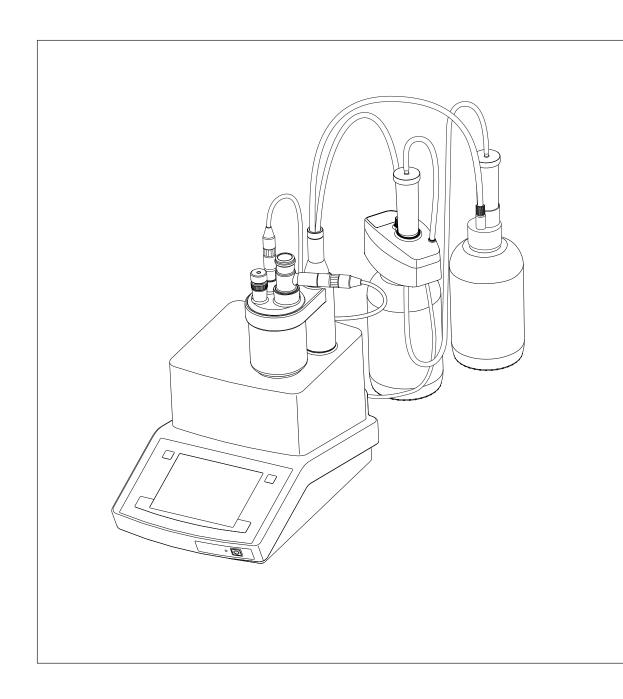
# **Karl Fischer Coulometer**

C20/C30





# **Table of Contents**

| 1 | Introduction         |                  |   | 7        |
|---|----------------------|------------------|---|----------|
| 2 | Safety Notes         |                  |   | 8        |
|   |                      | 2.1              | Definition of Signal Warnings and Symbols                                 | 8        |
|   |                      | 2.2              | Product Specific Safety Notes   | 8        |
| 3 | Karl Fischer Wa      | ıter Determina   | tion - Measuring Principle  | 10       |
| 4 | Fundamentals o       | of Coulometric   | Water Content and Bromine Index Determination                             | 11       |
|   |                      | 4.1              | Fundamentals of coulometric water content determination                   | 11       |
|   |                      | 4.2              | Coulometric bromine index determination                                   | 11       |
|   |                      | 4.3              | Generator electrodes  | 11       |
| 5 | Description of F     |                  | 0.1.0.41.0.1.0.1.0.4.0.0  | 13       |
|   | 2000 <b>p</b> o 0. 1 | 5.1              | Layout of the Terminal  | 13       |
|   |                      | 5.2              | Operating the Touchscreen   | 13       |
|   |                      | 5.3              | The Homescreen  | 13       |
|   |                      | 5.4              |   |          |
|   |                      | 5.4<br>5.4.1     | The User Interface Entering Data in the User Interface                    | 13<br>14 |
|   |                      | 5.4.2            | Shortcuts and Direct Shortcuts  | 14       |
|   |                      | 5.4.3            | The Start analysis dialog   | 15       |
|   |                      | 5.4.4            | Online Dialog   | 15       |
|   |                      | 5.4.4.1          | Pretitration  | 15       |
|   |                      | 5.4.4.2          | Standby   | 16       |
| 6 | Setup                |                  |   | 18       |
|   |                      | 6.1              | Hardware  | 18       |
|   |                      | 6.1.1            | Sensors   | 18       |
|   |                      | 6.1.2            | Pump  | 19       |
|   |                      | 6.1.3<br>6.1.3.1 | Peripherals<br>Balance  | 19<br>19 |
|   |                      | 6.1.3.1          | Barcode reader  | 20       |
|   |                      | 6.1.3.3          | USB-Stick   | 20       |
|   |                      | 6.1.3.4          | Printer and USB data export   | 20       |
|   |                      | 6.1.3.5          | PC settings   | 22       |
|   |                      | 6.1.3.6          | Network settings  | 22       |
|   |                      | 6.1.3.7          | Fingerprint reader  | 22       |
|   |                      | 6.1.3.8          | LevelSens   | 23       |
|   |                      | 6.1.4            | Titration stands  | 24       |
|   |                      | 6.2              | Global settings   | 24       |
|   |                      | 6.2.1            | System  | 24       |
|   |                      | 6.2.2            | User Management   | 25       |
|   |                      | 6.2.3            | Analysis and resources behavior   | 26       |
|   |                      | 6.2.3.1<br>6.2.4 | Monitoring the Expiration Date and Life Span of Resources Reagent control | 28<br>29 |
|   |                      | 6.3              | _   | 30       |
|   |                      | 6.3.1            | User settings<br>Language   | 30       |
|   |                      | 6.3.2            | Screen  | 30       |
|   |                      | 6.3.3            | Beep  | 31       |
|   |                      | 6.3.4            | Shortcuts   | 31       |
|   |                      | 6.3.5            | Keyboards   | 31       |
|   |                      | 6.4              | Maintenance & Service   | 31       |
|   |                      | 6.4.1            | MT service  | 31       |
|   |                      | 6.4.2            | Import/Export   | 31       |

