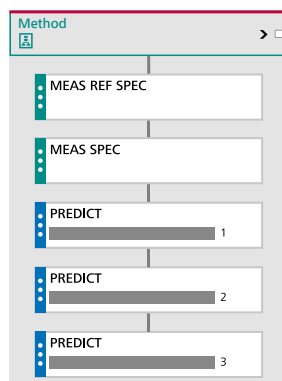
- Create model hierarchy (see *"Inserting models in a model hierarchy"*, chapter 8.1.2, page 137).

- Insert quantification models in the model hierarchy (see *"Inserting models in a model hierarchy", chapter 8.1.2, page 137*).
- 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 148*).
- 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

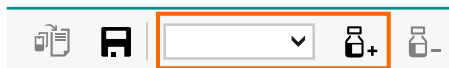
For a verification, the product for which the sample is verified must be defined in the sample data. The product input field must be created for use as a product. Not case-sensitive.

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 160).

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			4.7		
2			6.1		
3			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.

i Prediction results can also be displayed in the subsample data if required (see "*PREDICT command variables*", chapter 11.4, page 170).

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.

- [illegible]

- ## 10.2 Wavelength calibration

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

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.

If reference standards are used for external instrument performance tests, then these standards must be recertified periodically.

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- 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.

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.

- 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:

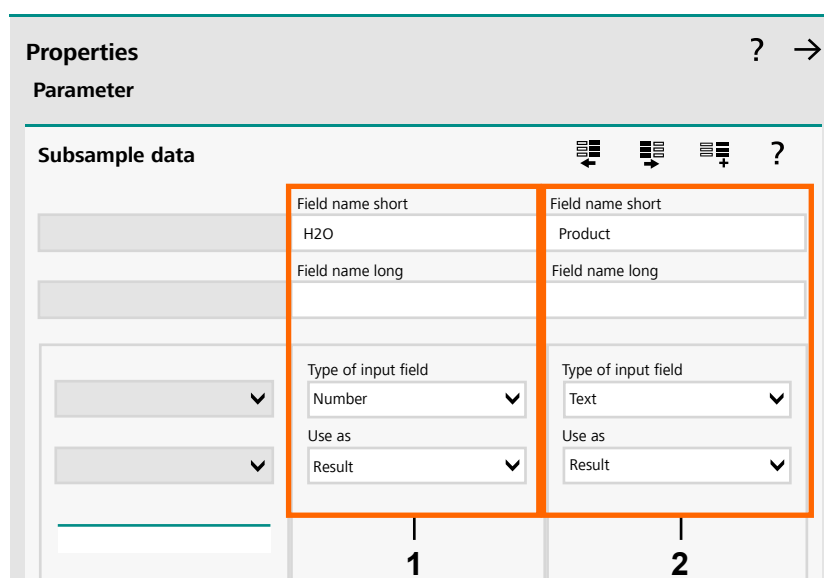
A method and an operating procedure have been created to prepare the prediction (see *"Preparing the prediction", chapter 9.1, page 148*).

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):



Subsample data	
Field name short	Field name short
H2O	Product
Field name long	Field name long
Type of input field	Type of input field
Number	Text
Use as	Use as
Result	Result
1	2

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

- 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 175)
- 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.

Variables ?

.CurrentSubsampleData X

Variables category Subcategory

Subsample data ▼

▼

Enter search term 🔍

Search result

.CurrentSubsampleDate

.CurrentSubsampleData

Apply
Cancel

- 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:

Sample name	Subsample name	

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.

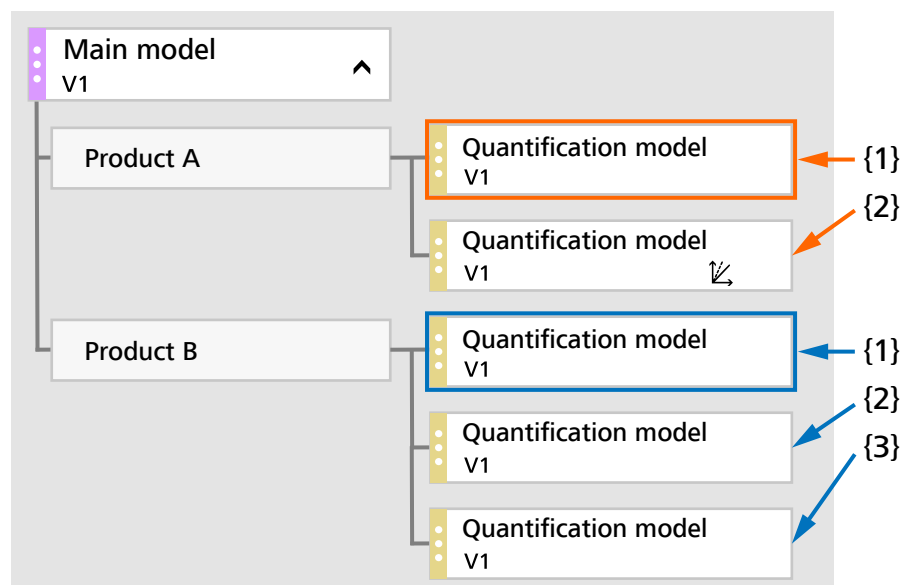


Figure 7 Index numbers for quantification models in a model hierarchy

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.

