V20/V30

Karl Fischer Titrators

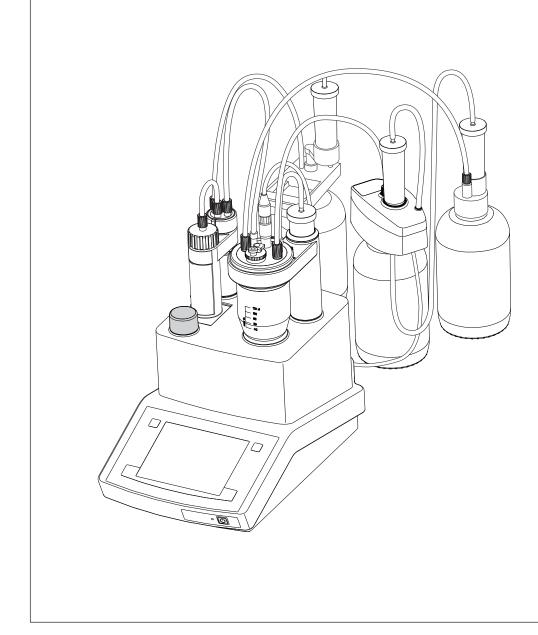




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1 Introduction

Simple and compact

The devices in METTLER TOLEDO's Titration Compact Line are modern, compact titrators for use in a wide variety of application areas. They can be used, for example, in quality control as well as in research and development and satisfy the most demanding of requirements.

The titrators in the Titration Compact Line perfectly combine simple, easy-to-understand operation with an extremely high level of precision and outstanding reliability. Thanks to automatic titrant recognition (Plug & Play burettes), the titrator independently identifies which titrant is required without intervention from the operator. Settings no longer need to be adjusted manually, even when connecting a printer or a Stromboli oven sample changer.

Titration Compact devices can either be controlled by touchscreen or by using LabX PC software. The large color touchscreen allows intuitive control by the user and flexibility in its adjustment options. All functions can be activated directly from the home screen via shortcuts which can be freely created, making everyday use extremely easier. The touchscreen control of the titrator and all adjustable parameters are described in detail in the operating instructions.

The separate installation information explains all the necessary steps for installing and commissioning your device. The enclosed "Quick Guide" then guides you through the first titration using a practical example. If you have any additional questions, METTLER TOLEDO is always available to assist you.

2 Safety Notes

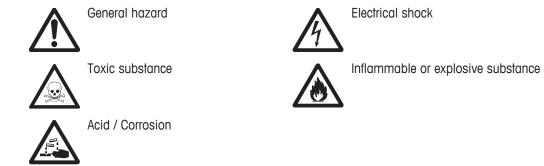
2.1 Definition of Signal Warnings and Symbols

Safety notes are marked with signal words and warning symbols. These show safety issues and warnings. Ignoring the safety notes may lead to personal injury, damage to the instrument, malfunctions and false results.

Signal words

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	WARNING	for a hazardous situation with medium risk, possibly resulting in severe injuries or death if not avoided.
	CAUTION	for a hazardous situation with low risk, resulting in damage to the device or the property or in loss of data, or minor or medium injuries if not avoided.
	Attention	(no symbol) for important information about the product.
	Note	(no symbol) for useful information about the product.

Warning symbols



2.2 Product Specific Safety Notes

Your instrument represents state-of-the-art technology and complies with all recognized safety rules, however, certain hazards may arise in extraneous circumstances. Do not open the housing of the instrument; it does not contain any parts that can be maintained, repaired or replaced by the user. If you ever have problems with your instrument, contact your authorized METTLER TOLEDO dealer or service representative.

Intended use



This instrument is designed to be used in analytical laboratories and is suitable for the processing of reagents and solvents.

The use therefore requires knowledge and experience in working with toxic and caustic substances as well as knowledge and experience working with application-specific reagents, which may be toxic or hazardous.

The manufacturer shall not be held liable for any damage resulting from incorrect usage divergent to the operating instructions. Furthermore, the manufacturer's technical specifications and limits must be adhered to at all times and in no way exceeded.

Location



The instrument has been developed for indoor operation and may not be used in explosive environments.

Place the instrument in a location which is suitable for the operation, protected from direct sunlight and corrosive gases. Avoid powerful vibrations, excessive temperature fluctuations and temperatures below 5 °C and above 40 °C.

Protective Clothing

It is advisable to wear protective clothing in the laboratory when working with hazardous or toxic substances.

A lab coat should be worn.



Suitable eye protection such as goggles should be worn.



Use appropriate gloves when handling chemicals or hazardous substances, checking their integrity before use.

Safety notes



WARNING



Use only 3-pin grounded electrical outlet and extension cables to connect the instrument.

- a) Only 3-pin grounded electrical outlet and extension cables for connecting your instrument must be used.
- b) Intentional disconnection of the equipment grounding conductor is prohibited.



WARNING

Risk of corrosion

Leaks in tubing connections and loose titration vessels are a safety risk.

- a) Tighten all connections well by hand, avoid applying excessive force to tubing connections.
- b) Always test the titration vessel for firm seating in the titration head.



WARNING

Flammable solvents

All relevant safety measures must be observed when working with flammable solvents and chemicals.

- a) Keep all sources of flame away from the workplace.
- b) When using chemicals and solvents, comply with the instructions of the producer and the general lab safety rules.

WARNING



Chemicals

All relevant safety measures are to be observed when working with chemicals.

- a) Set up the instrument in a well-ventilated location.
- b) Any spills should be wiped off immediately.
- c) When using chemicals and solvents, comply with the instructions of the producer and the general lab safety rules.

3 Karl Fischer Water Determination - Measuring Principle

The Karl Fischer procedure is a titration method used for the quantitative determination of water content in liquids and solids. Karl Fischer titration is used in a variety of areas, e.g. for determining the water content of groceries, chemicals, pharmaceuticals, cosmetics and mineral oils.

To determine the water content, first sulfur dioxide and water react with iodine:

 $2 H_2 O + SO_2 + I_2 \rightarrow SO_4^{2-} + 2 I^- + 4 H^+$

The addition of alcohol (e.g. methanol, ethanol), causes a preliminary reaction to take place in which sulfur dioxide forms an acidic ester, which is then neutralized by the addition of a base (e.g. imidazole, referred to in the following as "RN"):

 $CH_3OH + SO_2 + RN \rightarrow (RNH) \cdot (CH_3OSO_2)$

In the presence of water, the alkyl sulfite anion is oxidized to alkyl sulfate by the iodine. This process reduces the yellow-brown iodine to colorless iodide:

 $(\mathsf{RNH}) \cdot (\mathsf{CH}_3\mathsf{OSO}_2) + \mathsf{I}_2 + \mathsf{H}_2\mathsf{O} + 2 \ \mathsf{RN} \rightarrow (\mathsf{RNH}) \cdot (\mathsf{CH}_3\mathsf{OSO}_3) + 2 \ (\mathsf{RNH}) \cdot \mathsf{I}$

The overall reaction proceeds as follows:

 $H_2O + I_2 + SO_2 + CH_3OH + 3 RN \rightarrow [RNH]SO_4CH_3 + 2 [RNH]I$

The reaction runs until all the water has been consumed and hence free iodine is detected in the titration solution. The end point is determined using bivoltametric indication, i.e. the potential at the polarized double-platinum-pin electrode falls below a certain value (e.g. 100mV).

3.1 Volumetric water content determination

In Karl Fischer (KF) volumetric determination, a titrant containing iodine is gradually added to the water-containing sample until the water is completely displaced and free iodine can be detected in the titration solution. The end point of the titration is recorded using bivoltametric indication. Volumetric Karl Fischer titration is suitable for samples with a water content in the range 100 ppm to 100 %. The optimum recording range is 10mg of water per sample.

For optimal execution of the KF titration, the pH of the solution should be in the range between 4 and 8. Acidic and basic samples should be buffered, ideally with imidazole for acids and salicylic acid for basic samples.

The following two conventional reagents are used for titration:

a) The single-component reagent

The titrant consists of iodine, sulfur dioxide and imidazole. The solvent is methanol.

The single-component reagent is simple to use and cost efficient. However, it is not stable for titration.

b) The two-component reagent

The titrant is an iodine solution containing methanol. The solvent for the sample contains sulfur dioxide and imidazole dissolved in methanol.

The two-component system can be used to perform very fast titrations (two to three times quicker than with the single-component reagent). Both components can be easily stored. The reagent is stable for titration, however, the solvent capacity is limited.

4 Description of Functions

4.1 Layout of the Terminal

The control panel of the terminal consists of an integrated touchscreen and the following buttons, located next to the touch-sensitive surface of the display:

- The Reset button ends all tasks that are currently running.
- The Info button accesses the interactive online help for the content of the current dialog.
- Two Home buttons always return you to the homescreen.

You can press these buttons any time, regardless of which dialog you are currently using.

i The **Reset button** acts as an "EMERGENCY STOP" switch. If the titrator malfunctions or there is an operating error, you can stop all current tasks by pressing the reset button. Afterward, for each task, you can decide whether to end it completely or continue.

4.2 Operating the Touchscreen

The touchscreen is automatically activated when the instrument is switched on.

To select a button or an input element in the dialog window, you simply touch the screen using a soft blunt object or a fingertip.

It is also possible to select input elements using a USB mouse. To do this, simply connect the mouse to a suitable USB port on the instrument.

Never touch the surface of the touchscreen with pointed or sharp objects! This may damage the screen!

4.3 The Homescreen

The homescreen is the first screen that is displayed when you start up the titrator.

The homescreen contains five buttons that lead to the following dialog windows:

- Methods: This button takes you to the method editor, where you can create and manage methods.
- Series templates: In this dialog, you can create and manage series of individual samples, e.g. for using a sample changer.
- Results: You manage the results of your analyses here.
- Setup: The hardware and resources the titrator uses are configured in the Setup. You can also make global and user settings here.
- **Manual**: This button takes you to manual operations. You can operate stirrers, sensors, pumps, etc. here, independently of the analyses.

In addition, there is another area that can be configured individually by each user (with the necessary authorization). Each user can store up to 12 shortcuts here. You can use this shortcut to launch defined methods, series, and manual operations directly from the homescreen.

By tapping the **Home** key in the terminal control panel, you can return to the homescreen from any screen.

See also

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• Shortcuts and Direct Shortcuts (page 12)

4.4 The User Interface

The graphical user interface consists of the following five basic elements:

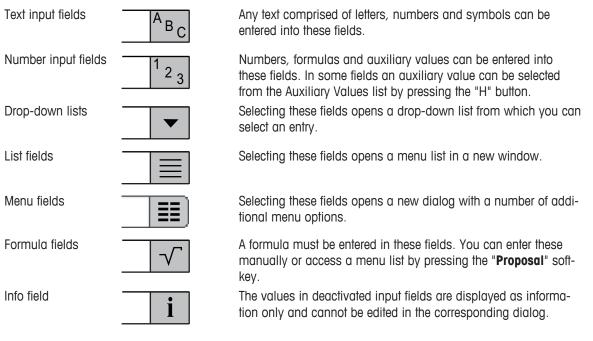
- The title bar at the top of the display specifies the name of the current dialog.
- In the top right-hand corner, the Tasks button informs you which processes are currently running. You use Tasks to access a Tasks dialog that displays an overview of all running tasks. From the Tasks dialog you can navigate to any process that is currently running.
- The navigation bar, located below the title bar, specifies the path to the current dialog.

- The **scroll bar** on the right-hand side of the screen becomes visible if the content of the screen extends beyond the viewable area. If this occurs, use either the arrows or the area in between them to move the viewable area of the screen up or down.
- Five **buttons** are located at the bottom of the screen. The function of these buttons varies and depends on the context of the current dialog.

4.4.1 Entering Data in the User Interface.

There are different types of input fields in the user interface. They allow you to enter data or select data from a list. Input fields can also be deactivated and their contents are then displayed as information only and cannot be changed in the corresponding dialog.

The various types of input fields are identified by an icon to the right of the screen:



In addition to the input fields there are checkboxes that can be checked in order to select certain functionalities. Checkboxes can affect the scope of the corresponding dialog, i.e. input fields can be hidden or visible depending on whether the checkbox is checked.

Sorting Lists

All of the lists displayed in the user interface can be sorted alphabetically or numerically by column in ascending or descending order. To do this, simply touch the parameter in the header row by which you would like to sort the list. A small arrow in the header row indicates the parameter by which the list is sorted and whether it is sorted in ascending or descending order.

Dialog window: Tasks (V30 only)

The **Tasks** dialog is accessed using the Tasks button (on the top right of the screen). It provides an overview of all running tasks. Selecting an individual task from the list displays the relevant online dialog box, from where you can exit the task.

The list contains a maximum of 10 running tasks.

4.4.2 Shortcuts and Direct Shortcuts

Shortcuts allow you to start methods, series, and manual operations directly from the homescreen. You can place up to twelve different shortcuts on the homescreen by tapping **AddToHome**. **AddToHome** is located in the start dialog of each method, series and manual operation.

Shortcuts are user-specific, i.e. each individual user can create a maximum of twelve shortcuts for the tasks they personally conduct the most with the titrator.

The titrator supports two types of shortcuts. Direct shortcuts which, when selected, start the task immediately without warning (only if the other settings allow this), and normal shortcuts which take you to the corresponding start dialog from which you can start the task.



Shortcuts for methods, series or manual operations that take you to the corresponding start dialog.

Shortcuts for methods, series or manual operations with integrated reference symbols in the icon that start the corresponding task when selected provided the other settings allow for it.

Shortcuts are managed in **Setup** > **User settings**. Here you can delete or modify shortcuts, or change their position on the homescreen (see "User settings: Shortcuts").

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Tasks started using the shortcuts can begin immediately without warning. Therefore, always make sure that all tubes are connected to suitable vessels prior to using a shortcut.

• Once the maximum number of shortcuts (12) has been created in the Homescreen, the button **AddToHome** is deactivated in the start dialog for methods, series and manual operations.

4.4.3 The Start analysis dialog

There are several different ways to start an analysis on the titrator:

- By choosing Start from the Method editor.
- By choosing **Start** from the **Home** dialog.
- By using a shortcut (or direct shortcut) from the **Home** dialog.
- By choosing Start from the Series dialog.

The **Start analysis** dialog is always the first dialog that appears after you choose "**Start**" or the relevant shortcut.

When a direct shortcut is activated, the **Start analysis** dialog does not appear and the respective method starts immediately, provided that the other settings allow this.

The parameters for the previously used method or series appear in the **Start analysis** dialog so that the same method can immediately be restarted.

Of course, all of the settings can also be adjusted prior to pressing "Start" The type and number of settings displayed in the Start analysis dialog depends on the type of analysis to be started and the resources used.

4.4.4 Online Dialog

The Online screen is displayed when an analysis or manual operation is being performed.

The method ID of the current method or the type of manual operation is displayed in the title bar. In the navigation bar below, the sample index, e.g. displayed as "Sample 2/5" (second of a total of five samples) and loop index, displayed as "Loop 1/3" (first of three loops) are shown. (The Loop index is only displayed if the method actually contains more than on loop). The navigation path is displayed in the navigation bar while a manual operation is being performed. The remainder of the online dialog is divided into a graphical area (left) and a data area (right). During a titration or measurement, the graphical area displays the measurement curve.

4.4.4.1 Pretitration

Immediately following the start of a Karl Fischer titration, the online window for pretitration appears. In this window, the following buttons are available:

Results

In **Results**, the results and statistics for the samples to be analyzed following the analysis are displayed. The system displays the results of the active determination type (sample, concentration, blank value). Furthermore, the screen contains the following buttons:

- Add result
- Recalculate
- Undo all
- Outlier test

Samples

You can change sample and series data. However, the number of samples cannot be changed while a concentration or blank value determination is in progress.

More

The **More** button provides you with additional functions. Using the More button in **Pretitration** mode, you can perform the following:

End series

You can end a series if all predefined samples have been processed. Any changes made in the **Start analysis** dialog or later are no longer taken into account. After the series has ended, you return to the pretitration or standby mode and the series can be restarted again. A new series is entered in the results. The system then uses the original sample parameters.



• The End series function triggers printouts defined Per series.

Stop method

The current method is stopped immediately. No printout is generated.

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Before actually stopping the process, the system displays a system message asking you to confirm the action.

Save series data

The analysis of a series is saved in its entirety under a name freely chosen by the titrator in the form "SeriesXY". Only sample data is included in the series. Standard data and blank value data are not included in the generated series. If the maximum number of permitted series has already been reached, the series is not saved.

Axes

You can select the units for the horizontal and vertical axes from a list.

Sample size calculation

The optimum sample size can be calculated from the standby of an analysis.

The determined limits for the sample size do not have any impact on the lower and upper limits in the method or for the sample data memory.

You can determine the following parameters:

Parameters Description		Values
Content	Expected water content of the sample.	0106
Unit	Unit for the content.	[%] I [ppm]

Use the **Calculate** button to obtain the upper and lower sample size limits for optimum titration.

4.4.4.2 Standby

If the system switches automatically from **Pretitration** to **Standby** mode (see "KF analysis flow diagram"), you can use the **More** button to view additional buttons:

Drift determination

You have to add at least one titrant increment for a drift determination. When the determination has been completed successfully, the determined drift value is entered in the setup of the titration stand. The system then generates an automatic printout containing the sample data, raw results, and resource data.



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- The message No titrant added / generated. Drift not determined. can be confirmed, or the message disappears after a certain period of time (60s).
- If the drift falls below a defined value, the system automatically switches to **Standby** mode.
- The sample size should be selected so that between 30 % and 70 % of the burette volume can be titrated.

In **Standby** mode, you can conduct concentration determination for the titrant, start sample analysis, or conduct blank value determination for the "external extraction" method type. The following buttons are available for this:

Concentration Determination

You use this button to determine the concentration of the titrant. No predispensing is performed. The determined concentration or the mean value for a series of concentration determinations is entered in the Setup for the relevant titrant, if this falls within the limits. If the mean value falls outside the specified limits, this is not transferred to the Setup, but the system still switches to Standby. After the concentration has been determined successfully, the user receives a printout. If the value is not transferred to Setup, the system issues a message to inform you of this.

Tapping the **Start conc.** button opens the **Concentration sample** window. You can enter a comment and the temperature. When you tap **OK**, an **Info** dialog is displayed as a prompt to add the standard.

Start blank determination

Blank value determination can be performed for the method type **Ext. Extraction**. No predispensing is performed. The determined blank value or the mean value for a series of blank determinations is entered in the Setup for the relevant titrant, if this falls within the limits. If the mean value falls outside the specified limits, this is not transferred to the **Setup**, but the system still switches to **Standby**. After the blank value has been determined successfully, you receive a printout. If the value is not transferred to **Setup**, the system issues a message to inform you of this. When you tap this button, an **Info** dialog is displayed prompting you to add the sample.