|   |                   | 6.4.3<br>6.4.4<br>6.4.5<br>6.4.6                 | Reset to factory settings<br>Titrator firmware history<br>Board firmware<br>Terminal   | 32<br>32<br>32<br>32       |
|---|-------------------|--|--|----------------------------|
|   |                   | 6.4.7<br>6.4.8<br>6.4.9<br>6.4.10                | Board data Upgrade Update Delete Mettler method template   | 32<br>33<br>33<br>33       |
|   |                   | 6.5<br>6.5.1<br>6.5.2                            | Values<br>Blanks<br>Auxiliary values   | 33<br>33<br>33             |
| 7 | Manual operations | S  |  | 35                         |
|   |                   | 7.1  | Stirrer  | 35                         |
|   |                   | 7.2  | Pump   | 35                         |
|   |                   | 7.3<br>7.3.1                                     | Sensor<br>Polarized sensor   | 36<br>36                   |
| 8 | Methods           |  |  | 38                         |
|   |                   | 8.1  | METTLER TOLEDO Methods   | 38                         |
|   |                   | 8.2<br>8.2.1                                     | Creating Methods<br>Method Templates   | 39<br>40                   |
|   |                   | 8.3  | Modifying or Deleting Methods  | 40                         |
|   |                   | 8.4  | Starting Methods   | 41                         |
|   |                   | 8.5  | Terminate method   | 41                         |
|   |                   | 8.6<br>8.6.1<br>8.6.2<br>8.6.3<br>8.6.4<br>8.6.5 | Method Syntax — Rules for Establishing a Method<br>Possible Number of Method Functions<br>Types and Possible Number of Loops<br>Sample loops<br>Method Functions Within a Loop<br>Method Functions Outside of a Loop | 41<br>42<br>42<br>43<br>43 |
|   |                   | 8.7  | Overview of method functions   | 44                         |
|   |                   | 8.8<br>8.8.1                                     | Method functions Title   | 45<br>45                   |
|   |                   | 8.8.2<br>8.8.3<br>8.8.4<br>8.8.5                 | Drift determination Sample (KF) Titration stand Mix time   | 46<br>46<br>48<br>49       |
|   |                   | 8.8.6<br>8.8.7<br>8.8.8<br>8.8.9                 | Titration (KF Coul) Titration (EP Coul) Auxiliary value Blank  | 49<br>50<br>51<br>51       |
|   |                   | 8.8.10<br>8.8.11                                 | Instruction<br>Calculation   | 52<br>52                   |
|   |                   | 8.8.12<br>8.8.13<br>8.8.14                       | Report<br>End of sample<br>Standby   | 53<br>54<br>54             |
|   |                   | 8.8.15   | Hidden method functions  | 54<br>54                   |
| 9 | Series Templates  |  |  | 55                         |
|   |                   | 9.1  | Sample series  | 55                         |
|   |                   | 9.2  | Sample parameters  | 55                         |