- 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.

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 148*). 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 157).

 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.

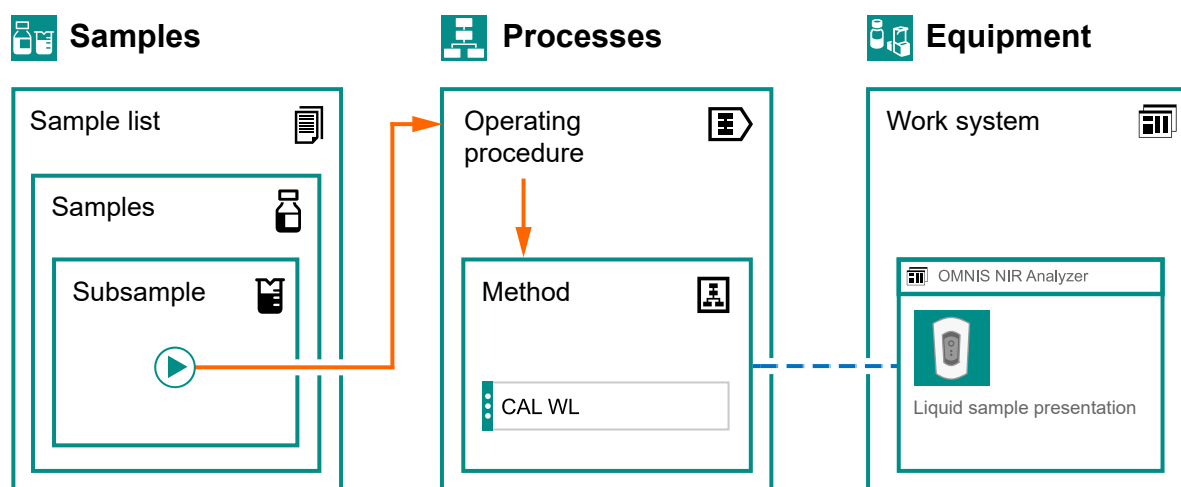


Figure 8 Instrument setup



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.



Samples

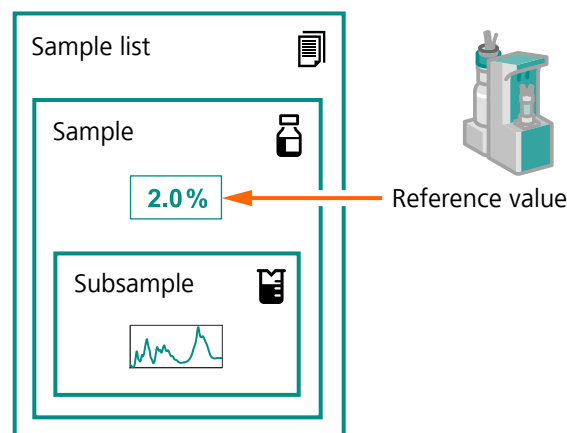


Figure 9 Record a reference value

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.

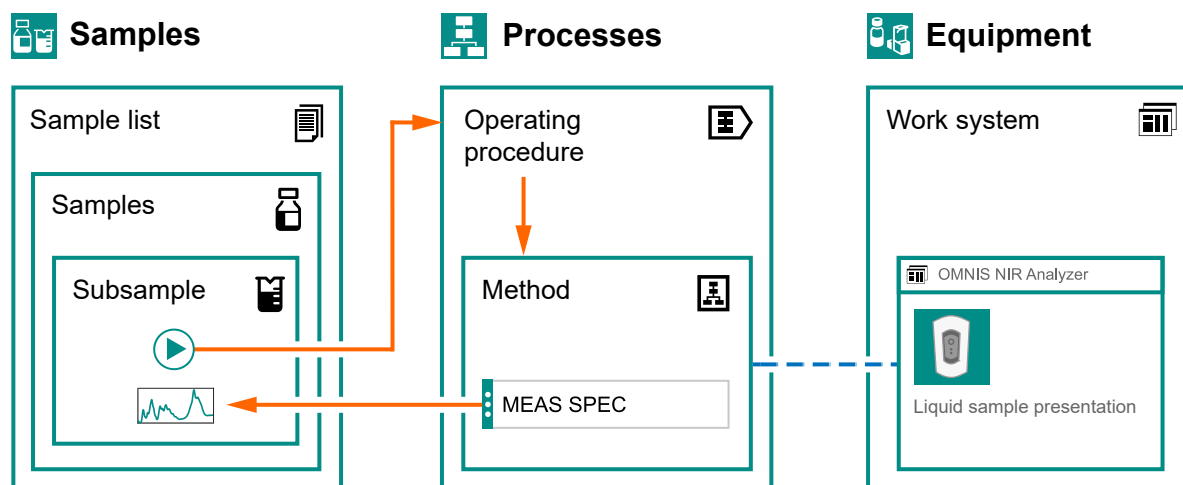


Figure 10 Acquiring a spectrum of a calibration sample



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.