Start sample

This button is used to perform a sample analysis. When you press this button, an **Info** dialog is displayed prompting you to add the sample.

Once a sample has been added and the analysis started, you can use the **Samples** button to enter the sample size (see Method Function: **Sample (KF)** > **Sample**.

Measured values

You can use the **More** and **Measured values** buttons to display a table of measured values during an analysis as an alternative to the online dialog.

Samples

You can use this button to change the sample size of the sample currently being processed or to define the sample size for a new sample.

Stop analysis

You can use this button to cancel the measurement immediately during a sample, concentration, or blank value determination.

Before actually stopping the process, the system displays a message asking you to confirm the action.

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5 Setup

This section tells you how to set up the titrator in accordance with your requirements so that you can carry out titration.

Chemicals	Titrant
	Concentration standards
Hardware	Sensors
	Pumps
	Peripherals
	Titration Stands
	Homogenizer
User settings	Language
	Screen
	Веер
	Shortcuts
	Keyboard
Global settings	System
	User management
	Analysis and resources behavior
	Solvent Control
Values	Blank
	Auxiliary values
Mainten. & Service	MT-Service
	Import / Export
	Reset to factory settings
	Titrator firmware history
	Board firmware
	Terminal
	Board data
	Drives
	Burettes
	Update

Expired Resources

Navigation: Home > Setup

Resources for which monitoring was selected in the settings can expire. Tap [**Expired Resources**] to open an overview of all expired resources with the type, name and date of expiry of the respective resource.

5.1 Chemicals

You can use the **Chemicals** dialog to configure and manage titrants and concentration standards. You can view and print out lists of chemicals that have already been defined. You can also define new chemicals or delete existing chemicals.

Titrants are managed together with burettes and burette drive (PnP with chip and traditional burettes without chips).

For classical burettes, the relevant titrant data is entered manually. For PnP (Plug&Play) burettes, the data is automatically read from the chip and automatically transferred to the instrument. If the chip is still blank, the data must be entered in **Setup** or assigned to a titrant. The data is saved in both the titrator and in the chip.

Adding a titrant

- In Titrants choose [New].

 \Rightarrow The windows to edit the parameters opens.

Define the following parameters for each titrant here:

5.1.1 Titrant

Parameters	Description	Values
Name	Specify a descriptive name of your choice.	Arbitrary
Reagent type The type of Karl Fischer titrant can be selected. This Influences the control behavior of a titration.		1-comp 2-comp
Nominal conc.	Specified concentration of the Karl Fischer titrant in [mg/mL].	0.1100
Current conc.	Actual concentration of the Karl Fischer titrant in [mg/mL].	0.1100
Monitoring usable life	Specifies whether the usable life of a resource or a value is to be monitored.	Yes No
Monitoring life span	Specifies whether the life span of the resource is to be monitored.	Yes No
Lot/Batch	The lot or batch of the reagent. Enter any designation.	Arbitrary
Fill rate	The filling rate of the burette in percent. 100% stands for maximum filling rate.	30100
Burette volume	Select the burette volume in [mL].	1 5 10 20
Drive	Defines the drive on which you will use the burette containing the titrant. Select the "PnP" entry for available but unused PnP burettes.	18 PnP
Serial number	The serial number of the relevant device type.	Arbitrary

• Titrants (independently of the type) must each be assigned to a drive.

• In PnP burettes, the serial number is entered automatically. These can, however, be changed.

5.1.2 Concentration standards

Navigation: Setup>Chemicals>Concentration standards

The concentration standards required for determining the concentration of the titrant you use can also be saved and managed in the titrator.

To define a new concentration standard, proceed as follows:

Select the New button in the Concentration standards dialog window to open the Concentration standard parameters dialog.

You can	define tl	he followina	parameters	for each	concentration standard:	
rou ourr		no ionowing	paramoioro			•

Parameters	Description	Values
Name	Specify a descriptive name of your choice.	Arbitrary
Water content	The water content of a Karl Fischer standard.	0.00001106
Unit	Unit for the water content of the Karl Fischer standard.	mg/g mg/mL % ppm mg/piece
Density	The density of a liquid standard, in [g/mL]. Only for Type = liquid or KF .	0.0001100
Lot/Batch	The lot or batch of the reagent. Enter any designation.	Arbitrary
Monitoring usable life	Specifies whether the usable life of a resource or a value is to be monitored.	Yes No

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• All fields except for "Lot/Batch" must be filled before the standard can be saved.

• A maximum of 50 concentration standards can be defined in the device.

The following Karl Fischer standards are predefined:

1. Water standards in [mg/g] :

0.1

1.00

10.0

2. Sodium tartrate dihydrate 15.66%

3. For solid samples: Water standard for KF oven 5.55 %

5.2 Hardware

Navigation: Setup>Hardware

In this dialog window you can configure all the hardware components connected to the titrator, such as:

- Sensors
- Pump
- Peripherals (instruments such as printers or balances)
- Titration stands (KF stand and additional Stromboli TTL for V30)
- Homogenizer TTL (V30 only)

5.2.1 Sensors

Navigation: Setup > Hardware > Sensors

In this dialog you can configure and manage the sensors to be used with the titrator. A polarized sensor is used for the Karl Fischer titration. The unit of measure can be set to [mV] or $[\mu A]$.

To create a new sensor in the titrator, open the **Sensor parameters** dialog using the **New** button in the **Sensors** dialog. You can determine the following parameters:

Parameters	Description	Values
Name Specify a descriptive name of your choice.		Arbitrary
Serial number	The serial number of the relevant device type.	Arbitrary
Monitoring life	Specifies whether the life span of the resource is to be monitored.	Yes I No
span		

• If a PnP sensor is connected to the sensor input, this automatically generates an entry in the setup. The titrator updates all the details (sensor name, type or inputs).

5.2.2 Pump

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Navigation: Setup>Hardware>Pumps

The Solvent manager is predefined in the Setup and cannot be configured individually. A connected Solvent manager generates a corresponding entry, which is displayed in the **Pumps** dialog. The parameters "Type", "Name" and "Pump output" are displayed in the information fields in the **Pump parameters** window.

5.2.3 Peripherals

Navigation: Home > Setup > Hardware > Peripherals

These settings encompass all input and output devices that belong to the titrator environment but that are not essential instruments for processing an analysis (peripherals cannot be accessed in methods). The computer also counts as a peripheral device. The list of all peripheral instruments defined in the titrator, together with the parameters of each individual instrument can be printed out by a printer.

5.2.3.1 Balance

Navigation: Home > Setup > Hardware > Peripherals > Balance

Before defining a balance, you need to select the balance type. The titrator supports the following types of balance:

Balance type	Supported balances	
Mettler	AB PB PB-S AB-S PB-E AB-E College-S SB CB GB College-B HB AG PG PG-S SG HG XP XS XA XPE XSE XVE AX MX	
	UMX PR SR HR AT MT UMT PM AM SM CM MS ML	
Sartorius	Sartorius	
More		

METTLER TOLEDO Balances

These balances support Plug'n'Play and are automatically recognized and configured by the titrator.

For automatic balance recognition, you need to ensure the following:

- 1. The balance has been started up and is connected to the titrator by a suitable cable,
- 2. The balance has been set to "Bidirectional" (if necessary, set the "Host" parameter accordingly),
- 3. The parameters for the RS-232 interface on the balance correspond with those on the titrator.
- As long as the balance is not connected to the titrator, the settings "Baud Rate", "Data Bit", "Stop Bit", "Parity" and "Handshake" can be entered manually. These are however automatically overwritten with the values identified by the PnP as soon as the user sets the same transmission parameters at the balance and the titrator.

Sartorius | Others

After you have selected this option and the system has recognized the balance, you can define the following parameters:

Parameters	Description	Values
Name	Specify a descriptive name of your choice.	Arbitrary
Serial number	The serial number of the relevant device type.	Arbitrary
Connection	The serial port to which the device is connected. Possible con- nections are located on the mainboard, the analog board and the conductivity board.	MB/COM1 MB/COM2 AB1/COM More depending on configuration
Baud rate	The baud rate for data transmission via the RS-232 interface.	1200 2400 4800 9600 19200
Data bit	Defines the number of data bits.	718
Stop bit	Defines the number of stop bits. (2 stop bits can only be selected if 7 data bits are also selected at the same time.)	1 2
Parity	Defines the parity protocol.	Even Odd None
Handshake	Data transmission via the RS-232 interface. (Only the handshake option "Xon-Xoff" is available for serial connections on the analog and conductivity board in conjunction with a baud rate of 9600.)	None Xon-Xoff

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- The settings for the baud rate, data bit, stop bit, parity, and handshake must agree for the balance and titrator!
- If None is selected as balance type that means that no balance is to be connected to the titrator.

5.2.3.2 Barcode reader

Navigation: Home > Setup > Hardware > Peripherals > Barcode reader

When a barcode is imported, the system checks whether the imported barcode is suitable for starting the method. If so, the analysis start dialog is opened; all known data is entered there. If not, the barcode is ignored. If an analysis is already running with the same method ID, the sample is added to the end of the current analysis. An exception to this occurs if the **End series** barcode has previously been read. In this case, a new analysis is started (with the same method).



• Only one barcode reader can be defined.

Define the following parameters for a barcode reader:

Parameters	Description	Values
Name	Specify a descriptive name of your choice.	Arbitrary
Serial number	The serial number of the relevant device type.	Arbitrary
Transfer Smart-	Transfer barcode to LabX.	Yes No
Codes to LabX		

5.2.3.3 USB-Stick

Navigation: Home > Setup > Hardware > Peripherals > USB-Stick

Commercially available USB sticks from USB Version 1.1 are supported.

You can assign a relevant name to the USB stick.

5.2.3.4 Printer and USB data export

Navigation: Home > Setup > Hardware > Peripherals > Printer

The titrator supports the following types of printers and data export via the USB interface:

- USB printer with PCL record for Version 4 and higher.
- USB compact printer (stripe printer).
- USB data export.

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- For data export, to an RS interface, you will need the USB RS232 adapter, the so-called **USB data export box**.
- Data export is not supported by the models "x20"

The USB compact printer prints out the following data:

Results	All except for curves and tables of measured values
Method function Record	Overview
	Results
	Raw results
	Resource data
	Sample data
	Method data
Setup	List printouts
	Parameter printouts
	Total printouts
Methods	List printout
	Parameter printouts
Series	List printout
	Parameter printouts

- The compact printer does not generate the automatic printouts generated by the method functions **Instruc**tion and **Calculation** during the analysis. It also does not support all languages.
- For Karl Fischer determinations, manual concentration, drift and blank value analyses are printed out automatically.

Define the following parameters:

Parameters	Description	Values
Printer type	Selection of a printer, The USB compact printer does not support all languages. This printer can only print out a limited quantity of analysis data and results. For USB data export, the data is transmitted regardless of the selected language. Only a limited quantity of data and results are exported.	USB printer USB compact printer USB data export

Information on the USB data export

- In the method function Record, the parameter Summary must be activated (Record = outside loop) or Per sample or Per series (Record = inside loop). The other settings in Record have no effect.
 - If the method function **Record** is inserted outside the loop, the data from the preceding loop and the data between the **End of sample** and method functions **Record** are output.
- For a KF method in the method function **Record** (within the loop), the parameter **Summary** should be selected with the value **Per sample** or **Per series**.

If data export is activated, the following data is transmitted:

- The most important sample data and results, either per sample or per series, according to the parameter setting in **Summary** of the method function **Record**.
- Automatic reports for drift, blank value and concentration determination in a Karl Fischer water content determination if the global setting Print autom. KF protocols is also activated Navigation: Home > Setup > Global settings > Analysis and resources behavior > Analysis sequence settings

Depending on the selected **printer** type, the following parameters appear in the printer dialog box:

• USB printer and USB compact printer

Parameters	Description	Values
Status	Indicates whether the selected printer type is installed.	Installed
Name	Specify a descriptive name of your choice.	Arbitrary
Serial number	The serial number of the relevant device type.	Arbitrary
Connection	Information on the USB port to which the printer is connected. PnP is displayed if the printer is not connected to the titrator.	MB1/USB1 PnP

• USB data export

The USB data export box is required for USB data export. If it is connected to the titrator using the USB interface, the box is automatically detected (PnP detection).

Parameters	Description	Values
USB data export box	Indicates whether the USB data export box is installed (info field).	Installed Not installed
Connection	The USB port to which the USB data export box is connected (info field). PnP is displayed if the box is not connected to the titrator.	USB PnP
Baud rate	The baud rate for data transmission via the USB interface.	1200 2400 4800 9600 19200
Data bit	Information on the number of data bits is displayed.	8
Stop bit	Information the number of stop bits is displayed.	1
Parity	Defines the parity protocol.	Even Odd None
Handshake	Data transmission via the USB interface.	None Xon-Xoff

The max. Xoff duration for outgoing data is around 30s.

5.2.3.5 PC settings

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Navigation: Home > Setup > Hardware > Peripherals > PC settings

Configure these settings if you have your instrument connected to the PC software LabX.

- The PC with LabX installed must always be connected to the PC (USB) or Ethernet port on the rear panel.
- After the settings have been modified, it may be necessary to restart the instrument.

Parameters	Description	Values
Connect to LabX at start-up	If this parameter is activated, a connection to LabX will be estab- lished on startup.	Yes I No
Connection type	Defines how the titrator is connected to the PC, either via the net- work connection or via the USB connection.	Network USB
Status	Information on the connection status from the instrument to LabX.	Connected Discon- nected
Port number	Defines the port for a network connection of the titrator to LabX. Only appears for Connection type = Network .	102465535

5.2.3.6 Network settings

Navigation: Home > Setup > Hardware > Peripherals > Network settings

Configure these settings if you have your instrument connected to a network.

Parameters	Description	Values
Obtain IP address auto- matically	Indicates whether the IP address should be automatically obtained over the network.	Yes I No

IP address	If the IP is not to be automatically obtained, you can enter it here.	000.000.000.000 255.255.255.255
Subnet mask	If you want to run the titrator on a local subnetwork, you can define the subnet mask here that you want to use to link the sub- net's IP address.	000.000.000.000 255.255.255.255
Standard gate- way	This is where you can enter the address of the standard gateway for communication between the various networks.	000.000.000.000 255.255.255.255

5.2.3.7 Fingerprint reader

Navigation: Home > Setup > Hardware > Peripherals > Fingerprint reader

You can use a fingerprint reader to authenticate users on the titrator. In order to do this, the fingerprint reader must be activated on the titrator. The following parameters are available for this:

Parameters	Description	Values
Activate finger- print reader	Activates the fingerprint reader for authenticating users when log- ging onto the titrator.	Yes I No
Status	Indicates whether the fingerprint reader is connected to the titra- tor.	Installed Not installed
Name	The designation of the fingerprint reader.	Arbitrary
Connection	Information on the USB port to which the fingerprint reader is con- nected. PnP is displayed if the fingerprint reader is not connected to the titrator.	PnP USB 1

Register fingerprint

Navigation: **Home** > **User data**

The following procedure must be performed in order to register each user:

- 1 Log on to the titrator with your user name (and possibly your password).
- 2 In **Home**, tap [**User data**] to open the corresponding window.
- 3 In User data, tap [Register fingerprint] to open the corresponding window.
- 4 Place the preferred finger on the fingerprint reader and repeat the step as prompted.
 ⇒ When completed, the message **Registration successful.** appears.
- 5 Confirm the message with the [OK] to return to the User data window.
- 6 Confirm with [**OK**] to return to the homescreen.
- ⇒ The next time you log on, the **Fingerprint login** window will appears. To log on, place the appropriate finger on the fingerprint reader.
- You can only log on using the fingerprint reader if Activate fingerprint reader is selected. Navigation: Home > Setup > Hardware > Peripherals > Fingerprint reader
- You are still able to log on using a password. To do this, tap [Password login].

5.2.3.8 LevelSens

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Navigation: Home > Setup > Hardware > Peripherals > LevelSens

The level sensor (LevelSens) can be used either to monitor the fill level of titration or solvent vessels or to prevent the overflow of waste vessels.

The level sensor is connected to the "LevelSens box", which is connected to the titrator via the CAN interface. The titrator automatically recognizes up to two of these boxes (PnP recognition). These appear in the settings. Navigation: **Home** > **Setup** > **Hardware** > **Peripherals** > **LevelSens**

- 1 In LevelSens, tap on a "LevelSens box".
 - \Rightarrow The windows to edit the parameters opens.
- 2 The parameters Level, Waste or Inactive can be defined for the relevant sensor type

Activating level monitoring

- At the start of a method or a manual operation.
 The level is checked for all activated and connected sensors, regardless of whether they are used in the method.
- At the start of each sample (GT).
- After completion of a Karl Fischer analysis (KF).
- Before the start of a KF Stromboli method.
- Before replacing the solvent.
- During the course of the following manual operations: Burette (Rinse, Rinse multiple burettes, Dispense, Manual titration), Pump, Auxiliary instrument (output 24V), Sample changer (Pump, Rinse).

If the fill level is not reached or exceeded, a message appears with a prompt either to empty or fill the vessel (depending on the Setup setting: **Waste** or **Level**). The analysis is interrupted during this time. After the vessel has been emptied or filled and the message has been confirmed, the analysis is resumed.

- Only two LevelSens boxes can be entered in the settings. Additional boxes do not generate an additional entry.
- Entries in the settings can only be deleted if the corresponding LevelSens box is not installed.
- The sensor must be fitted in such a way that when the maximum fill level is reached, the analysis of a sample, the entire loop of a Stromboli method or a solvent replacement can be performed.
- The fill level is only checked before a sample analysis, at the start of a Stromboli method or before a solvent replacement.

Parameters	Description	Values
Name	Information on the designation of the LevelSens box. In the settings, the first detected box is entered as LevelSens Box 1, the second as LevelSens Box 2.	-
Chip ID	Information on the Chip-ID of the detected LevelSens box.	-
Position	Information on the position of the LevelSens box connected to the titrator.	PnP PnP1 PnP2
Sensor 1 typeSensor 4 type	Specifies the sensor type to be used.	Level Waste Inactive

5.2.4 Titration stands

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Navigation: Setup > Hardware > Titration Stands

In this dialog, you can add new titration stands or select existing stands that are connected to the titrator and change their parameters. You can also print the list of entered titration stands. You can also delete individual titration stands.

You can create the following titration stands:

- Stromboli TTL
- Karl Fischer stand

Choose the New button in the Titration stands dialog to open the Titration stand parameters dialog box.