| 10 | Results          |        |  | 57 |
|----|------------------|--------|--|----|
|    |                  | 10.1   | Results proposal lists                                     | 57 |
|    |                  | 10.1.1 | Internal calculations                                      | 59 |
|    |                  | 10.2   | All results  | 59 |
|    |                  | 10.3   | Add result   | 60 |
|    |                  | 10.4   | Statistics   | 60 |
|    |                  | 10.4.1 | Outlier test   | 60 |
|    |                  | 10.5   | Samples  | 61 |
|    |                  | 10.6   | Undo changes   | 61 |
|    |                  | 10.7   | Delete all results   | 62 |
| 11 | Analysis Sequen  | ces    |  | 63 |
|    |                  | 11.1   | Starting an Analysis                                       | 63 |
|    |                  | 11.2   | KF Analysis sequence                                       | 64 |
|    |                  | 11.2.1 | Series analyses with the "Stromboli" oven sample changer   | 66 |
|    |                  | 11.2.2 | External extraction  | 67 |
|    |                  | 11.2.3 | Switching between determination types                      | 67 |
|    |                  | 11.2.4 | Replacing the reagent solution                             | 67 |
| 12 | Analysis data    |        |  | 68 |
| 13 | Evaluate and cal | culate |  | 69 |
|    |                  | 13.1   | Indexing of method functions                               | 69 |
|    |                  | 13.2   | Naming conventions for using analysis data in calculations | 69 |
|    |                  | 13.3   | Formulas   | 71 |
|    |                  | 13.3.1 | Using analysis data in formulas                            | 71 |
|    |                  | 13.3.2 | Mathematical functions and operators                       | 72 |
|    | Index            |        |  | 73 |

# 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

**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



General hazard



Electrical shock



Toxic substance



Inflammable or explosive substance



Acid / Corrosion

# 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  $5\,^{\circ}\text{C}$  and above  $40\,^{\circ}\text{C}$ .

### **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**

#### Risk of electric shock

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.

- 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:

$$2 H_2O + SO_2 + I_2 \rightarrow SO_4^{2-} + 2 I^- + 4 H^+$$

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:

$$(RNH)\cdot(CH_3OSO_2) + I_2 + H_2O + 2 RN \rightarrow (RNH)\cdot(CH_3OSO_3) + 2 (RNH)\cdot I$$

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).

# 4 Fundamentals of Coulometric Water Content and Bromine Index Determination

With the METTLER TOLEDO Coulometers C20 and C30 you can perform coulometric Karl Fischer titration as well as determine the bromine index (C30 only) simply, quickly and reliably. The fundamentals of coulometric water content and bromine index determination are summarized below.

#### 4.1 Fundamentals of coulometric water content determination

In coulometric Karl-Fischer titration, iodine is generated in an electrochemical reaction by the anodic oxidation of iodide at the generator electrode:

$$2 \vdash \rightarrow l_2 + 2 e^-$$

If water is present in the analyte, the generated iodine reacts directly with water.  $I_2$  and  $H_2O$  react in the ratio 1:1. According to Faraday's law, the quantity of iodine generated is proportional to the electrical load (10.712 mC = 1  $\mu$ g  $H_2O$ ). The coulometric consumption up to the end point is therefore a measure of the quantity of water present.

Once all the water has been consumed by the reaction, the measurement solution contains a small excess of iodine. This iodine excess is detected by the polarized measurement electrode and the electrical current for iodine generation is stopped. Generator electrodes with and without a diaphragm are available. The generation and detection of iodine is the same in both cases.

Karl Fischer titration runs at a maximum rate in the pH range 5.5 to 8. In practice, therefore, the solution should not exceed pH 8 or fall below pH 4. For acidic and basic samples, the pH value must be altered to remain within the ideal range by the addition of buffer substances (imidazole for acids, salicylic acid for bases).

The titration cell consists of the anode compartment and the cathode compartment, which may be separated by a diaphragm. The anode compartment contains the anolyte, which contains sulfur dioxide, imidazole and iodide. Methanol or ethanol are used as the solvent. The cathode compartment contains the catholyte. Depending on the manufacturer, this may either be a specific reagent, or the same solution as in the anode compartment.

The coulometric Karl Fischer procedure is suitable for samples with a low water content (1 ppm to 5 %).