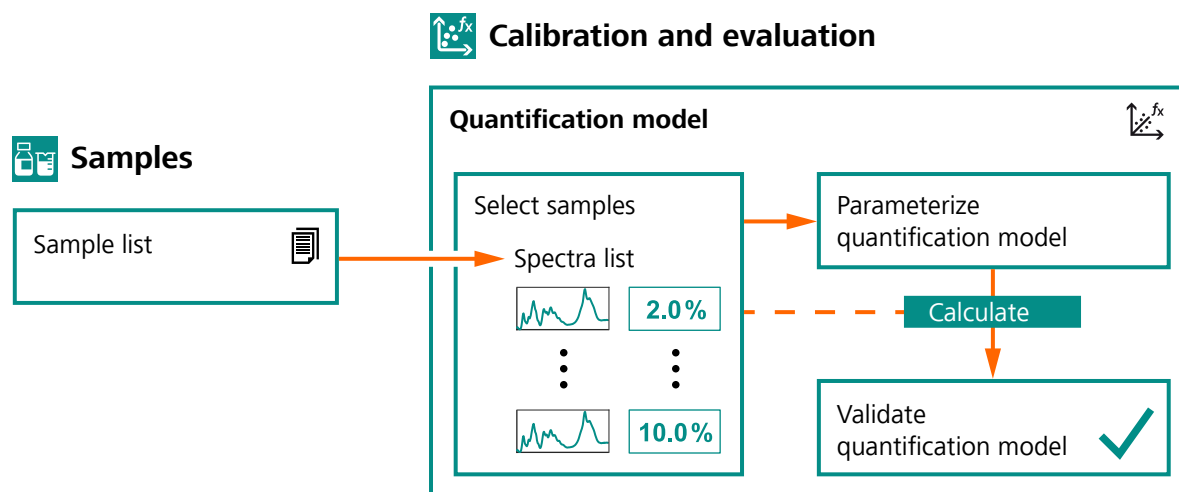


Figure 11 Developing a model (example for quantification)

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.

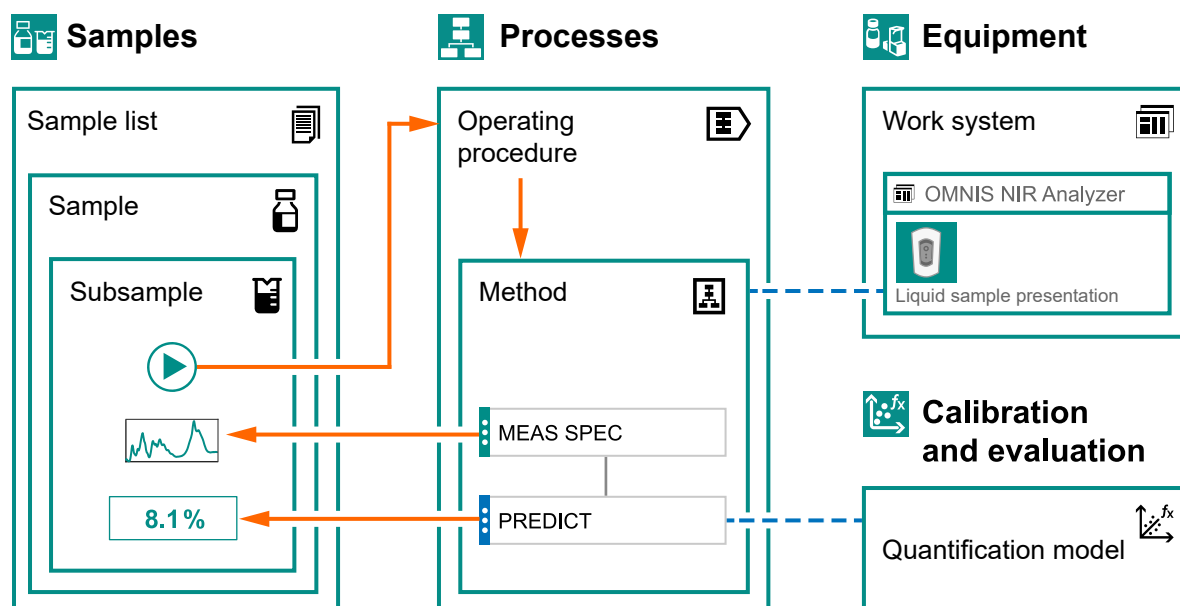


Figure 12 Predicting parameters of interest (example for quantification)

	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%.