To configure the new titration stand you have just created, you can determine the following parameters:

Parameters	Description	Values
Туре	The type of the titration stand to be used.	KF stand I Stromboli TTL
Name	Specify a descriptive name of your choice.	Arbitrary
Stirrer output	Defines the stirrer output.	MB/Stirrer1 MB/Stir- rer2 AB1/Stirrer Internal stirrer More depending on configu- ration

• The Stromboli oven sample changer is connected to the TTL port of the mainboard.

5.2.5 Homogenizer

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Navigation: Setup>Hardware>Homogenizer

The Homogenizers dialog displays the available homogenizers (control type TTL).

You can assign a titrator output to the listed homogenizer:

Parameters	Description	Values
Output	Indicates which port on the titrator you want to use.	TTL Out 14

5.3 User settings

Navigation: Home > Setup > User settings

These settings contains the options that can be made specifically for each currently logged in user.

You can configure the language, the screen settings (for the touchscreen), the layout of the alphanumeric and numeric keyboard, the use of beeps, and shortcuts for each user.

5.3.1 Language

Navigation: Home > Setup > User settings > Language

Define the following parameters:

Parameters	Description	Values
Touchscreen	Defines the language for operation of the terminal.	German I English I French I Italian I Span- ish I Chinese I Russian I Polish I Korean
Record	Defines the language in which the protocols are to be printed out.	German I English I French I Italian I Span- ish I Chinese I Russian I Polish I Korean

• For the Chinese and Korean language settings, it is not possible to print using the USB-P25 tape printer.

• For Polish, records can be printed on the USB-P25 tape printer without special characters.

5.3.2 Screen

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Navigation: Home > Setup > User settings > Screen

Define the following parameters:

Parameters	Description	Values
Primary color	Here various color schemes for the user interface can be selected.	Gray Blue Green Red
Brightness	Specifies the display brightness in [%].	50 60 70 80 90 100 [%]
Button shape	Defines whether to display the buttons in the menus with square or rounded corners.	Rounded Square
Screen saver	Here you can define whether the screen saver should be used.	Yes No
Wait time	Defines how long in [min] the system should wait after the user's last action on the terminal before activating the screen saver.	11000

5.3.3 Beep

Navigation: Home > Setup > User settings > Beep

Define the following parameter:

Parameters	Description	Values
At push of a but-	Enables a beep when tapping on the touch screen.	Yes I No
ton		

5.3.4 Shortcuts

Navigation: Home > Setup > User settings > Shortcuts

Each user can manage the shortcuts that they have created. Individual shortcuts can be selected and deleted and the following parameters of a shortcut can be changed:

Parameters	Description	Values
Description	Any name for the shortcut.	Arbitrary
Immediate start	The method, series, or manual operation can be started immedi- ately. This enables you to start the analysis without any interfer- ing dialog.	Yes No
Homescreen position	You can select the free position for the shortcut on the Home- screen.	112

5.3.5 Keyboards

Navigation: Home > Setup > User settings > Keyboards

In this dialog, you can define the layout for the alphanumeric and the numeric input fields. The following settings are available:

Parameters	Description	Values
ABC keyboard	Determines the layout of the alphanumeric input field.	English French Ger-
		man
123 keyboard	Defines the organization of the keys for the numeric input field.	Calculator Phone

5.4 Global settings

Navigation: Setup > Global settings

In the **Global Settings** dialog, you can make general settings on the titrator that apply for all users. The settings in this dialog can only be changed by users with the appropriate authorizations.

Global settings include:

- System settings that apply for all users.
- User management for creating user accounts and assigning rights.
- The settings for Analysis and resources behavior regarding the sequence and monitoring the expiration
 dates and life span of resources (determining the actions of the titrator before, during and after the performance of an analysis), and the response of the titrator when resources are deleted or when PnP resources
 are identified.
- **Solvent control** instructs the user to replace the reagent solution. For information on the process for replacing the solution.

5.4.1 System

Navigation: Home > Global settings > System

Titrator identification

You can enter and assign any ID consisting of at least four characters to the titrator.

Parameters	Description	Values
Titrator ID	Define the instrument identification.	-
Titrator	Indicates the titrator type.	T50 T70 T90
Serial number	Information on the serial number of the instrument.	-
Titrator FW ver-	Information on the firmware version of the instrument.	-
sion		

Date / Time

You can define the format used to display the date and time and set the titrator date and time.

Parameters	Description	Values
Date format	Defines the format for displaying the date.	mm/dd/yyyy dd/mm/yyyy

Time format	Defines the format for displaying the time.	24h a.m./p.m.
Date	Enter the current date.	-
Time	Enter the current time.	-

Header and footer

Define whether all printouts generated by the titrator should have a header or footer. The content of these headers and footers can be entered directly into the respective setting.

As part of the end of record, signature fields are appended to the respective printout consisting of a declaration (e.g. **Approved by**) followed by an empty line. A personal signature can be then be entered on this line.

Parameters	Description	Values
Header	Activates the header on print outs.	Yes No
Text	Defines the text for the header. Only for Header = Yes .	Arbitrary
Footer	Activates the footer on print outs.	Yes No
Text	Defines the text for the footer. Only for Footer = Yes .	Arbitrary
End of report	Select the information to be printed at the end of a report.	Created by Modified by Checked by Approved by

Data storage

In the T50 and T70, the system only saves the results from the last analysis (series or individual sample). In the T90 select [Select Series] to select the results from the last two analyses.

Parameters	Description	Values
Delete data on	Define if analysis data is to be deleted from the titrator memory	Yes I No
shut down	when the titrator is shut down.	

5.4.2 User Management

Navigation: Global settings > User management

Here you manage users, user groups, and account policies for the titrator. A maximum of 30 different users can be defined for the titrator, but only one user at a time can be logged onto the instrument (single user operation). One user with administrative rights is already saved on the instrument.

User accounts can be deleted, printed out and edited.

Users

1 In User management tap [Users] to open the list of users.

- 2 To add a new user, tap [New].
 - or -

Edit an existing user.

You can define the following parameters for each user account:

Parameters	Description	Values
User Name	The user's login ID.	Arbitrary
Full Name	The user's full name.	Arbitrary
Groups	User group that is assigned to the user.	Experts Routine-User
Description	Any description for the user account or for the user.	Arbitrary
Reset password	If activated, the user's password is reset to "123456" and the user is prompted to change their password the next time they log in. Only appears if Enforce password/fingerprint = Yes is selected in Account Policies .	Yes I No
Lock user	If activated, the user account is locked. Only appears if Enforce password/fingerprint = Yes is selected in Account Policies .	Yes I No

Enforce pass-	If activated, the user is forced to change their password the next	Yes No
word change	time they log on to the titrator.	
	Only appears if Enforce password/fingerprint = Yes is selected	
	in Account Policies.	

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- If the parameter **Reset password** is activated, the parameter **Enforce password change** will be automatically activated.
- The default password for this user (User ID: "Administrator") is "123456" (do not enter inverted commas).

User group

Two fixed user groups are defined in the titrator: **Experts** and **Routine-User**. Any user can belong to one of the user groups (with different authorizations). User management for "x20" models only contain the **Experts** group.

- 1 In User management select [Groups].
 - \Rightarrow The two fixed defined groups are listed in this window.
- 2 Select one of these groups to access the **Group parameters** dialog.
- \Rightarrow This is where the parameters and authorization rights of the user groups are defined.
- Users in the **Experts** (system administrators) group have all the rights listed below:
 - Editing methods Users can create methods in the Method editor and have full editing rights.
 - Editing series and samples Users can create series templates and samples and have full editing rights.
 - Editing resources and peripherals Users can create resources and peripherals and have full editing rights.
 - Editing global and analysis sequence settings: Users can edit the global settings in Setup.
 - Editing user-specific settings: Users can edit the user-specific settings in Setup.
 - Editing results: Users can edit the saved results.
 - Starting methods and series: Users can start methods from the method list or the Start analysis dialog.
 - Executing manual operations: Users can execute manual operations.
- Users in the Routine-User (operators) group can start methods and series and execute manual operations.

*Basic functions: Starting drift or blank determination.

Account Policies

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In Account Policies define the actions of the titrator when it is started up.

Parameters	Description	Values
Enforce pass-	If this parameter is activated, the titrator always starts with the	Yes No
word/fingerprint	login screen (even if only one user is defined for the instrument).	
	The user name must always be entered in the login screen man-	
	ually (the corresponding input box is always initially empty).	

If this option is selected for an instrument with factory settings, the titrator will demand the password for the
predefined user (User ID: "Administrator") the next time it is booted up. This password is "123456" (do not
enter inverted commas).

Min. no. of char-	Specifies the minimum number of characters required for user	Yes No
acters	passwords. If this parameter is changed, then users whose pass-	
	word does not meet this requirement will be requested to change	
	their password accordingly the next time that they log in.	

5.4.3 Analysis and resources behavior

The settings that you make here relate to the sequence of the analysis of samples or series with the aid of methods.

- You can define the actions of the titrator when started, during an analysis and afterward.
- You can also program the response of the titrator to the deletion of resources or when it identifies PnP resources.

Analysis sequence settings

The analysis sequence settings can only be modified if no tasks are currently being performed by the titrator.

You can make the following settings that influence the sequence of an analysis.

Parameters	Description	Values
Show required resources at start	When an analysis is started a screen appears displaying all resources required for the analysis and their status (available, not-available, locked or in use). If an individual entry is selected from this screen then the user receives additional information about the respective resource. However, if "no" is selected, then the required resources are still checked when the analysis is started and if necessary, an appro- priate error message is issued.	Yes I No
Show SOP	If an SOP (standard operating procedure) has been defined in the Title method function then this will be displayed before the method is started provided that "yes" has been selected.	Yes I No

 If Show required resources at start and Show SOP parameters were set to "Yes" at the start of a job series, all SOPs and subsequently all resources for the individual sample series must be confirmed before the job series is started. In this way a job series can be performed after it has been started without any further interruption.

LabX: Warning	If "yes" is selected then a warning is issued when the analysis is	Yes No
when not con- nected	started if there is no connection to LabX.	
Show results after analysis	If a manual or external titration stand is being used, then the results for a sample are automatically displayed after they have been analyzed and must be acknowledged by the user before the analysis can be continued. With Auto stand the results are displayed for a certain period and are not to be confirmed.	Yes I No
	The activated parameter is valid for the following functions: Analysis : the results for a sample are shown Calibration/Loop : The results of the calibration are shown (slope, zero point) Sensor test : The results of the test are shown (slope, zero point, drift and sensor test OK / not OK)	
Check local printer connec- tion and wait	When selected, the availability of a printer is checked at the beginning of the method. If not selected and no printer is connected, the method function Record is skipped. If a printer is connected, the method function Record is executed even if this parameter is not selected.	Yes I No
Print autom. KF protocols	Controls the printouts in manual drift or blank determination.	Yes No
Save results	Defines if date, time, user, Methods ID, sample ID, results, result unit per sample is saved in a file. If this parameter is activated and the stick is not detected during the data writing process, you can stop the process or you can plug in another USB-stick for the data writing and to continue the method run. Only for Check USB-Stick connection and wait = Yes .	To USB-Stick No
Check USB-Stick connection and wait	If this parameter is activated, the presence of a USB - Stick is val- idated at the start of the analysis.	Yes I No

Resources behavior

Use the following parameters to configure how the titrator responds to the deletion of resources and its response to the automatic identification of PnP resources.

Action when exceeding usable life

If it is determined by the titrator that the expiration dates of a resource have been exceeded, then the titrator may perform various actions.

Warning	The user is warned that the resource's usable life has been exceeded and the raw results and
	results determined with the respective resource will be labeled accordingly.

Block The user is notified that the usable life of the resource has been exceeded and it is no longer possible to start the analysis with the affected resource. (Methods that result in renewal of this resource can however still be started.)

None If you select "none" then the analysis is started without message in spite of the exceeded usable life. The expiry of the expiration date will, however, be logged.

Parameters	Description	Values
Auxiliary values	This action is performed if the system determines at the start of an analysis that the usable life of an auxiliary value that is to be used in the analysis has been exceeded.	None Warning Block
Blank values	This action is performed if the system determines at the start of an analysis that the usable life of a blank value that is to be used in the analysis has been exceeded.	None Warning Block

Action when exceeding life span

If, at the start of an analysis, it is determined that the life span of a resource to be used for the analysis has expired, the titrator can set various actions.

Warning	The user is warned that the resource life span has been exceeded and the raw results and results determined with the respective resource will be labeled accordingly.
Block	The user is notified that the life span of the resource has been exceeded and it is no longer possible to start the analysis with the affected resource.

None The analysis is started in spite of the exceeded life span.

	Parameters	Description	Values
;	Sensors	The action is executed if the system determines at the start of an	None Warning Block
		analysis that the usable life of a sensor has been exceeded.	

5.4.3.1 Monitoring the Expiration Date and Life Span of Resources

For certain resources, the titrator provides automatic monitoring of the usable life/life span.

Monitoring the expiration date of a resource

The expiration date is the period after which the values for a specific resource should be remeasured. These values depend on the nature of the resource:

- The numerical value of an auxiliary value.
- The numerical value of a blank.

Whether the expiration dates should be monitored can be defined in the setup for each individual resource.

If monitoring is activated then additional parameters become available in the respective resource with which the duration of the expiration dates can be determined. In addition a reminder may optionally be issued by the titrator before the expiration dates expire.

Parameters	Description	Values
Time period	Specifies the time range.	Days Hours
Usable life	Defines the time span of the expiration dates either in days or hours (depending on: Time period).	Days : 11000 Hours : 110 ⁴
Reminder	Determines whether the titrator should issue a warning before the service life or usable life of a resource elapses.	Yes I No
Days before expiration	Determines the number of days before the service life of the resource that the titrator should issue a warning. The value entered must be less than the value in Usable life . Only if Time period = Days and Reminder = Yes .	01000

You can define the following parameters:



If a resource is updated, the Date/Time field in Setup of the affected resource is automatically adjusted and the expiration date (or time) is recalculated.

In the "Global settings" under "Analysis and resources behavior", you can define how the titrator deals with the relevant resource if the expiration dates have been exceeded at the start of the analysis (see "Action when exceeding usable life (page 29)").

Monitoring the Life Span of a Resource

The life span describes the period of time after which a resource is consumed and should be replaced. In the sensor Setup (accessible via the Hardware button), you can determine whether or not the titrator should monitor the life span.

If monitoring is activated then additional parameters become available in the respective resource with which the date of initial operation of the resource and the duration of its life span can be defined.

You can define the following additional parameters:

Parameters	Description	Values
Initial operation	Here you can enter the date of initial activation of the resource.	Date
Life span	Defines the life span of the resource in months.	0100

In the "Global settings" under "Analysis and resources behavior", you can define how the titrator deals with the relevant resource if the life span has been exceeded at the start of the analysis (see "Action when exceeding life span (page 29)").

5.4.4 Solvent Control

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For Karl Fischer titration, the solvent must be replaced at regular intervals to prevent results from becoming corrupted. The titrator system monitors the usable life and the capacity of the solvent and the number of samples.

Before you can activate Solvent Control, at least one of the following monitoring parameters must be defined:

- The time interval for the use of the solvent.
- The capacity limit, i.e. a fixed maximum value of the total water volume of samples titrated (including standby and pretitration) in the same solvent.
- Maximum number of samples to be titrated in the solvent.

In order to monitor the solvent, the system records and adds up the time, water volume and number of samples for each titration. When the defined monitoring parameters have been reached, a system message is displayed. The user then has an opportunity to replace the solvent. The Solvent Manager is started to pump the solvent away. The cell is then filled with new solvent. All counters are reset to zero.

 For sample analysis using the Stromboli oven sample changer, the solvent can only be replaced in standby mode before analysis of the first sample, or at the end of the series, in case the titrator returns to standby mode.

Parameters	Description	Values
Monitoring usable life of solvent	Specifies whether the usable life of the solvent is to be monitored.	Yes I No
Usable life	Defines the time interval in days for the use of the solvent.	1104
Enforce replace- ment when exceeding usable life	Forces the user to perform the solvent replacement immediately after the message is issued.	Yes I No
Autom. exchange when exceeding usable life	The exchange of solvent is performed automatically when exceeding the specified usable life.	Yes I No
Monitoring capacity of sol- vent	Specifies whether the capacity of the solvent is to be monitored.	Yes I No

Navigation: Home > Setup > Global settings > Solvent Control

Enter max. amount of water	The maximum volume of water in [mg] for a solvent. Only for Monitoring capacity of solvent = Yes .	010 ⁶
Enforce replace- ment when exceeding capacity	Forces the user to perform the solvent replacement immediately after the message is issued.	Yes I No
Autom. exchange when exceeding capacity	The exchange of solvent is performed automatically when exceeding the specified capacity of the solvent.	Yes I No
Monitoring no. of samples	Specifies whether the system should monitor the number of samples.	Yes I No
Autom. exchange at max. no. of samples	Maximum number of samples (concentration and blank value do not count) after which the solvent is to be replaced.	0120
Enforce replace- ment at max. no. of samples	Forces the user to perform the solvent replacement immediately after the message is issued.	Yes I No
Autom. exchange at max. no. of samples	The exchange of solvent is performed automatically when exceeding the specified maximum number of samples.	Yes I No
Stir	Enables the stirrer during solvent exchange.	Yes I No
Fill time	Defines the pumping time for filling a fluid (for Autom. exchange).	01000 ∞
Drain time	Defines the pumping time for draining a fluid (for Autom. exchange). The duration of the hose drain operation should be as long as possible to ensure that the hoses are completely free of fluid fol- lowing draining.	01000∣∞

See also

• Monitoring the expiration date of a resource (page 29)

5.5 Maintenance & Service

Navigation: Home > Setup > Mainten. & Service

5.5.1 MT service

Select the MT service button to open the Last MT services dialog box.

In this dialog, you can view and print out a list of the most recent (max. 10) METTLER TOLEDO services. Under each date, the user name of the METTLER TOLEDO service technicians and the date and time of the service appointment are displayed. The most recently performed service always appears at the top of the list.

The **Settings** button in the **Last MT services** dialog window opens the **Service data** dialog, in which you can change the service life (in days) of the last service date and configure the titrator to issue a warning at a defined time before the service life elapses (requires administrator rights). You can define the following parameters:

Parameters	Description	Values
Service life	Defines the service life (in days) of the most recently performed service.	0104
Reminder	Determines whether the titrator should issue a warning before the service life or usable life of a resource elapses.	Yes I No

Days before expi-	Determines the number of days before expiry of the service life	01000
ration	that the titrator should issue a warning.	
	The value entered here must be smaller than the service life.	
	(Appears only if "Reminder" is activated.)	

5.5.2 Import/Export

You can use this function to save titrator data on a USB stick (export) and reload the data back to a titrator later on (import).