#### 4.2 Coulometric bromine index determination

In coulometric bromine index determination, electrochemically generated bromine reacts with the double bonds in organic compounds according to the following equation:

$$Br_2 + R1-CH=CH-R2 \rightarrow R1-CHBr-CHBr-R2$$
 (1

The bromine index [mg bromine / 100 g sample] specifies how much bromine is used, according to the equation (1), to react a sample.

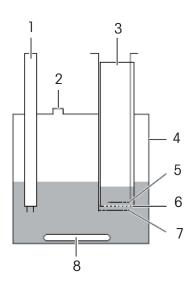
The bromine is generated at the anode of the generator electrode:

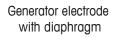
$$2 Br^{-} \rightarrow Br_2 + 2 e^{-} \tag{2}$$

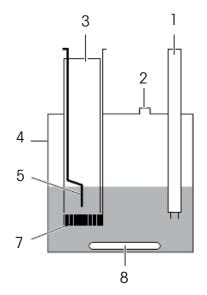
#### 4.3 Generator electrodes

METTLER TOLEDO offers generator electrodes with and without diaphragms for both iodine and bromine generation (see schematic diagram below).

For bromine index determination we recommend the generator electrode without a diaphragm, as it is easier to clean.







Generator electrode without diaphragm

- 1: Measuring electrode
- 2: Septum
  3: Generator electrode
  4: Cell
- 5: Cathode
- 6: Diaphragm 7: Anode
- 8: Magnetic stirring rod

# 5 Description of Functions

# 5.1 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.

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.

# 5.2 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!

#### 5.3 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

Shortcuts and Direct Shortcuts (page 14)

#### 5.4 The User Interface

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

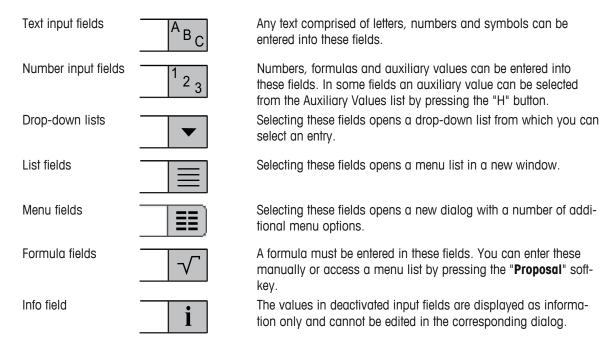
- The title bar at the top of the display specifies the name of the current dialog.
- In the top right-hand corner, the Tasks button informs you which processes are currently running. You use
  Tasks to access a Tasks dialog that displays an overview of all running tasks. From the Tasks dialog you
 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

# 5.4.1 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: Tasks (C30 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.

# **5.4.2** 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").



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.

# 5.4.3 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 short-cut.

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.

#### 5.4.4 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.

#### 5.4.4.1 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

Select the **Results** button to display the results and statistics for the samples to be analyzed following the analysis. The system displays the results of the active determination type (sample, blank value). Furthermore,

the "Results" dialog also contains the following buttons: **Add Result, Recalculate, Undo Changes, Perform Outlier Test**.

#### **Samples**

You can change sample and series data. However, the number of samples cannot be changed while a blank determination is in progress. For changes to the sample data, refer to the analysis sequences: Starting an analysis

#### 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.



 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. Blank value data is 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 | Parameters Description                |                   |
|------------|---------------------------------------|-------------------|
| Content    | Expected water content of the sample. | 0 10 <sup>6</sup> |
| Unit       | Unit for the content.                 | [%] I [ppm]       |

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

### 5.4.4.2 Standby

If the drift drops below a predefined value, the system automatically switches from **pretitration** to **Standby** mode (see "Analysis flows: Analysis flow diagram").

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

#### Start drift

For a drift determination, at least one increment of titrant must be generated. 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.