Thus is it possible to create a backup of most data that has been changed from the titrator's default settings.

Uploading data from a backup copy results in the existing data in the titrator being overwritten. In this way you can immediately duplicate the status of one titrator in another one or restore titrator settings after repair.

The following two rules should be observed:

- Memory copies can only be imported from the same type.
- Memory copies can only be imported from the same or from a lower software version.

You can select whether you want to export or import a backup copy, an individual method or the user management.

A **backup copy** includes, for example:

- All parameters of methods, series and firmware updates
- Setup inclusive of all resources
- All shortcuts

The backup copy does **not** contain all saved results, data saved on a PnP component and the default parameters for manual operations.

When you import/export an **individual method** you can select which method is to be exported or imported. You require the right to edit methods.

When you import/export **user management settings** the entire user management settings with all users and their properties are exported or imported.

- ► In the Maintenance & Service dialog, open the Import / Export dialog window.
- In this dialog, you can define the following parameters:

Parameters	Description	Values
Action	Here you can select whether you wish to export the titrator data to a memory stick or to import it from a memory stick to a titrator.	Export Import
Data	In this box you can select the data that you wish to export or import. You can select whether you want to export or import a backup copy, an individual method or the user management.	Export I Import
Method ID	Here you can select the Method ID for the relevant method.	Method list

5.5.3 Reset to factory settings

Select the Reset to factory settings button to reset the titrator.

• In the process all data and changes to settings made by users of the titrator are lost.

5.5.4 Titrator firmware history

The **Titrator firmware history** button displays a list of the firmware updates or model upgrades. The first entry in the list represents the initial operation of the titrator.

All list entries are stored with date, type, FW version and the user name of the user who performed the action.

5.5.5 Board firmware

You use the **Board firmware** button to display a list of all boards and burette drives available on the titrator along with the relevant firmware version. You can carry out an update.

5.5.6 Terminal

You use the **Terminal** button to display the chip in the terminal.

5.5.7 Board data

You use the **Boards** button to display and print out a list of all the boards fitted in the titrator. Each board is listed by name and module location.

If a board is selected from the list, then its chip ID and all data on available inputs and outputs including the adjustment data will be displayed.

5.5.8 Upgrade

You use this dialog to upgrade a titrator from a lower to a higher model. To do this, you will need a product key that you can obtain from your METTLER TOLEDO Representative. You will need the data displayed in the screen to order the product key:

You can easily transfer the data from this screen by pressing the "**Print**" softkey.

If you have received your product key, you can enter it via the "Product Key" softkey and perform an upgrade.

5.5.9 Update

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You use the Update button to update the titrator firmware using a USB stick.

5.5.10 Drives

You use the **Drives** button to display and print a list of all connected drives. The list contains the position, serial number, chip-ID and status of each drive.

5.6 Values

Navigation: Home > Setup > Values

Blanks and auxiliary values can be created, edited and deleted and the list of defined blanks or auxiliary values can be viewed and printed out. It is also possible to print out the individual values with their parameters.

Settings	Explanation
Blanks	Blank values can be used in formulas for calculations.
Auxiliary values	You can use auxiliary values in formulas.

5.6.1 Blanks

Navigation: Home > Setup > Values > Blanks

Blanks can be used in formulas for calculations. They can either be created manually with the aid of their various parameters or generated as the result of a method. A resulting blank (or calculated mean value) can then be assigned to a blank using the method function **Blank**. The blank will then appear under the assigned name in the Blank list in Setup.

Adding a blank value

- In Blanks choose [New].
- \Rightarrow The windows to edit the parameters opens.

Define the following parameters to define the blank:

Parameters	Description	Values
Name	Specify a descriptive name of your choice.	Arbitrary
Unit	The units in which the blank is specified.	Arbitrary
Value	Here you can enter a numerical value.	-10 ⁸ 10 ⁸
Monitoring usable life	Specifies whether the usable life of a resource or a value is to be monitored.	Yes No

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- A maximum of 100 blanks can be saved in the titrator.
- Blanks cannot be deleted or modified if they are currently in use.
- When a blank is assigned with the "Blank" method function, this is updated in the setup immediately after completion of the method function.

5.6.2 Auxiliary values

Navigation: Home > Setup > Values > Auxiliary values

You can use auxiliary values in formulas. They can either be manually created and edited or can be generated using a method. A result, a mean derived from several results or a raw result can be assigned to an auxiliary value by means of the "Auxiliary Value" method function. The auxiliary value then appears under the assigned name in the auxiliary values list in the Setup.

Adding an auxiliary value

- In Auxiliary values choose [New].
- \Rightarrow The windows to edit the parameters opens.

Define the following parameters to define the auxiliary value:

Parameters	Description	Values
Name	Specify a descriptive name of your choice.	Arbitrary
Value	Here you can enter a numerical value.	-10 ⁸ 10 ⁸
Monitoring usable life	Specifies whether the usable life of a resource or a value is to be monitored.	Yes I No

• A maximum of 100 auxiliary values can be saved in the titrator.

• Auxiliary values cannot be deleted or modified when they are currently in use.

• When an auxiliary value is assigned with the method function **Auxiliary value**, this is updated in the setup immediately after completion of the method function.

5.7 Terminal

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You use the **Terminal** button to display the chip ID of the chip in the terminal (only for V30).

6 Manual operations

You can use manual operations to access various titrator functions that are not directly connected to the execution of an analysis, but that might be useful during the sample preparation, for example. You can call up the following manual operations from here with the relevant titrator components:

Hardware com- ponents	Possible manual operations	Possible usages
Stirrer	Stir	Dissolve a solid sample
Sensor	Measure	Voltametric indication
Burette	Rinse	Rinse burette before changing a titrant
	Dispense	Dispense during sample preparation
Pump	Pump	Fill, empty, or replace solvents.

• A maximum of four manual operations can be performed at the same time (one per hardware component).

- Manual operations can also be executed while an analysis is running, if the hardware components that you want to operate manually are not already being used by the analysis.
- The resource parameters in all editable fields can be changed temporarily (only for the execution of the manual operation in question) and can vary from the setup settings. The changes made will not be copied over to the setup, however.

6.1 Stirrer

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To switch a connected stirrer (Rod stirrer or magnetic stirrer) on or off for a definable time interval and at a definable stirring speed, select the following:

Navigation: Home > Manual > Stirrer

- 1 Make a selection in **Titration stand**.
- 2 Select the desired stirrer in Stirrer output and enter the speed in [%].
- 3 Enter the stir time in [sec] or select "∞" for an infinite duration.
- 4 Tap [Start] to start the stirrer.
- \Rightarrow The stirrer starts. Tap [**Stop**] to stop the stirrer at any time (terminating the manual operation).

Define the following parameters:

Parameters	Description	Values
Titration stand	Defines which titration stand is to be used.	List of available titration stands
Stirrer output	Defines the stirrer output.	MB/Stirrer1 MB/Stir- rer2 AB1/Stirrer Internal stirrer More depending on configu- ration
Speed	Defines the stirring speed in [%].	0100
Stir time	The stirring time, in [sec], during which the stirrer should be in operation. Select "∞" for unlimited stirring time.	010 ⁴ I ∞

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Entries made here will only be applied to the manual operation and will have no effect on the instrument settings.

6.2 Sensor

Navigation: Home > Manual > Sensor

- 1 Select the sensor you want to use from the list of sensors defined in the settings.
- 2 Determine the polarization current.
- 3 Select the relevant titration stand.
- 4 Select the stirrer output for the stirrer and enter a speed.
- 5 Enter the duration of the measurement in [sec].

- 6 Select whether to output a record on the printer.
- 7 If you want to output a record on the printer, use dt [sec] to define the time interval between measurements.
- 8 Tap [Start] to start the measurement.
- 9 Tap [Stop] to terminate the procedure at any time.

During the measurement, the system will display the online curve (measured values in the selected unit versus time). You can also tap [**Measured values**] to display a table of measured values instead of the curve.

You can define the following parameters for polarized sensors:

6.2.1 Polarized sensor

Parameters	Description	Values
Sensor	Select a sensor from the list. The list depends on the sensor type selected in Type .	List of available sensors
Ipol	Ipol is the polarization current, in [μ A], for the voltametric indication.	0.024.0
Titration stand	Defines which titration stand is to be used.	List of available titration stands
Stirrer output	Defines the stirrer output.	MB/Stirrer1 MB/Stir- rer2 AB1/Stirrer Internal stirrer More depending on configu- ration
Speed	Defines the stirring speed in [%].	0100
Temperature	Input field for the temperature [°C].	-20200
Duration	The measurement and stirring time, in [sec]. Select " ∞ " for unlimited measurement time.	0104 ∞
Record	If activated, the measured values will be printed out.	Yes No
dt	Defines the time interval in [sec] for outputting measured values to the printer. Only appears if Record = Yes was selected.	16000

Changes made in this dialog will only be applied to the manual operations "Sensor" and have no effect on the settings made in the Setup.

6.3 Burette

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Navigation: Home > Manual > Burette

In the Burette dialog window you can carry out various manual operations with the available burettes.

To rinse an available Burette, dispense a defined quantity of titrant or run a manual titration with a selected burette, select:

6.3.1 Rinse burette

The "Rinse" operation allows you to rinse a burette and its connecting tubes and fill it with fresh titrant, for example if you want to remove air bubbles from the system.

Select:

Navigation: Home > Manual > Burette > Rinse

- 1 Select the titrant that you want to use for rinsing.
- 2 Enter the number of rinse cycles.
- 3 Enter the discharge volume in [%] to define the percentage of the burette's total volume that you want to discharge during each rinse cycle.
- 4 Enter the filling rate in [%] to define the speed at which you want to refill the burette. (100% is the maximum rate.)
- 5 Select "Start" to start the measurement.
- 6 You can select "Stop" to terminate the procedure at any time.

You can determine the following parameters:

Parameters	Description	Values
Titrant	Select a titrant from the list of the defined titrants.	Titrant list
Cycles	Defines the number of rinse cycles to be executed.	1100
Discharge vol-	The volume of titrant, in [mL], to be discharged during the rins-	10100
ume	ing process.	
Fill rate	The filling rate of the burette in percent. 100% stands for maximum filling rate.	30100

6.3.2 Dispense

Navigation: Home > Manual > Dispense

The **Dispense** operation lets you manually dispense a defined quantity of titrant.

- 1 Select the titrant you would like to dispense.
- 2 Select the drive on which the titrant is installed. (For PnP burettes, the system automatically selects the appropriate drive.)
- 3 Enter the volume to be dispensed in [ml].
- 4 Enter the filling rate in [%] to define the speed at which you want to refill the burette. (100% is the maximum rate.)
- 5 Tap [Start] to start the measurement.
- 6 Tap [**Stop**] to terminate the procedure at any time.

You can determine the following parameters:

Parameters	Description	Values
Titrant	Select a titrant from the list of the defined titrants.	Titrant list
Drive	The drive on which the burette with the selected titrant is installed.	18
Volume	Defines the volume to be dispensed, in [mL].	0.001100
Fill rate	The filling rate of the burette in percent. 100% stands for maxi- mum filling rate.	30100

6.4 Pump

Navigation: Home > Manual > Pump

You can use the **Pump** operation to fill or drain (depending on the hose connections) any volume of liquid from the titration beaker using the Solvent manager.

Proceed as follows to start a pump process:

- 1 Choose the action that you want to perform (empty, fill, replace solvent).
- 2 Enter the duration of the relevant action in [sec].
- 3 Tap [Start] to start the measurement.
- 4 Tap [Stop] to terminate the procedure at any time.

You can determine the following parameters:

Parameters	Description	Values
Action	Determines the actions for the pump process.	Drain Fill Replace solvent
Drain time	Defines the pumping time for draining a fluid. The duration of the drain operation for the tubes should be as long as possible to ensure that the tubes are completely free of liquids following draining.	01000 ∞
Fill time	Defines the pumping time for filling a liquid.	01000 ∞
Reset counter	If this parameter is set, all counters are reset when cell filling commences (applies to current capacity per number of sam- ples). The fill date for the cell is also reset.	Yes No
Stirrer	A stirrer can be switched on. Only for Action = Fill or Drain .	Yes No

Titration stand	The name of the titration stand. only if stirrer is activated.	List of titration stands
Stirrer output	Specifies the stirrer output at the relevant board (only available if Stirrer is activated).	Internal stirrer More depending on configu- ration
Speed	Speed in [%]. Only if stirrer is activated.	0100

7 Methods

To carry out an analysis with the titrator, you require a **method**. A method is an analysis program and consists of a sequence of method functions (some with method subfunctions), which are processed by the titrator in sequence.

In this chapter, you will learn how to access and define methods.

The basic building blocks of a titration method consist of sample preparation, stirring and wait times, the actual titration, result calculation and a record. The titrator defines these partial steps as functions that consist of parameters whose values can be changed.

Types of Methods

The titrator distinguishes between the following method types with different objectives:

KF-vol

Method for volumetric water content determination with the Karl Fischer method.

External Extraction

"External extraction" is a KF method for samples with extremely inhomogeneous water dissipation. It is also used for insoluble solids which only release water slowly, even if broken into smaller pieces.

• Stromboli

Method for selecting the Stromboli oven sample changer as the titration stand.

Preloaded Methods

A number of methods have already been stored in the device. These methods were developed by METTLER TOLEDO for specific uses and can be used immediately for the corresponding analysis.

You can change these methods in line with your requirements and save them as user methods.

When creating methods you can revert to method templates, which, according to their objective, specify the structure of the method, and whose parameters already contain the most suitable default values.

Method ID

You can distinguish between different types of method and single methods of the same type using their ID:

- Each method has its own unique method ID.
- The method ID of the Mettler method for volumetric Karl Fischer titration consisting of "KFV", followed by a sequential number (KFV01, KFV02...).
- You can enter a name of your choice for the method ID for a user method, however it cannot begin with "KFV"followed by a number.

7.1 METTLER TOLEDO Methods

METTLER TOLEDO methods are preprogrammed methods for executing specific applications. These methods are stored in the device prior to shipping (Methods List) and can be started immediately by the user. METTLER methods not only provide the sequence of the method functions, but they also define all of the method function settings. A METTLER method can be saved as a user method by saving it under a different method ID.

ID	Title	Description	Titrant	Sensor
M300	Water standard 10.0 mg/g	KFVol	KF 1-comp KF 2-comp	DM143-SC
M301	Di-Sodium-Tartrate 15.660%	KFVol	KF 1-comp KF 2-comp	DM143-SC
M302	Toluene dry	KFVol	KF 1-comp KF 2-comp	DM143-SC
M303	Acetone dry	KFVol	KF 1-comp KF 2-comp	DM143-SC
M304	Milk powder (homogenizer)	KFVol	KF 1-comp KF 2-comp	DM143-SC
M305	Tobacco (external extraction)	KFVol external extraction	KF 1-comp KF 2-comp	DM143-SC
M306	Corn starch (manual oven)	KFVol	KF 1-comp KF 2-comp	DM143-SC

ID	Title	Description	Titrant	Sensor
M307	Air (gaseous samples)	KFVol	KF 1-comp KF 2-comp	DM143-SC
M311	Water standard tablet 10 mg	KFVol	KF 1-comp KF 2-comp	DM143-SC
M312	Oven stand. 5.55% (Stromboli)	KFVol Stromboli	KF 1-comp KF 2-comp	DM143-SC
M313	Temp. ramp (Stromboli)	KFVol Stromboli	KF 1-comp KF 2-comp	DM143-SC

7.2 Creating Methods

You create a new method by changing the parameters of a delivered METTLER method and saving it under a new method ID or by selecting an appropriate method template from the list of proposals, modifying it, and saving it under a new ID.

Navigation: Home > Methods

- 1 Tap [New] to create a new method on the basis of a template.
- 2 From the available templates, choose the one that is most similar to the method you wish to create.
 - ⇒ You can now modify this method in line with your requirements by inserting or removing method functions or modifying its parameters.
- 3 In the method function **Title**, enter a new method ID. Afterwards, a new method will be stored under this method ID.
- 4 Assign a title to your new method.
- 5 Select available method functions to modify their parameters in line with your requirements.
- 6 Tap [Insert] to add additional method functions to the template.
- 7 Now use the arrow-shaped button to select the required position for the new method function in the method. (You will only be able to insert the method functions that are allowed in the corresponding location based on the method syntax.)
- 8 From the list, select the method function that you want to insert.
- 9 Modify the individual parameters of the method function in line with the resources.
 - \Rightarrow The new method function appears in the method.
- 10 To delete a method function, select the function in question and then tap [Delete].
 - \Rightarrow The method function disappears from the method.
- 11 After inserting all required method functions, tap [Save].
- \Rightarrow The method is saved under the method ID and appears in the list of available methods.
- When establishing a new method, follow the rules specified by the instrument.

7.2.1 Method Templates

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When you create a new method, the method templates prescribe the sequence of the method functions. These method templates are not application-specific, but are dependent on the type of titrator. They allow the user to establish user methods quickly and easily. Most of the settings in the method functions that occur in a standard method already have default values.

To convert a method template into a user method, it has to be saved under a method ID.

Title	Type of instrument	Method type
EQP	T50/T70/T90	General titration
EP	Т50/Т70/Т90	General titration
Stating	Т50/Т70/Т90	General titration
Measure	T50/T70/T90	General titration
2-phase	T50/T70/T90	General titration
EQP detection	Т50/Т70/Т90	General titration
Titer with EQP	Т50/Т70/Т90	Titer determination

Titer with EP	T50/T70/T90	Titer determination
Calibration	T50/T70/T90	Calibration
Segmented calibration	T50/T70/T90	Calibration
Blank with EQP	T50/T70/T90	General titration
Blank with EP	T50/T70/T90	General titration
EP/EQP	T50/T70/T90	General titration
EQP/EQP	T50/T70/T90	General titration
EP/EP	T50/T70/T90	General titration
Titer with EQP & EQP	T70/T90	General titration
Titer with EP & EP	T70/T90	General titration
Calibration & EQP	T70/T90	General titration
Calibration & EP	T70/T90	General titration
Calibration & titer with EQP & EQP	T70/T90	General titration
Calibration & titer with EP & EP	T70/T90	General titration

7.3 Modifying or Deleting Methods

You can change user methods or METTLER methods and store them under new method IDs.

i Once a METTLER method has been modified, you will only be able to save it as a copy (or as a user method) with a new method ID.

Modifying a method

To modify a method, select:

Home > Methods

- 1 From the displayed list of methods, select the method that you want to modify.
- 2 As soon as the methods functions of the selected method appear on the screen, you can modify the method.
- 3 In the "Title" method function, enter a new method ID. Afterwards, a new method will be stored under this method ID. You can enter up to twenty alphanumeric characters.
- 4 Select available method functions to modify their parameters in line with your requirements.
- 5 Choose Insert to add additional method functions to the template.
- 6 Now use the arrow-shaped "Insert" button to select the required position for the new method function in the method. (You will only be able to insert the method functions that are allowed in the corresponding location based on the method syntax.)
- 7 From the list, select the method function that you want to insert.
- 8 Modify the individual parameters of the method function.
- \Rightarrow The new method function appears in the method.
- 1 To delete a method function, select the function in question and then choose **Delete**.
- 2 After you have made all of the necessary adjustments, you can store the method in the titrator by choosing **Save**.

Deleting Methods

You can easily delete user-defined methods from the titrator. Select:

Home > Methods

- 1 Select the method that you want to delete.
- 2 Choose **Delete method** to delete the method from the titrator's memory.

7.4 Starting Methods

The titrator offers various ways of starting a method:

- From the method editor
- By choosing Start from the Home dialog

- By using a shortcut on the Home screen
- Via the Series dialog
- By using the "**Setup**" dialog (to perform a calibration or titer determination)

You can use the method editor to start any method stored in the titrator.

- 1 From the displayed list in the **Methods** dialog, select the method that you wish to start (Home > Methods).
- 2 As soon as the method functions of the selected method appear on the screen, you can open the **Start analysis** screen by choosing **Start**.
- 3 Choose **Start** again to reach an overview screen on the resources required for the method. (Only if this was defined in the analysis sequence settings.)
- 4 To execute the method, confirm the screen by choosing **OK**.

7.5 Stopping methods

To terminate an analysis or series while it is running, cancel the relevant method as follows:

 Select the Stop method button in the online dialog to terminate the current analysis. The system then automatically enters Standby mode. Canceled sample, concentration, and blank determinations are listed and marked in the results.

If a drift determination is canceled, this is not entered in the results.

 You can use the More button in the online dialog to open the More KF functions window. Here you can use the Stop method button to cancel a method directly. The titrator performs no further actions. No printout is generated, and you return directly to the Home dialog.

You can use the **End series** button to terminate a series. Once the series is completed, the titrator switches to Standby or Pretitration mode and the series can be restarted. A new series is entered in the "Results" and the original sample parameters are reused.

You can use the **Save series data** button to save the series that is assigned to the analysis to be terminated as a complete file in the form "SeriesXY". You can also save sample data from a method that is currently in the queue. If the maximum permitted number of series (see "Series templates (page 56)") has been reached, the series is not saved.

For an explanation of the remaining buttons in the **More KF functions** dialog, refer to "The user interface: online dialog for KF(vol) titrations (page 13)").

See also

• Series templates (page 56)

7.6 Method Syntax – Rules for Establishing a Method

A method consists of a sequence of method functions that are executed sequentially when a method is processed. When establishing a method, certain rules (method syntax) must be followed. These fundamental rules are described below:

7.6.1 Possible Number of Method Functions

The following table shows the method functions for the Karl Fischer method types (KF vol, external extraction and Stromboli). The maximum number of functions per method is listed.

Method function	Max. Number per Method for All KF Method Types Except Stromboli	Max. Number for Stromboli Methods
Title	1	1
Sample (KF)	1	14
Titration stand (KF)	1	14
Titration (KF Vol)	1	14
Calculation	40	40
End of sample	1	14
Auxiliary value	30	30

Method function	Max. Number per Method for All KF Method Types Except Stromboli	Max. Number for Stromboli Methods
Blank	-	10
Instruction	10	10
Record	10	14
Drift determination	-	14
Mix time	1	15
Homogenizer	2	-
Standby (Stromboli)	-	1

- Stromboli methods are only available for the model V30.
- The model V20 has restricted functions. The following method functions are not available: "Stromboli", "Homogenizer", "Auxiliary value".

For the "Calculation" method function, in contrast to the V30, a maximum of only three per method is permitted, and only one each for "Instruction" and "Record".

7.6.2 Types and Possible Number of Loops

A method can contain one or more loops, depending on the type of instrument. The ranges of a method through which several samples will pass are defined using a "loop". The method functions before and after a loop are each conducted only once, even if an analysis contains several samples.

The beginning and end of a sample loop are defined by the method functions "Sample" and "End of Sample". The "End of Sample" method function is executed, and the sample loop stopped, only after the last sample in a series.

- Only entire loops can be inserted into, or deleted from, a method.
- Nested loops are not possible.

Different numbers of KF loops are allowed within a method, depending on the method type:

Method type	Maximum number of loops per method
KF vol	1
External extraction	1 (for V30 only)
Stromboli	14 (for V30 only)

7.6.3 Sample loops

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Two templates exist for the "Stromboli (KF vol)" method type; one with and one without a blank value. The template for the KF sample loop with the "Blank value" method function is shown below (for V30 only):

	•	
Sample loop (KF):	Sample (KF)	
	Titration stand (Stromboli)	
	Mix time	
	Titration (KF vol)	
	Calculation	
	Report	
	End of sample	
	Blank	

7.6.4 Method Functions Within a Loop

The method functions that are permitted within a loop between the "Sample" and "End of sample" method functions are limited and depend on the loop type.

The following method functions are essentially permissible within a sample loop in arbitrary order. However, for the method functions Titration Stand, Calculation, and Record, certain rules must be followed:

- The "Titration stand" method function must follow immediately after the "Sample (KF)" function that introduces the loop.
- It is best to insert the method function "Calculation" after the method function that determines the raw results for the calculation.
- The method function "Record" can be only inserted after the method function that generates the results the record should contain.

For KF method

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- Titration Stand (KFStand | Stromboli | External KF stand)
- Mix time
- Homogenizer (for V30 only, not for method types "Stromboli and ext. extraction)
- Titration (KF vol)
- Auxiliary value
- Instruction
- Report
- Calculation

The following method functions are only available for the method type "Stromboli":

- Blank
- Drift determination

7.6.5 Method Functions Outside of a Loop

In addition to the preset "Title" method function, which always appears at the start, additional method functions can be inserted outside a loop depending on the method type:

No selection options are available outside the sample loop for the "KF-Vol" method type.

For the method type "Stromboli KFVol", you can use the following method functions:

- Calculation
- Blank
- Auxiliary value
- Instruction
- Record
- Drift determination
- Standby

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The "Standby" method function must be in the final position in the method.

7.7 Overview of Method Functions

Functions	Explanation	Inside loop	Outside loop
Title	Title and characteristics of the method.	No	Yes
Sample (KF)	Start of a sample loop.	Start of loop	
Titration stand KF stand	Select a titration stand (KF stand , Stromboli)	Yes	No
Mix time	Duration of the mixing process This value is gained from experience. It can be entered specifically for each sample.	Yes	No
Homogenizer	Controls a homogenizer and defines the speed (only for RS homogenizer) and duration of its usage (not for method types Stromboli or ext. extraction).	Yes	No
Titration (KF Vol)	Conducts a Karl Fischer titration.	Yes	No

Overview of method functions: KF Vol

Functions	Explanation	Inside loop	Outside loop
All auxiliary values	Assigns a result or an arbitrary value to an auxiliary value and updates the value stored in Setup.	Yes	Yes
Instruction	Halts the analysis and displays instruc- tions on the screen for the user.	Yes	Yes
Record	Defines the record data to be output to the printer.	Yes	Yes
Drift determination	Determines the drift for Karl Fischer titra- tions (only for method type Stromboli).	Yes	Yes
Blank	Assigns a result or an arbitrary value to a blank and updates the value saved in the setup (only for method type "Stromboli").	Yes	Yes
Calculation	Converts the analysis results.	Yes	Yes
End of sample	Concludes a sample loop.	End of loop	Yes
Standby	Returns the titrator to standby mode on completion of the Stromboli series, so that new series can be started quickly.	No	Yes

Note

The following sequence must be observed within the Karl Fischer loop:

- 1. Sample
- 2. Titration stand
- 3. Drift determination (only for Stromboli)*
- 4. Homogenizer*
- 5. Mix time
- 6. Titration
- 7. Calculation*
- 8 Record*
- 9. End of sample
- 10. Standby*(only for Stromboli)

Functions marked with * are optional.

7.8 Method functions

All definable parameters for the following method functions are described below.

7.8.1 Title

Defines the title and type of a method of a method, and manages properties such as the creation and change date, the author, and whether or not the method is to be protected.

Parameters	Description	Values
ID	Unique ID of the method.	arbitrary (cannot start with "M" followed by a numeric character)
Title	Title of the method.	Arbitrary
Protect	Protects the method against changes and deletion by any user other than the author or the administrator. \circledast	Yes I No
SOP	Standard operating procedure (SOP: (SOP)	None Text Link
SOP-Text	Text for a standard operating procedure (if "SOP" = "text" is selected.)	Arbitrary text
SOP ID	ID for the link to a standard operating procedure (if "SOP" = "link" is selected.)	Arbitrary

7.8.2 Drift determination

You can use the "Drift determination" method function to record the drift for Karl Fischer titration after a specific wait time. This method function can be inserted both within the loop (per sample) and outside the loop (per series).

The memod function. Drift determination, upplies only for memods of the type. Shortboll .		
Parameters	Description	Values
Wait time	Here you can enter the time in [s] until the drift is to be recorded.	01000
Duration	You can enter the length of time in [min] for which the drift deter- mination should last.	0 10
Interval	Defines the drift determination interval, i.e., after how many samples the drift determination will be performed. Appears only if the method function is used within a loop.	0 10

The method function "Drift determination" applies only for methods of the type "Stromboli"

7.8.3 Sample (KF)

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The "Sample (KF)" method function for Karl Fischer titration is subdivided into the subfunctions "Sample", "Concentration", and "Blank value" (only for external extraction). You can define the following parameters:

Parameters	Description	Values
Number of IDs	Defines the number of sample IDs to be defined.	13
ID 1ID 3	The name defined here will be used as the default name for the respective sample on the sample loop. Only appears subject to the settings made in Number of IDs .	Arbitrary
Entry type	Defines whether the sample should be added with a defined mass, defined volume or defined number of pieces. The sample data query will then adjust according to the unit of measurement. Fixed volume or Fixed pieces : The sampling weight, sample volume or number of pieces will be entered as the parameter in this method function and will not be prompted when conducting the method.	Weight Fixed weight Volume Fixed volume Pieces Fixed pieces
Lower limit	Defines the lower limit for the variable entry of data. The unit will depend on the setting for the Entry type parameter. Only appears if for Entry type no "Fixed" values are selected.	[g]: 0 1000 [mL]: 0 10 ⁴ [pcs.]: 010 ⁶
Upper limit	Defines the upper limit for the variable entry of data. The unit will depend on the setting for the Entry type parameter. Only appears if for Entry type no "Fixed" values are selected.	[g]: 0 10 ³ [mL]: 0 10 ³ [pcs.]: 0 10 ⁶
Weight	Weight in [g]. Appears only if Entry type = Fixed weight was selected.	01000
Value	Volume in [mL]. Only appears if for Entry type "Fixed" values are selected.	0104
Pieces	The number of sample(s). Appears only if Entry type = Fixed pieces was selected.	010 ⁶
Weight per piece	The weight in [g] per pirece. Appears only if Entry type = Pieces or Fixed pieces was selected.	0 1000
Density	The density of a liquid sample substance, in [g/mL]. Appears only if Entry type = Weight , Volume , Fixed weight or Fixed volume was selected.	0.0001100
Solvent weight	Quantity of solvent in [g] in which the sample was extracted or dissolved. Only for method type = Ext. Extraction .	01000
Wt. extracted sample	Total weight of sample in [g] which was extracted or dissolved in the solvent. Only for method type = Ext. Extraction .	01000
Correction factor	Any correction factor that can be used in calculations.	0.000110 ⁶

Subfunction: Sample

Temperature	The temperature in [°C] during the analysis. If temperature moni- toring is activated in a titration function, the system will ignore the sample temperature given here.	-20200
Autostart	If activated, KF titration starts following a significant signal increase within 30 seconds after the start of the analysis (not for Stromboli methods). If deactivated, the sample addition must be confirmed before titration can begin.	Yes I No
Analysis start	If Automatic is selected, the analysis begins without any user confirmation if the value falls below the maximum start drift and the defined stability criterion Drift stability /dt and the set temper- ature are reached. If the standby is executed by the method function Standby (only for Stromboli), the automatic start is not performed (affects the 2nd, 3rdseries). If the analysis is started manually, the Stromboli series must be started explicitly in the Standby dialog.	Automatic Manual
Drift stability	Maximum permitted drift difference in [µg/min]. Only for "Stromboli" method type and if Analysis start = Auto- matic is selected.	01000
Entry	 Determines the input time for the sample size. Before: The sample size must be entered before the titration. Arbitrary: The sample size will have to be entered at any time during the titration (no later than when it is used during the calculations). Only appears if for Entry type no "Fixed" values are selected. After addition: You are prompted to enter the sample data once the sample has been added. The sample size - even during the method execution - can be entered later on (however, no later than when required for use in formulas). 	Arbitrary After addi- tion

Subfunction: Concentration

To correctly determine the water content of a sample, the concentration of the titrant should be determined using Karl Fischer water standards (see "Appendix"). The concentration determination is performed using rule and cancellation parameters.

Any predispensing defined in the method is not performed. A defined blank value is also not taken into account in the calculation. Following a concentration determination, the system always switches to standby mode to enable double and multiple determinations.

The concentration determination can be started manually. You can start the concentration determination of the KF titrant from "Standby" of any Karl Fischer (KF) method. You can determine the following parameters:

Parameters	Description	Values
Standard	Select the name of the standard from the standards list.	Select from the stan- dards defined in the set- up.
Entry type	Defines whether the sample should be added with a defined mass, defined volume or defined number of pieces. The sample data query will then adjust according to the unit of measurement. Fixed volume or Fixed pieces : The sampling weight, sample volume or number of pieces will be entered as the parameter in this method function and will not be prompted when conducting the method.	Weight Fixed weight Volume Fixed volume Pieces Fixed pieces
Lower limit	Defines the lower limit for the variable entry of data. The unit will depend on the setting for the Entry type parameter. Only appears if for Entry type no "Fixed" values are selected.	[g]: 0 1000 [mL]: 0 10 ⁴ [pcs.]: 010 ⁶
Upper limit	Defines the upper limit for the variable entry of data. The unit will depend on the setting for the Entry type parameter. Only appears if for Entry type no "Fixed" values are selected.	[g]: 0 10 ³ [mL]: 0 10 ³ [pcs.]: 0 10 ⁶
Weight	Weight in [g]. Appears only if Entry type = Fixed weight was selected.	01000

Value	Volume in [mL].	0104
	Only appears if for Entry type "Fixed" values are selected.	
Pieces	The number of sample(s).	010 ⁶
	Appears only if Entry type = Fixed pieces was selected.	
Mix time	The duration of stirring in [s] with the defined "Stir" speed.	0104
Autostart	If activated, KF titration starts following a significant signal increase within 30 seconds after the start of the analysis (not for Stromboli methods). If deactivated, the sample addition must be confirmed before titration can begin.	Yes No
Entry	 Determines the input time for the sample size. Before: The sample size must be entered before the titration. Arbitrary: The sample size will have to be entered at any time during the titration (no later than when it is used during the calculations). Only appears if for Entry type no "Fixed" values are selected. After addition: You are prompted to enter the sample data once the sample has been added. The sample size - even during the method execution - can be entered later on (however, no later than when required for use in formulas). 	Arbitrary After addi- tion
Lower limit	Defines the lower limit of the concentration limit.	0.1100
Upper limit	Defines the upper limit of the concentration limit.	0.1100

Outside of these limits, the actual concentration is not entered in the setup.

Subfunction: Blank value

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The "Blank value" method function assigns a determined water content to the solvent. The blank can be a fixed value, can be taken from the setup, or can be requested by the system.

The method function "Blank value" is only available for the method type "External extraction".

You can determine the following parameters:

Parameters	Description	Values
Source for blank	Setup : After the blank value is determined, the value and the unit of the blank are transferred to the settings.	Setup Fix value [%] Fix value [ppm]
	Fix: The value defined in the method is used.	Request [%] Request
	Request : The blank value in the relevant unit is requested before each sample.	[ppm]
	The specified blank value is labeled with "B" in the method func- tion Calculation .	
Value	Here you can enter a numerical value. Only appears if for Entry type "Fixed" values are selected.	010 ⁶
Blank	The blank value assigned to the solvent to be determined.	Value from the settings
	You can select a blank value defined in the settings.	
Unit	Defines the unit in which the blank value is calculated and used in a calculation. The unit for calculation with a blank value must be the same as the unit set here. Applies for the Setup option only.	% I ppm
Entry type	Defines whether the sample should be added with a defined mass or defined volume. The sample data query will then adjust according to the unit of measurement. For Fixed weight or Fixed volume , the sample mass and the sample volume are entered as parameters in the method function and not requested in the sequence of the method.	Weight Fixed weight Volume Fixed volume
Lower limit	Defines the lower limit for the variable entry of sample data in [mL] or [g]. The unit will depend on the setting for Entry type parameter. Applies only for Entry type = Weight and Volume .	01000

Upper limit	Defines the upper limit for the variable entry of sample data in [ml] or [g]. The unit will depend on the setting for the Entry type parameter.	01000
Weight	Applies only for Entry type = Weight and Volume. Weight in [g]. Appears only if Entry type = Fixed weight was selected.	01000
Volume	Volume in [mL]. Appears only if Entry type = Fixed volume was selected.	01000
Density	The density of the liquid sample in [g/mL] for Entry type = Vol-ume or Fixed volume .	01000
Mix time	The duration of stirring in [s] with the defined "Stir" speed.	0104
Autostart	If activated, KF titration starts following a significant signal increase within 30 seconds after the start of the analysis (not for Stromboli methods). If deactivated, the sample addition must be confirmed before titration can begin.	Yes I No
Limits	Determines whether limits should be taken into account for acquisition of a value. If the value is outside these limits, the value is not transferred to Setup.	Yes No
Upper limit	Defines the upper blank limit. Appears only if "limits" = "yes" was selected. Outside these limits, the blank value is not entered in the setup.	010 ⁶

See also

• Blanks (page 33)

7.8.4 Titration stand

You can use the following parameters to specify the relevant titration stand.

The "KF stand" titration stand is available for Karl Fischer (KF) methods, and the "Stromboli TTL stand" is available for KF Stromboli methods.

Parameters	Description	Values
Titration stand	Defines which titration stand is to be used.	List of available titration stands
Source for driff	 Online (not for Stromboli): For calculations, the drift determined in the online procedure is used. Determination: The drift saved in the titration stand setup for the KF titration stand selected in the method is used. Fix value: The drift value determined in the method. Request: The drift value is requested before each sample or Stromboli series. 	Online Determination Fix value Request
Drift	Value of the drift in [µg/min].	01000
Max. start drift	The maximum drift for which a sample determination can still be started.	01000
Oven tempera- ture	Temperature setting in [°C] for the "Stromboli" oven sample changer. Only for Titration stand = Stromboli TTL .	50300

7.8.5 Homogenizer

The method function "Homogenizer" applies only for Karl Fischer titrations when not using the Stromboli oven sample changer, and does not apply for the method type "External extraction". The TTL homogenizer can be switched on for a defined period of time.

For the TTL homogenizer, you can also determine the stir time:

Parameters	Description	Values
Duration	Duration in [s].	1104

7.8.6 Mix time

You can use the "Mix time" method function to define the stir time in [sec] for Karl Fischer titration. This value is obtained from experience and can be entered individually for each sample.

The stir speed, however, is entered in the method function "Titration" using the "Stir" parameter. This applies for the whole method.

Parameters	Description	Values
Duration	Duration in [s].	1104

7.8.7 Titration (KF vol)

The Karl Fischer titration is performed using the "Titration (KF vol)" method function. This function contains subfunctions, which each have their own parameters.

You can determine the relevant parameters for the following subfunctions:

Parameters	Description	Values
Titrant	Select a titrant from the list of the defined titrants.	Titrant list
Subfunction: Se	nsor	
Parameters	Description	Values
Sensor	Select a sensor from the list. The list depends on the sensor type selected in Type .	List of available sensors
For Karl Fischer	titrations, only polarized sensors can be selected.	
Ipol	Ipol is the polarization current, in [µA], for the voltametric indica- tion.	0.024.0
Subfunction: St	ir	
Parameters	Description	Values
Speed	Defines the stirring speed in [%].	0100
Subfunction: Pr	edispensing	
Parameters	Description	Values

Parameters	Description	Values
Mode	Specifies the type of addition: Volume : predispenses a specific volume. None : does not predispense.	Volume None
Volume	The volume to be predispensed in [mL]. Only for Mode = Volume .	0.00011000 Auxil- iary value Formula
Wait time	Defines a waiting time, in [sec]. After predispensing or, if Mode = None , before the start of titra- tion.	032000

Subfunction: Control

The maximum dosing rates are dependent on the size of the burette. The user is able to edit the entire value range. When Start is pressed, the system then checks whether the entered values are actually possible with the current burette size.

Burette size [ml] Maximum dosing rate [mL/min]
1	3
5	15
10	30
20	60
Daramotore	Description

Parameters	Description	Values
End point	End point in [mV] of the Karl Fischer titration and the standby	-2000 2000
	titration.	

Control band	The value in [mV] defines the width of the control band. Outside the control band, the system will titrate with the maximum dis- pensing rate. The control band allows the dynamic behavior of the controller to be influenced. Reducing the control band causes a more aggressive control behavior, while increasing the control band gives a gentler control behavior. When the measurement curve reaches the control band, the titrator slows down the addi- tion of titrant to approach the end point cautiously.	0.12000
Dosing rate (max)	The maximum dosing rate in [mL/min].	0.00160
Dosing rate (min)	The minimum dosing rate in [µL/min].	1104
Start	Cautious or normal start of a Karl Fischer titration.	Cautious Normal

Subfunction: Termination

Parameters	Description	Values
Туре	Termination of titration following defined drift and if the value falls below the end-point value (EP). Drift stop relative: Actual drift stop value = online drift + drift Drift stop absolute: Actual drift stop value = drift Delay time: Termination after a delay time below the EP.	Drift stop relative Drift stop absolute Delay time
Drift	The drift value in [µg/min] for the termination criterion drift stop relative or drift stop absolute.	1.0 10 ⁶
Delay time	Time in [s] from the time the end point is first reached until the termination of the titration.	06000
Min. time	Titration is not to be terminated before this time in [s] is reached (exception: the maximum volume has been reached).	010 ⁸ Auxiliary value
Max. time	Maximum duration of the titration (without post-consumption measurement).	0…10 ⁸ I ∞ I Auxiliary value
At Vmax	Titration is terminated at the latest when the maximum volume is reached (without post-consumption measurement), even if the minimum time has not yet elapsed.	Yes No



The titration is terminated when the maximum time, the maximum volume and the drift stop are reached.

7.8.8 Auxiliary value

This method function assigns a result or arbitrary value to an auxiliary value.

Parameters	Description	Values
Name	Specify a descriptive name of your choice.	Arbitrary
Formula H=	Here you can enter a formula that will be used to convert the result of the sample loop to the auxiliary value. You can also enter a number or an auxiliary value.	Formula (see "Evalua- tion and calculation (page 71)") Auxiliary Value Number
Limits	Determines whether limits should be taken into account for acquisition of a value. If the value is outside these limits, the value is not transferred to Setup.	Yes I No
Interruption out- side limits	Determines whether the method should be interrupted if a value lies outside the defined limits (only appears if the "Limits" para- meter has been activated). A message (which must be acknowledged) appears advising that the process has been interrupted during the time that the message is displayed.	Yes I No
Lower limit	Appears only if "limits" = "yes" was selected.	-10 ⁸ 10 ⁸
Upper limit	Appears only if "limits" = "yes" was selected.	-10 ⁸ 10 ⁸

7.8.9 Blank

This method function assigns a result or an arbitrary value to a blank value, including units (only for methods of type "Stromboli").

Parameters	Description	Values
Name	Specify a descriptive name of your choice.	Arbitrary
Value B=	Here you can enter a formula that will be used to convert the sample loop result to the blank. You can also enter a number or an auxiliary value.	Formula (see "Evalua- tion and Calculation (page 71)") Auxiliary Value Number
Unit	The units in which the blank is specified.	Arbitrary
Limits	Determines whether limits should be taken into account for acquisition of a value. If the value is outside these limits, the value is not transferred to Setup.	Yes No
Interruption out- side limits	Determines whether the method should be interrupted if a value lies outside the defined limits (only appears if the "Limits" para- meter has been activated). A message (which must be acknowledged) appears advising that the process has been interrupted during the time that the message is displayed.	Yes No
Lower limit	Appears only if "limits" = "yes" was selected.	-10 ⁸ 10 ⁸
Upper limit	Appears only if "limits" = "yes" was selected.	-10 ⁸ 10 ⁸

7.8.10 Instruction

Interrupts the analysis and outputs an on-screen instruction to the user. Either the user has to confirm the instructions or they will disappear automatically after a certain period.

Parameters	Description	Values
Instruction	The text of the instructions to be output to the display. This text can also contain a formula or auxiliary values, enclosed in percent symbols. Example: "Add %VEQ*m/z% g".	Arbitrary, including enclosed formula (con- trol characters: %)
Continue after	Confirmation : The analysis will continue as soon as the user confirms the instructions. Time interval : The analysis is continued after the defined time period has elapsed.	Confirmation Time interval
Time interval	The time period, in [sec], during which the analysis is terminated and the instructions are displayed on the screen. Only appears if Continue after = Time interval is selected.	010 ⁶
Print	If selected, the instructions will be output to a connected printer.	Yes No

7.8.11 Calculation

For converting the titration results.

Parameters	Description	Values
Result type	If the result type "Automatic" is selected, a predefined result from the proposal list is used. The parameters "Result", "Unit", "Formu- la", and "Constant" are adjusted automatically in accordance with the entry type selected in the method function sample (KF) and cannot be changed. If "User defined" is selected, all parameters can be edited. You can also select a predefined result type from the results proposal list.	Automatic I User defined

- The "Result type" parameter is not available for KF titrators in the 20 series, and is set to "Automatic".
- "Result type" is hidden for the "Stromboli" method type and is fixed to "user-defined".

automatically set the "Result unit", "Formula" and "Constant" parameters. But you will be able to make any changes to them that you would like without having the system adjust the other	Results list Arbitrary
parameters. You can also enter any number.	

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Result unit	The unit of the result. (Is not automatically adjusted after changes to "Formula" or "Constant".) You can also use " Proposal " to	Device list Arbitrary
Formula	select from a predefined suggestion list. The formula for calculating the result. You can use " Results pro-	Formula list I Arbitrary
Constant	posals " to select from a predefined suggestion list. Definition of the C constant which can be used in the calculation. The constant can itself be a formula. You can also use "Proposal" to select from a predefined suggestion list.	Constants list Arbitrary
Decimal places	The number of decimal places for the result.	06
Result limits	Defines whether limits should be observed for the result. If this function is activated, there will be a message in the record if the result falls outside the defined limits.	Yes I No
Lower limit	Defines the lower result limit. Appears only if "result limits" = "yes" was selected.	-10 ⁸ 10 ⁸
Upper limit	Defines the upper result limit. Appears only if "result limit" = "yes" was selected.	-10 ⁸ 10 ⁸
Interruption out- side limits	Determines whether the method should be interrupted if a value lies outside the defined limits (only appears if the "Limits" para- meter has been activated). A message (which must be acknowledged) appears advising that the process has been interrupted during the time that the message is displayed.	Yes I No
Record statistics	Specifies whether statistics should also be issued with the results in the report along with the result. The statistics are not printed, if in the method function "Protocol" the parameter "Results" = "No" is selected.	Yes I No
Extra statistical functions	You can use this parameter to switch on extra statistical func- tions. For example, this will allow you to define a maximum val- ue for the relative standard deviation which, if violated, will cause individual results to be listed in the record. The settings for this parameter will only be taken into consideration if the "Calcula- tion" method function is used within a sample loop.	Yes I No
Multiple determi- nation	This function helps you do statistical evaluations of sample groups. The sample groups are defined with the "Number of samples" parameter. Appears only if "additional statistic functionalities" = "yes" was selected.	Yes I No
Max. srel	If the relative standard deviation for the calculated result is above the "Max. srel", the system will output a corresponding message in the record. Appears only if "Extra statistical functions" = "Yes" was selected (and if available "Multiple determination" = "Yes").	0100
Number of sam- ples	Defines the sample groups for a multiple determination. For example, a value of 3 means that the system will run a statistical evaluation on three consecutive samples. Appears only if "Extra statistical functions" and "Multiple determi- nation" = "Yes" were selected.	29
Interruption above max. srel	Specifies whether an analysis series should be terminated as soon as the relative standard deviation of a sample group within a multiple determination is above the "Max. srel". Appears only if "Extra statistical functions" and "Multiple determi- nation" = "Yes" were selected.	Yes I No
Record	If "yes" is selected for "Record", the multiple determination func- tion will create a record listing the groups after a double determi- nation whose relative standard deviation lies above a "Max. srel" number defined in the method. Appears only if "Extra statistical functions" and "Multiple determi- nation" = "Yes" were selected.	Yes I No

7.8.12 Record

This method function defines the type and scope of the data to be output for a report using the printer (see "Peripherals>Printer").

If the method function "Record" is placed within a sample loop, the record will include all previous method functions within the current sample loop.

If the method function "Record" is placed outside of a sample loop, the record will include all previous method functions listed after the last sample loop in the method procedure. A few settings are not available outside of a loop.

Parameters	Description	Values
Overview	States whether or not a short summary of the results should appear at the top of the protocol.	No I Per sample I Per series out of loop: No I Yes
Results	The results from the "Calculation" method functions. Any statistic selected will be recorded after the last sample of a series or multiple determination.	within loop: Per Sam- ple Per Series No Out of Loop: No Yes
Raw results	The raw results produced during the determination	within loop: Per Sam- ple Per Series No Out of Loop: No Yes
Measured value table	The table of measured values of the current sample (not avail- able out of loop).	Yes I No
Sample data	The sample data of a sample loop. (Not available outside of loop)	Per sample Per series No
Resource data	All data in the setup regarding the resources used in the method.	Per Sample Per Series Non Per Sample Per Series No
E – V	Titration curve of the current sample. The potential is plotted against the volume (not available out of loop).	Yes I No
E – †	Titration curve of the current sample. The potential is plotted ver- sus the time (not available outside of loop).	Yes I No
V – †	Titration curve of the current sample. The volume is plotted ver- sus the time. (not available outside of loop)	Yes I No
H2O -†	Titration curve of the current sample. The water content is plotted against the time (not available out of loop).	Yes I No
Drift-t	The titration curve "Drift" against "Time" (not available out of loop).	Yes I No
H2O-t & Drift-t	Two overlaid titration curves " $H_2O - t$ " and "Drift-t" of the current sample (not available out of loop).	Yes I No
V-t & Drift-t	Two overlaid titration curves "V $-$ t" and "Drift-t" of the current sample (not available out of loop).	Yes I No
Method	Printout of the method used.	No I Yes
Series data	All data from the series run.	No I Yes

7.8.13 End of sample

The "End of sample" method function closes a sample loop. A sample loop refers to the range of a method through which a sample series will pass for each sample. The commencement of a sample loop is specified using the "Sample" method function.

Parameters	Description	Values
Open series	Determines whether the subsequent method functions are processed after the method function "End of sample", or if the titrator returns to Standby mode.	Yes No

i The parameter "Open series" is only available for Karl Fischer titration without the Stromboli oven sample changer. If "Open series" is set, the titrator enters "Standby" mode at the end of the analysis and the method remains active. If "Open series" is not set, the method is ended after the final sample.

7.8.14 Standby

The "Standby" method function can only be inserted in methods of the type Stromboli after the method function "End of sample". This method function determines whether the method is terminated at the end of the series, or if the method remains active and then enters standby mode for the first loop.

7.8.15 Hidden method functions

The following hidden functions exist for Karl Fischer methods: Pretitration and standby.

When a Karl Fischer method is started, the system first performs a pretitration. The titrator then switches to Standby mode. The system switches automatically between Standby and Pretitration. The switch criterion is the drift value.

If the pretitration lasts longer than 30 minutes, a system message is displayed informing you that the pretitration cannot be ended because the drift value is too high. You can end the pretitration, and then cancel the method or series or restart the pretitration.

8 Series templates

Series templates are used for a sequential series of samples processed using the same method (templates for sample series (SAS)).

Using series templates, you can group multiple (up to 120) individual samples into one **sample series**, so that all the samples in the series can be analyzed consecutively using a defined method.

The list of series templates shows you all the series templates defined in the titrator. Each series template is shown in the list with its type (SAS = Sample series) and name.

If you select a series template from this list, you can change its parameters or delete the entire template.

- You can create a shortcut on the Home screen for all series templates.
- A maximum of 60 sample series can be saved in the titrator.
- Series templates cannot be created for concentration and blank samples.

8.1 Sample series

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Select the **New** button in the **Series templates** dialog to create a new series template. The following parameters will be available:

Parameters	Description	Values
Sample series ID	Here you can assign any ID to the sample series.	Arbitrary
Method ID	Here you can select the Method ID for the relevant method.	Method list
Comment	ment You can enter a brief comment about the series.	
Number of sam- Defines the number of samples to be analyzed.		1120
ples		

If you select a template of type "Stromboli", the "Loop" and "No. of samples" parameters are repeated according to the number of loops in the assigned method (maximum 14).

During the creation of a sample series, you can use the **Samples** button to go to the loop list (if the assigned method includes more than one loop) or go directly to the sample list (if the assigned method only contains one loop).

From the loop list, you can select a loop to go to the sample list for that loop.

8.2 Sample parameters

The sample list, which can be opened by choosing **Samples** in the series template, displays all samples of a loop with a number, the first ID, and the sample size (depending on entry type - see "Method functions: Sample" (page 46)). You can also edit the samples here.

Series IDs must be unique, although sample IDs do not have to be.

If you want to select an entry from the list or use the **New** button to create a new list entry, you can define the following parameters for each sample:

Parameters	Description	Values			
Number	Defines the number of the sample.	1120			
A number is auton	natically assigned when you create a new list entry.				
ID	A user-defined name for the ID of the sample, in accordance with Arbitrary the "Sample" method function.				
Sample size	You can enter the sample size here. $01000 [g]$ For fixed entry types, this field only appears as an info field. $010^6 [pcs]$				
Density [g/mL]	The density of the sample for the entry types "Weight", "Fixed weight", "Volume" and "Fixed volume".	0.0001100			
Weight per piece	The weight in [g] per pirece. Appears only if Entry type = Pieces or Fixed pieces was selected. 0 1000				
Solvent weight	Quantity of solvent in [g] in which the sample was extracted or dissolved. Only for method type = Ext. Extraction .	01000			

Wt. extracted sample	Total weight of sample in [g] which was extracted or dissolved in the solvent. Only for method type = Ext. Extraction .	01000		
ID 2ID 3	The name defined here will be used as the default name for the Arbitrary respective sample on the sample loop. Only appears subject to the settings made for Number of IDs .			
Comment	You can enter a brief comment about the series.	Arbitrary		
Correction factor	Any correction factor that can be used in calculations.	0.000110 ⁶		
Temperature	The temperature in [°C] during the analysis.	-20200		

For entering the sample parameters, particularly for numerous samples, the titrator provides you with assistance in the entry windows of the "ID 1" and "Sample size" parameters:



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These extra icons are a quick, direct way to jump to the entry window of the previous sample or next sample.

9 Analysis Sequences

9.1 Starting an Analysis

An analysis, whether it be a single or multiple determination, can be started on the titrator in several different ways:

- 1. By choosing the following options:
 - Start from the method editor
 - Start from "Home"
 - Start from the "Series" dialog
- 2. Using a user-specific shortcut or a direct shortcut from "Home".

When you create a shortcut with **AddToHome** (see "Function description>The user interface:Start analysis dialog"), the following parameters are available:

Parameters	Description	Values		
Description	Any name for the shortcut.	Arbitrary		
Immediate start	The method, series, or manual operation can be started immedi- ately. This enables you to start the analysis without any interfer- ing dialog.			
Homescreen position	You can select the free position for the shortcut on the Home- screen.	112		

After you create the shortcut, it appears in the selected position in "Home", from where you can select it by tapping the touchscreen.

When you start an analysis, whether by using a button or with a shortcut, the system always opens the **Start analysis** dialog (see "Descripton of Functions > The User Interface > The Start Analysis Dialog"). The only exceptions are direct shortcuts ("Immediate start" = "Yes"), whose selected settings permit a direct start.

At the start of an analysis, you can still make changes to various settings in the **Start analysis** dialog. It is therefore possible, for example, to modify the sample size and define the number of samples to be determined.

If the analysis you want to start is a single determination, you can enter the sample size or sample ID directly as a parameter in the **Start analysis** dialog.

In general, the sample data can be entered for each individual sample using the **Samples** button in the **Start analysis** dialog. In the **Sample data** dialog that opens when you choose this button, a list of the individual samples is displayed.

In addition, the status is displayed for every sample (regardless of the loop type) in the **Sample data** dialog. The following status levels can be assigned to a sample:

- Idle: The sample is not yet running and the sample data can still be edited
- Running: The sample is running but the sample data can still be edited
- Active: The sample is running and the sample data can no longer be edited
- Done: Done the sample has run and concluded and the sample data can no longer be edited

If you select a sample, you can define the following sample data.

Parameters	Description	Values
ID 1	The ID for the first or only sample of an analysis.	Arbitrary
Sample size	You can enter the sample size here. For fixed entry types, this field only appears as an info field.	01000 [g] I [mL] 010 ⁶ [pcs.]
Density	You can enter the sample's density, in [g/mL], here. Does not appear for the Entry type = Pieces and Fixed pieces .	0100
Weight per piece	The weight in [g] per pirece. Appears only if Entry type = Pieces or Fixed pieces was selected.	0 1000
Solvent weight	Quantity of solvent in [g] in which the sample was extracted or dissolved. Only for method type = Ext. Extraction .	01000

Wt. extracted sample	Total weight of sample in [g] which was extracted or dissolved in the solvent. Only for method type = Ext. Extraction .	01000			
ID 2ID 3	The name defined here will be used as the default name for the Arbitrary respective sample on the sample loop. Only appears subject to the settings made for Number of IDs .				
Comment	You can enter a brief comment about the series. Arbitrary				
Correction factor	Any correction factor that can be used in calculations.	0.000110 ⁶			
Temperature	The temperature in [°C] during the analysis. If temperature moni- toring is activated in a titration function, the system will ignore the sample temperature given here.	-20200			

You can enter the following parameters in the **Start analysis** dialog, depending on the type of analysis to be started and the resources used:

Parameters	Description	Values			
Number of sam-	Defines the number of samples to be analyzed.	1120			
ples					
ID 1	e ID for the first or only sample of an analysis. Arbitrary				
Sample size	You can enter the sample size here.	01000 [g] I [mL]			
	For fixed entry types, this field only appears as an info field.	010 ⁶ [pcs.]			

• All the parameters that can be edited in the **Start analysis** dialog or the sample data dialog will overwrite the settings defined in the method for the same parameters.

- All non-editable parameters that are displayed as an info field are only shown for orientation purposes and list the settings from the method.
- If the sample size must be entered before the analysis but the user does not do so, the user will be required to enter it immediately before the start of the analysis.

See also

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• Sample (KF) (page 46)

9.2 Analysis sequence steps

9.2.1 KF Analysis sequence

The following describes the sample analysis sequence for a Karl Fischer titration using the "KF stand" and "Stromboli" titration stands.

When a KF method is started, the system first performs a pretitration.

The pretitration always takes place to ensure that the Karl Fischer reagent is in a water-free state.

When a particular drift value is reached, the system switches to Standby mode (see "Function description", the user interface Online dialog"). Standby mode is used to stabilize the potential as much as possible around the end point.

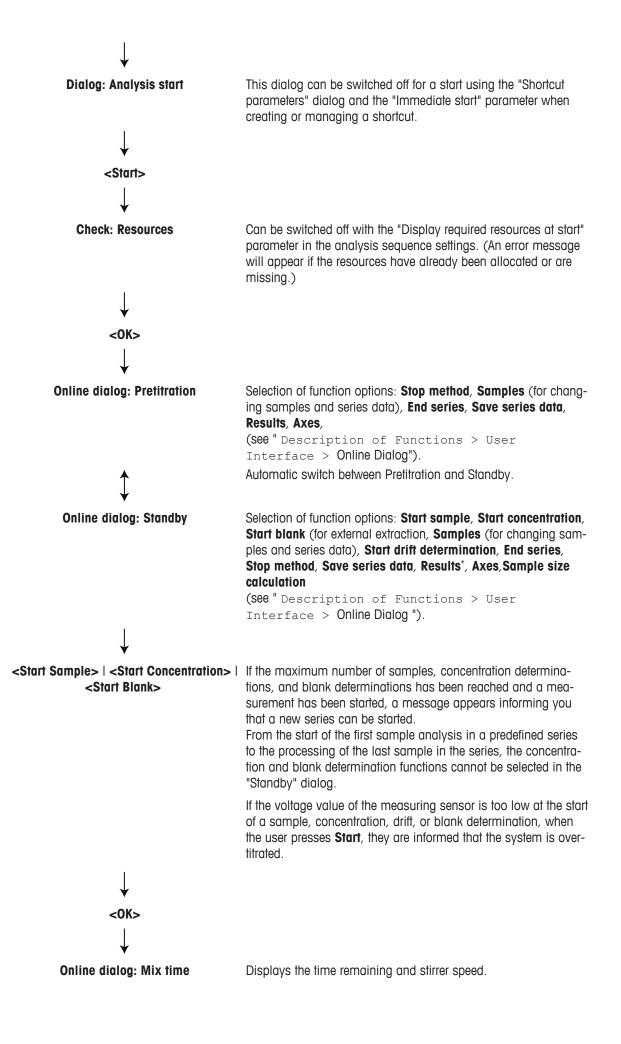
The system switches automatically between Pretitration and Standby. The determined drift value is used as the criterion for switching between the different modes. If the pretitration is not finished, the system issues a message after 30 minutes informing you that the pretitration cannot be completed because the drift values are too high. You can then end the pretitration, thus terminating the method or series, or restart the pretitration.

On the other hand, if the system switches from Standby to Pretitration during a parameter request, or if the maximum start drift is exceeded, you can end data entry and save the data by choosing **OK**.

You can start an analysis from Standby mode.Once the analysis has finished, the KF method returns to Standby mode. You have the option to start another sample analysis. A series analysis can be subsequently expanded, i.e., you can start a series with a defined number of samples, but whether further samples in the current series are to be started remains open ("Open Series", see method function: End of sample. Once the loop is complete or **Terminate series** is selected, the analysis returns to Standby mode. If the "Open series" parameter is not set, the series is automatically ended after the final sample.

The start of a new KF method or a spontaneous concentration or blank determination automatically terminates the current series.

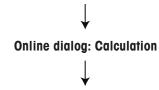
<Start>



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Online dialog: Titration (KF Vol)

You have the following options during an analysis: Cancel the sample, concentration, drift, blank determination or the method, you can modify sample data, view results or measured values, save a series, or specify the axes of the measurement diagram.



Online dialog: Standby

*During Standby or Pretitration mode, you can access the results of the current determination type (sample, concentration, blank determination). Here you can perform the following actions (see "Results"):

- Recalculate (can only be changed for individual samples, and not for a whole loop)¹
- Exclude samples¹
- Perform outlier test¹
- Results
- Display statistics
- Undo changes

¹For titrations using the "Stromboli" oven sample changer, these functions are only available at the start of the series or in Standby mode and with Analysis start "manual".

Drift determination

There are several different ways to determine the drift:

- Using the "Drift determination" method function. Here you can enter the duration of determination. The method function can be inserted outside the loop (in determination per series) or inside the loop (in determination per sample) (only for KF methods of the type "Stromboli").
- Spontaneous drift determinations: The drift can be determined from Standby mode of any KF method. The drift determined here is used if the "Source for drift" parameter in the "Titration stand" method function is set to "Setup".
- 3. Online drift determination: The drift that is constantly determined during standby operation is the current and correct drift value which is used in the calculations. To enable this, the "Source for drift" parameter in the "Titration stand" method function must be set to "Online" (see "Method functions: Titration stand").

Concentration determination

The concentration determination of the titrant can only be started spontaneously, and hence only from "Standby" mode.

When a concentration determination is started, as with a sample determination, this opens an "Open series". After every determination, the mean value of the open concentration determination series is assigned to the titrant in Setup. The open concentration determination series is ended by the start of a sample and blank determination (and vice versa). In general, the open series can be terminated by choosing **End series**, **Stop method**, or **Reset**. A spontaneous drift determination, however, does not terminate the series.

9.2.1.1 Series analyses with the "Stromboli" oven sample changer

Before starting a Stromboli method, the pump must be switched on and the set temperature specified. Every Stromboli method begins in the Start position (beaker is in the drift position). In this position, the pretitrations, the manual and automatic drift determinations, and the concentration determinations (only for KF VoI) are performed. The pretitration already takes place during the heating process.



The heating and the pump remain active in Standby mode. When a Stromboli method is active, the set temperature is controlled automatically.

After a series analysis is started via **Start** in the **Standby** dialog or because Analysis start = "automatic" is set, the series is processed automatically. After each sample is processed, the next sample is analyzed without prompting. To enable automatic analysis start, the following conditions must be fulfilled:

- The set temperature must be reached.
- The online drift must be smaller than the maximum start drift.
- The system must not be overtitrated.
- The drift stability must be fulfilled.

Once the titration is complete, Standby mode is active until the end of the loop in the current sample beaker. If the maximum start drift value is exceeded during this time, the sample changer returns to pretitration (drift position). The analysis is then continued automatically when the maximum start drift value is reached. If the last sample in a loop has been processed and further loops still remain, the current position (sample or drift beaker) is maintained until the next sample is approached. The current drift is reviewed before the start of the next sample. The following is a description of the behavior when particular actions are performed:

Analysis start

Each analysis starts in position 1, immediately after the "Driff" position.

When using Stromboli, no positions can be controlled. The sample changer always moves forward by one position and performs an analysis or a blank determination in that position. Stromboli only returns to the "Driff" position to perform a drift determination.

Cancel method

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The method is terminated with no further action. The temperature control and the pump are switched off immediately. Stromboli returns to the "Drift" position.

Before actually stopping the process, the system displays a system message asking you to confirm the termination.

Drift determination

The manual drift determination and the drift determination via method functions always take place in the "Drift" position. After manual drift determination, the sample changer remains in this position. In contrast, with drift determination using the method function, the sample changer moves to the next planned sample position.

Concentration determination

The manual concentration determination is performed in the drift beaker. The heating and pump remain active.

Canceling the drift determination or concentration determination

Because the drift and concentration determination is performed in the drift beaker, terminating the process has no effect on the actions of the sample changer. The standby titration is started again.

Pressing Reset

If the Reset button is pressed while a KF analysis or manual operations are active, all Karl Fischer methods and manual operations are terminated. For Stromboli, this means that the pumps are switched off, the titrator returns to the resting position (via the drift beaker), and the heating is turned off. If a KF analysis (method or sample series) is terminated, the system continues with the pending analyses from the list. The sample data for the terminated samples or sample series (such as weigh-ins, etc.) are still saved with the results.

9.2.1.2 External extraction

For the Karl Fischer "External extraction" method type, there is no automated sample analysis. Each sample in a series must be started individually from "Standby" mode.

If the "Open series" parameter is set (see "Method Function: End of Sample"), additional samples can be added after a series has been processed. If "Open series" is not set, the series is completed after the specified number of samples, and the method is stopped.

Manual blank value determination can be carried out from "Standby" mode..

9.2.1.3 Switching between determination types

You can determine statistics for sample, blank, and concentration determinations. If you switch between two determination types during an analysis, the determination series is ended. The system displays a message. You can then decide whether to choose **Cancel** to return to Standby mode, or choose **OK** to start the selected determination.

When you end a determination series, the relevant results are not deleted. The results memory of this determination type is not deleted and refilled until a new determination type is started and if results are available. The other determination types are not deleted and no new series entry is created in the results memory.

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For example: If you carry out multiple blank determinations and then carry out a concentration determination, the blank value statistics are terminated. The next time a blank determination is started, the memory for the blank determination is deleted and filled with new blank value data.



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There are no mean values for drift determinations, each determination generates a new drift value that is transferred to Setup.

9.2.1.4 Analysis records

Analysis records are the printouts specified by the user in the "Records" method function (see "Method functions: Records"). When a manual drift, concentration, and blank determination is created, the system creates a separate expression.

Printouts per series

Printouts "per series" are created if the user presses the **End series** button or if a series is terminated via **Start** concentration or **Start blank**.

An open series does not count as finished until **End series** has been selected or a series is ended by choosing **Start concentration** or **Start blank**. After confirming the corresponding message, the "Record" method function can be used to print out all parts of the record defined for each series.

Printouts per sample

The printouts are created for each sample when the "Records" method function is processed.

9.2.1.5 Replacing the titrant

The reagent in the beaker can be replaced when a certain number of samples is reached, if the solvent capacity is used up, or after a defined period of time (Intelligent Solvent Controlling). This causes a brief interruption in the series sequence.

The titrant replacement procedure is semi-automatic, i.e. the user has to initiate the replacement.

10 Results

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The Results dialog can be accessed directly from Home using the relevant button.

- In the V2O, only the results of the last analysis are saved, in the V3O, the results of the last two analyses can be selected by pressing the **Select series** button.
- When you start a new analysis, you will lose the results from the previous analysis.
- You can view results immediately after they have been recorded (for the same determination type: sample, concentration, or blank determination).

You can use the various buttons in the "Results" dialog to call up different functions. You can, for example,

- view all results of the last analysis (V20) or the last two individual samples or series (V30).
- add a supplementary result calculation both within the loop (for all samples in the analysis), and outside a loop (once for the entire analysis).
- view statistics, perform an outlier test and if necessary, exclude samples from the statistics.
- perform recalculations for results in which certain raw data (e.g. sample sizes) have to be adapted retrospectively for a single sample or for all samples in a series.
- view and print the status and the calculated results of each individual sample.

Results are retained until new results are generated by methods. The results of the "older" of the two sample series are replaced.

In KF methods, the **End series** function can be used to generate a new result entry during the execution of a method. After the series is finished, the original sample parameters are used, i.e. the changes made in the **Start analysis** dialog or later are not taken into account. A new series is entered in the results.

In Karl Fischer (KF) titrations, the results are divided into the three determination types "Sample", "Concentration", and "Blank". All options for managing results only have an effect on the determinations of one particular determination type.

While an analysis is running, only the current determination type is available.

If a determination type ("Sample", "Blank", or "Concentration") is started for a second time, existing data are overwritten.

All the changes performed on the saved results can be reversed with the "Undo changes" button.

Changes to results are indicated by an asterisk in the record. Example: VEQ*.

10.1 Result proposal lists for Karl Fischer titration

Predefined results for volumetric Karl Fischer titration are described below. You use the **Results proposals** button in the **Add result** dialog to open the result proposal lists.

Use the **Results proposals** button in the **Add result** dialog or in the **Calculation** dialog to reach the results proposal lists. If a results proposal is selected from the list, the parameters are set as follows: "Results", "Result unit", "Formula", and "Constant" parameters are automatically filled and cannot be changed (depends on titrator type).

The proposal lists are filtered by method type and entry type. "Result" and "Result unit" define the formula with the help of the entry type chosen in the "Sample KF" method function. If the entry type changes, the formula is modified if the result type is "automatic" (if this is possible for the unit in question). If no formula exists for the newly selected entry type, the system will detect this during method validation when the entry type is saved. The blank value of the "Sample (KF)" method function - "Blank" subfunction - is used for the blank values of the external extraction/solution.

The formulas listed below are result proposals.

Karl Fischer titration vol method type

Result	Unit	Replace "m" according to entry type	Formula R =	Constant
Consumption	ml		R = VEQ	C = 1
Mean con- sumption	µl/min		R=VEQ*1,000/TIME	C = 1

Results proposal list for KF vol

Result	Unit	Replace "m" according to entry type	Formula R =	Constant
Titration dura- tion	min		R=TIME	C = 1
Total water content	hð		R=CW	C = 1
Content	mg		R=(VEQ*CONC-	C = 1
	μg		TIME*DRIFT/1,000)*C	C = 1,000
	%	g	R=(VEQ*CONC-	C = 0.1
	ppm	ml: m*d pc: m*wp	TIME*DRIFT/1,000)*C/m	C = 1,000
	g/kg			C = 1
	mg/g			C = 1
	mg/ml	ml		C = 1
	g/ml	g: m/d		C = 0.001
	mg/pc	PC		C = 1
	µg/l	ml		C = 1,000,000
		g: m/d		
	µg/ml	ml g: m/d		C = 1,000

Method type Stromboli KF vol

Results proposal list for Stromboli KF vol

For the "Stromboli KF vol" method type, the following results are available in addition to the "Consumption", "Mean consumption", "Titration duration" and "Water content (total)" result types:

Result	Unit	Replace "m" according to entry type	Formula R =	Constant
Stromboli blank value	hð		R=(VEQ*CONC- TIME*DRIFT/1,000)*C	C = 1,000
Content blank value	mg		R=(VEQ*CONC-	C = 1
compensated (B in µg)	hð		B[Blank Stromboli]/1,000- TIME*DRIFT/1,000)*C/m	C = 1,000
	%	g	R=(VEQ*CONC-	C = 0.1
	ppm	ml: m*d	B[Blank Stromboli]/1,000-	C = 1,000
	g/kg	pc: m*wp	TIME*DRIFT/1,000)*C/m	C = 1
	mg/g			C = 1
	mg/ml	ml		C = 1
	g/ml	g: m/d		C = 0.001
	mg/pc	PC		C = 1
	µg/l	ml g: m/d		C = 1,000,000
	µg/ml	ml g: m/d		C = 1,000

Ext. extraction method type / KF vol solution

Results proposal list for external dissolution / extraction

Result	Unit	Replace "m" according to entry type	Formula R =	Constant
External disso- lution (B in %)		g ml: m*d pc: m*wp	R=C*[(msol+mext)/mext]- B*msol/mext	C=(VEQ*CONC- TIME*DRIFT/1,000)*0.1/m

Result	Unit	Replace "m" according to entry type	Formula R =	Constant
External disso- lution (B in ppm)	ppm	g ml: m*d pc: m*wp	R=C*[(msol+mext)/mext]- B*msol/mext	C=(VEQ*CONC- TIME*DRIFT/1,000)*1,000/m
External extraction (B in %)	%	g ml: m*d pc: m*wp	R=100/(100-C)*(C*msol/mext - B*msol/mext)	C=(VEQ*CONC- TIME*DRIFT/1,000)*0.1/m
External extraction (B in ppm)	ppm	g ml: m*d pc: m*wp	R=pw(6)/[pw(6)- C]*(C*msol/mext- B*msol/mext)	C=(VEQ*CONC- TIME*DRIFT/1,000)*1,000/m

10.1.1 Internal calculations

Result	Unit	Replace "m" according to entry type	Formula R =	Constant
Concentration (Std. in mg/g)	mg/ml	g ml: m*d	CONC=CONT*m/(VEQ- (DRIFT/CONC(alt))*TIME/1,000) ²⁾	C = 1
Concentration (Std. in mg/ml)	mg/ml	g: m/d ml	CONC=CONT*m/(VEQ- (DRIFT/CONC(alt))*TIME/1,000) ²⁾	C = 1
Concentration (Std. in %)	mg/ml	g ml: m*d	CONC=CONT*m*10/(VEQ- (DRIFT/CONC(alt))*TIME/1,000) ²⁾	C = 1
Concentration (Std. in ppm)	mg/ml	g ml: m*d	CONC=CONT*m/(1,000*VEQ- (DRIFT/CONC(alt))*TIME) ²⁾	C = 1
Concentration (Std. in mg/pc) 1)	mg/ml	PC	CONC=CONT*m/(VEQ- (DRIFT/CONC(alt))*TIME/1,000) ²⁾	C = 1

¹⁾ For standard Riedel de Haën FASTrate tablets (CONT in mg/pc , m=pieces)

²⁾CONC(alt) refers to the Setup value current at the time of calculation.

Result	Unit	Replace "m" according to entry type	Formula R =	Constant
Blank	%	g	Source for drift=Request / Online / Fixed	C = 0.1
	ppm	ml: m*d	Value: R=(VEQ*CONC-TIME*DRIFT/1,000)*C/m Source for drift=Setup: R=(VEQ*CONC- TIME*DRIFTV*CONC/1,000)*C/m	C = 1,000
Result	Unit	Replace "m" according to entry type	Formula R =	Constant
Drift	µg/min		DRIFT=DRIFTV*CONC	C = 1
CW	μg		CW=VEQ*CONC*1,000	C = 1

10.2 All results

You can use the **All Results** button to view the results of the last analysis and print them if a printer is connected to the titrator (see "Printer").

From the **Results** dialog, you also have the following additional options:

- New Add an additional result
- Samples View or print the results of an individual sample or exclude the entire sample
- Statistics Switch to the "Statistics" dialog

10.3 Add result

You can use the **Add result** button to add a subsequent result calculation to your analysis results. To do that, you will first have to specify whether the calculation should be run inside or outside a loop. For calculations within a loop, the result will be added for all the samples (of the same loop) of a series. You may still be able to select the required loop. The other parameters must be entered in accordance with the "Calculate" method function. (See "Method Function Settings").

You can use the **Calculate** button to calculate the additional result and add it to your analysis results. If you are missing raw data or raw results for the calculation and cannot calculate the result for that reason, the result "NaN" (Not a number) will be added.

Predefined results (see "Evaluation and calculation>Formulas>Results proposal lists") for the volumetric Karl Fischer titration, you can view the following by selecting the **Results proposals** button in the **Add result** dialog.

See also

• Result proposal lists for Karl Fischer titration (page 64)

10.4 Statistics

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For results within a sample loop, you can display and print out statistics.

- The statistics are only created if more than one sample was analyzed in the corresponding loop.
- If you selected "Statistics functionalities" = "Yes" and "Multiple determination" = "Yes" in the associated "Calculate" method function, the system will create the statistics for the entire series and also individually for multiple samples within the series.

The following calculated values will be displayed as statistical components:

- Mean value x of a result Rx (Mean [Rx])
- Standard deviation s
- Relative standard deviation srel
- Number of samples per loop nTOT

If a result was excluded from the statistics, all the results from that sample will always be excluded as well. The system will then recalculate the statistics without the excluded sample and label them accordingly. If the sample's results are then put back into the statistical evaluation, the label will be removed from the statistics.

From the **Statistics** dialog, you also have the following additional options:

- Samples View, print, or exclude the results of an individual sample
- Results View or print all results
- Outlier test Perform a test for outliers in the statistical evaluation.

10.4.1 Outlier test

If the results of individual samples in a measurement series deviate greatly from the calculated mean value, it may make sense to question the significance of these (few) results and treat them as "outliers".

Outliers will have the following effects on the overall result of an analysis:

- The mean value is significantly shifted higher or lower.
- The standard deviation is increased.
- The distribution of the individual values around the mean value is distorted and no longer follows a normal distribution.

The titrator has an automatic function for identifying and labeling outliers. You can call this function from the **Statistics dialog** using the **"Outlier test**" softkey.

You can run an outlier test if you have the results from more than three samples.

The procedure used by the titrator is the Grubbs outlier test. For this procedure, the measured value $[x^*]$ that has the greatest deviation from the calculated mean value is analyzed. This number is used in the following equation, together with the mean value $[\bar{x}]$ and the standard deviation [s]:

$$PG = \frac{|x^* - \bar{x}|}{s}$$

The test variable [PG] is then compared with the corresponding value in the Grubbs table G (N, 90%), which in turn depends on the number of measured values N:

N (number of samples)	1	2	3	4	5	6	7	8	9	10
90 %	-	-	1.15	1.46	1.67	1.82	1.94	2.03	2.11	2.18
N (number of samples)	11	12	13	14	15	16	17	18	19	20
90 %	2.23	2.29	2.33	2.37	2.41	2.44	2.48	2.5	2.53	2.56
N (number of samples)	21	22	23	24	25	26	27	28	29	30
90 %	2.58	2.6	2.61	2.63	2.65	2.67	2.69	2.7	2.72	2.74
N (number of samples)	31	32	33	34	35	36	37	38	39	40
90 %	2.75	2.77	2.78	2.79	2.81	2.82	2.83	2.84	2.86	2.87
N (number of samples)	41	42	43	44	45	46	47	48	49	50
90 %	2.88	2.89	2.9	2.91	2.92	2.92	2.93	2.94	2.95	2.96
N (number of samples)	51	52	53	54	55	56	57	58	59	60
90 %	2.97	2.97	2.98	2.99	3	3	3.01	3.02	3.02	3.03
N (number of samples)	61	62	63	64	65	66	67	68	69	70
90 %	3.03	3.04	3.04	3.05	3.05	3.06	3.06	3.07	3.07	3.08
N (number of samples)	71	72	73	74	75	76	77	78	79	80
90 %	3.08	3.08	3.09	3.09	3.1	3.1	3.11	3.11	3.12	3.12
N (number of samples)	81	82	83	84	85	86	87	88	89	90
90 %	3.12	3.13	3.13	3.14	3.14	3.15	3.15	3.16	3.16	3.17
N (number of samples)	91	92	93	94	95	96	97	98	99	100
90 %	3.17	3.17	3.18	3.18	3.19	3.19	3.2	3.2	3.21	3.21
N (number of samples)	101	102	103	104	105	106	107	108	109	110
90 %	3.21	3.22	3.22	3.22	3.23	3.23	3.23	3.23	3.24	3.24
N (number of samples)	111	112	113	114	115	116	117	118	119	120
90 %	3.24	3.22	3.25	3.25	3.26	3.26	3.26	3.26	3.27	3.27

If the calculated test variable PG is greater than the corresponding value taken from the table, the measured value x^* is identified as an outlier and marked accordingly.

After an outlier has been identified, the test is repeated with the remaining measured values (without the already identified outlier) using the newly calculated mean value and new standard deviation. This process is repeated continually until no further outlier can be identified.

It is then the user's responsibility to exclude any identified outliers from the statistics. After confirmation, the entire sample is excluded and the statistics are recalculated without the identified and excluded outliers.

10.5 Recalculate

10.6 Samples

You can use the **Samples** button to display and print the status and calculated results for each individual sample. The same applies for a series of additional data sets that can be accessed via "**Data**". This will let you view and print the sample, method and resource data for each sample and view and print the raw results and the measured values.

You can use the **Exclude** button in the Results: **Samples** dialog to exclude individual samples from the statistical evaluation. The system will not delete the results of the samples excluded in this way but merely label them as excluded. They will no longer be included in the statistics. Samples that have been excluded can be returned to the statistics at any time by choosing "**Include**".

After a sample is excluded, all affected calculations (inside and outside of loops) are performed again. In KF titrations, the new calculations refer only to the current determination type.



If you exclude a sample from a sample group in a multiple determination, no more statistics will be generated for that group. The system will continue to create individual statistics for the remaining sample groups and for all the remaining samples overall.

10.7 Undo changes

If you make changes to the results saved by the titrator after the analysis, you can use **Undo changes** to discard them. Afterward, the system will reinstate the initial status directly after the conclusion of the analysis, in its original and unchanged state.

10.8 Delete all results

You can use this button to delete all the data (raw data, raw results, and results) saved by the titrator in the results area. The deletion only ever refers to one determination type. If the last determination type in the sample series is deleted, the whole sample series is automatically deleted.

11 Analysis data

The "Analysis data" include different types of data that can be used at various times during the planning and execution of an analysis.

Displaying raw data	Raw data is defined when you create a method or series. It is auto- matically generated and stored during the analyses. Raw data is always created for each analysis and cannot be influenced by the user.
Method data	All data for the method run.
Series data	All data from the series run, such as e.g. the series ID and the num- ber of samples.
Sample data	All data from the analyzed samples, such as e.g. the sample size, sample density and sample ID.
Resource data	The data for all resources used during the execution of an analysis (e.g., concentration standards, blank value, homogenizer name, titra- tion stand). The data for a resource are copied from the setup at the time when it is used in an analysis.
Table of measured values	Tables of measured values are created by some method functions during an analysis and can be output in the record.
Raw results	Raw results are data determined by the titrator during an analysis, e.g. the drift value or titrant consumption.
	The raw results can be converted to the actual analysis results as a part of the "Calculation" method function using suitable symbols and formulas. Some raw results are always created automatically, while others are only generated if used during a calculation.
Results	Results are the results of the conversions of raw results run within the "Calculate" method function. The results of an analysis can be influenced by the user.

The system differentiates between the following types of analysis data:

Of these, the following can be used in calculations:

- Sample data (such as the sample size or the sample density)
- Resource data (e.g. blank value variable)
- Raw results (e.g. auxiliary value, titrant consumption)
- Results (the results of a calculation can then be used in a subsequent calculation.)

12 Evaluate and calculate

12.1 Indexing of method functions

The "KF titration" method functions return their own raw results within a method.

These raw results are saved in the titrator in the order in which the generating method functions are processed within the method. To ensure that any time these method functions are used more than once, the raw results can still be given a unique assignment to their individual method functions, they are divided into different groups:

If method functions from a group are used multiple times within a method, they are given indexes (beyond the loop limits). This group index allows unique referencing of the raw results during calculations.

If the structure of a method is changed, the group indices are automatically updated, guaranteeing the serial numbering at all times.

- Make sure your calculations take this into consideration!
- Calculations can be used independently of the method functions that generated the results. For the results, we suggest using the IDs R1...Rn, following the sequence of the calculations in the method.
- Multiple loops are only permitted for the Stromboli method type.
- The group index "1" can be omitted because when a group index is missing, the system automatically assigns the group index "1".

Method function	Group index	Result
Title		
Sample (KF)		
Titration stand (Stromboli)		
Mix time		
Titration (KF vol)	1	
Calculation		RI
Calculation		R2
Calculation		R3
End of sample		
Sample (KF)		
Titration stand (Stromboli)		
Mix time		
Titration (KF vol)	2	
Calculation		R4
End of sample		
Calculation		R5

12.2 Naming conventions for using analysis data in calculations

Within calculations (see the "Calculate (page 52)" method function), you can use formula symbols to access or generate certain analysis data (raw results, results, resource and sample data). These symbols consist of basic symbols and various types of symbol extensions. The basic symbols determine the type of data and the corresponding unit. The symbol extensions can specify the data more precisely and include an abbreviation for the group of method functions which the data is to reference.

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Note that the entry of symbols in formulas is case-sensitive.

Basic symbol	Unit	Possible symbol additions	Symbol	Meaning
V	[mL]	EQ	VEQ (=V)	Titrant consumed up to the end point of the titration method function.
VPOST	[mL]		VPOST	Volume of titrant for post-consumption measurement.

TIME	[min:s]		TIME	Duration of a sample analysis from the
	_			end of Standby until the end of the method
				function Titration (KF Vol)
t	[min:s]		t	Duration of a sample analysis.
E	[mV]	EQ	EEQ	Potential at the end point of the titration method function.
EST	[mV]		EST	Measured potential at the start of the titra- tion method function.
DRIFT	[µg(H ₂ O)/min]		DRIFT	Consumption (mass) per minute for the titration method function (water quantity per time unit that penetrates the titration stand).
DRIFTV	[µL/min]		DRIFTV	Volume of titrant consumption per minute for the drift determination.
CW	[µg]		CW	Volume of water titrated up to EP (without drift or blank value correction).
CWPOST	[µg]		CWPOST	Titrated volume of water during a post-con- sumption measurement (without drift or blank value correction).
CWPOSTMean			CWPOSTMean	Determined quantity of titrated water per unit of time during the post-consumption measurement (without drift or blank value correction).
CONC	[mg/mL]		CONC	Represents the actual titrant concentration.
B	Arbitrary for KF stand, e.g. [%] and [ppm], [µg] for Stromboli		B[Name]	A blank.
Н	arbitrary		H[Name]	Auxiliary value.
m	[mL] [g] [pcs]		m	The sample size.
	[g]	sol	msol	Solvent weight for titrations of type KF Ext. Extr. (External extraction).
	[g]	EXT	mext	Extracted sample quantity for titrations of type KF Ext. Extr.
d	[g/mL]		d	The density of a sample or a standard.
wp	[g/pcs]		wp	The weight per item (can be changed under "Sample").
f			f	A correction factor (as defined in the "Sample" method function).
CONT	[mg/g] [mg/mL] [mg/pc] [%] [ppm]		CONT	The concentration of a liquid KF standard.
Rx	Arbitrary		Rx	A result x.
C			C	A constant that uniquely belongs to the result Rx. It cannot be used in this form for the calculations of other results.
Mean	Arbitrary	Rx	Mean[Rx]	The mean value of a result Rx.
S	Arbitrary	Rx	s[Rx]	The standard deviation of a result Rx
srel	[%]	Rx	srel[Rx]	The relative standard deviation of the result Rx.
n			n	The sample number.

12.3 Formulas

Calculation formulas can be used in the "Calculate" and "Condition" method functions. Some parameters within method functions can also be defined in the form of formulas.

Formulas within the "Calculation" method function

One typical example for a formula within the "Calculation" method function would be the expression **R=VEQ** in the "Formula" parameter. In this case, the consumed volume of titrant up to the point at which the end point is reached is assigned to R. All the symbols can be used for analysis data in relations like this. The analysis data to be used must be generated by the method before the "Calculate" method function.

Formulas for entering values for parameters

Formulas can also be used to specify the values for some parameters. For example, you can enter the stirring time in a "Stir" method function in the form of a formula. The result of the formula will then be copied over as a nondimensional value in the unit of the parameter in question.

Conditions

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A condition is a formula whose result comes in the form of "true" or "false". Conditions can be used in various method functions in the "Condition" parameter or subfunction. Depending on the condition's result, the method function in question will be executed (condition true) or not executed (condition false).

- Auxiliary values and blanks defined in the setup can generally be used in formulas in the same manner as symbols. The general form for an auxiliary value is: H[Name] (as defined in the setup).
- Likewise, results from other "Calculation" method functions can be referenced in the "Calculation" method function. (E.g. R3=R2+R1)

(What is important in this case is to make sure that the results used must already be in existence at the time they are to be used!)

12.3.1 Using analysis data in formulas

All analysis data that can be accessed via a symbol can be used in calculation formulas (see "Naming Conventions for Using Analysis Data in Calculations").

All analysis data must be generated in the method before the point at which they will be used in a calculation formula. For some analysis data, this could be as checked early as during the validation in the processing of a method. For others, whether or not the data are available at the time in question may not be decided until the execution of the method. If the analysis data is not available at the time of the calculation, the result of the calculation formula will be "NaN" ("Not a number").

The formula must be assigned to a result (Rx) in the "Formula" parameter in the "Calculate" method function.

Shortcuts

- Instead of VEQ, you can also use the relevant short form V in the formulas.
- If you leave out the group index of a symbol, Group Index 1 will be used.
 - Example: VEQ stands for VEQ[1]

	Explanation	Examples
Basic symbol and	Taken together, they serve as an	VEQ represents the end point of the titra-
symbol extension	identifier for the analysis data.	tion.
Group index	Specifies which method function within a method function group gen-	VEQ[3] represents the titration end point of the third loop of the method of type
	erates the analysis data.	Stromboli (model V30 only).

12.3.2 Sample formulas

Method type GT

R = QEQ*C/m (standard formula)	Content of a sample or sample solution.
R = m/(VEQ*c*C)	Titer determined with the standard substance or standard solu-
	tion.
R = QEQ*C	Content per sample.
R = QEQ	Result in the form of substance quantity consumption.
R = VEQ	Result in the form of volume consumption.

R = VEQ/m	Result in the form of volume consumption per weight or per vol- ume.
R = (QENDDi-QEQ)*C/m	Back titration
	QENDDI: Dispensed substance quantity of the "Dispense" method function.
	QEQ: Substance quantity used until the end point or equivalence point of a "Titration" method function.
R = (QEQ-B[Name])*C/m	Solvent blank (B[Name]) referenced in the result calculation.
R = (QEQ/m-B[Name])*C	Matrix blank referenced in the calculation [mmol/g].
R = EST[2]	Initial potential of the second "Titration" method function.
R = pw(-E)*1000	The ion concentration in [mmol/L], measured as pX or pM with an ion-selective electrode.
Karl Fischer Titration Vol Method Type	
Consumption	R = VEQ
Mean consumption	R=VEQ*1000/TIME
Titration duration	R=TIME
Total water content	R=CW
Content	R=(VEQ*CONC-TIME*DRIFT/1000)*C
R=(VEQ*CONC-TIME*DRIFT/1000)*C/m	R=(VEQ*CONC-TIME*DRIFT/1000)*C/m
Ext. Extraction Method Type	
External dissolution (B in the correspond- ing unit)	R=C*[(msol+mext)/mext]-B*msol/mext
Stromboli KFVol Method Type	

Consumption	R = VEQ
Mean consumption	R=VEQ*1000/TIME
Titration duration	R=TIME
Total water content	R=CW
Stromboli blank value	R=(VEQ*CONC-TIME*DRIFT/1000)*C
Content blank value compensated (B in the corresponding unit)	R=(VEQ*CONC-B[Blank Stromboli]/1000-TIME*DRIFT/1000)*C/m
	R=(VEQ*CONC-B[Blank Stromboli]/1000-TIME*DRIFT/1000)*C

12.3.3 Mathematical functions and operators

The following mathematical functions and operators can be used in formulas:

Functions		Comparison operators	
Logarithm to the base 10	lg(x)	equal to	=
Logarithm to the base e	Ln(x)	larger than	>
Exponential to base 10	pw(x) or scientific notation	larger than or equal to	> =
Exponential to base e	ex(x)	smaller than	<
Square	sq(x)	smaller than or equal to	<=
Square root	sr(x)	x in the range of	< X <
		not equal to	<>
Mathematical operators		Logical operators	
Addition	+	and	AND
Subtraction	-	or	OR
Multiplication	*		
Division	/		



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