#### 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**.

#### 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.

#### 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.

#### **Cancel determination**

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

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

# Setup

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

| Hardware           | Sensors                         |
|--------------------|---------------------------------|
|                    | Pumps                           |
|                    | Peripherals                     |
|                    | Titration Stands                |
| User settings      | Language                        |
|                    | Screen                          |
|                    | Beep                            |
|                    | Shortcuts                       |
|                    | Keyboard                        |
| Global settings    | System                          |
|                    | User management                 |
|                    | Analysis and resources behavior |
|                    | Reagent Control                 |
| Values             | Blank                           |
|                    | Auxiliary values                |
| Mainten. & Service | MT-Service                      |
|                    | Import / Export                 |
|                    | Reset to factory settings       |
|                    | Titrator firmware history       |
|                    | Board firmware                  |
|                    | Terminal                        |
|                    | Board data                      |
|                    | 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.

# 6.1 Hardware

Navigation: **Setup** > **Hardware** 

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

- Sensors
- Pump
- Peripherals (devices such as printers or balances)
- Titration stands (KF stand and additional Stromboli TTL for C30)

#### 6.1.1 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 [µA].

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).

# 6.1.2 Pump

Navigation: **Setup** > **Hardware** > **Pumps** 

The Reagent manager is predefined in the Setupand cannot be configured individually. A connected Reagent manager creates the relevant entry.

The parameters **Type**, **Name** and **Pump output** are displayed in the information fields in the **Pump parameters** window.

# 6.1.3 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.

#### 6.1.3.1 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 connections are located on the mainboard, the analog board and the conductivity board. | MB/COM1  <br>MB/COM2   AB1/COM  <br>More depending on<br>configuration |
| Baud rate     | The baud rate for data transmission via the RS-232 interface.   | 1200   2400   4800  <br>9600   19200                                   |

| Data bit  | Defines the number of data bits.   | 7   8             |
|-----------|--|-------------------|
| 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.)   | 112               |
| 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   |



- 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.

#### 6.1.3.2 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 I No  |
| Codes to LabX   |  |           |

#### 6.1.3.3 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.

#### 6.1.3.4 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.



- 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 Instruction 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<br>compact printer   USB<br>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

 $\label{eq:navigation: home > Setup > Global settings > Analysis and resources behavior > Analysis sequence settings$ 

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.           | MB1/USB1   PnP |
|               | <b>PnP</b> is displayed if the printer is not connected to the titrator. |                |

#### 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). <b>PnP</b> 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  <br>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.

### 6.1.3.5 PC settings

i

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 established on startup.                                   | Yes I No                 |
| Connection type             | Defines how the titrator is connected to the PC, either via the network connection or via the USB connection.          | Network   USB            |
| Status                      | Information on the connection status from the instrument to LabX.  | Connected   Disconnected |
| Port number                 | Defines the port for a network connection of the titrator to LabX. Only appears for <b>Connection type = Network</b> . | 102465535                |

# 6.1.3.6 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<br>address auto-<br>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<br>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 subnet's IP address. | 000.000.000.000<br>255.255.255.255 |
| Standard gate-<br>way                   | This is where you can enter the address of the standard gateway for communication between the various networks.                                  | 000.000.000.000<br>255.255.255.255 |

## 6.1.3.7 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-<br>print reader | Activates the fingerprint reader for authenticating users when log-<br>ging onto the titrator.  | Yes I No                  |
| Status                           | Indicates whether the fingerprint reader is connected to the titrator.  | Installed   Not installed |
| Name                             | The designation of the fingerprint reader.  | Arbitrary                 |
| Connection                       | Information on the USB port to which the fingerprint reader is connected. <b>PnP</b> 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.
- i
- 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].

#### 6.1.3.8 LevelSens

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".
  - ⇒ 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<br>typeSensor 4<br>type | Specifies the sensor type to be used.  | Level   Waste   Inactive |

#### 6.1.4 Titration stands

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<br>TTL  |
| Name           | Specify a descriptive name of your choice.  | Arbitrary  |
| Stirrer output | Defines the stirrer output.                 | MB/Stirrer1   MB/Stir-<br>rer2   AB1/Stirrer  <br>Internal stirrer   More<br>depending on configu-<br>ration |

1

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

# 6.2 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.
- Reagent control instructs the user to replace the reagent solution. For information on the process for replacing the solution.

# **6.2.1** 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-<br>sion | Information on the firmware version of the instrument. | -               |

#### 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 I No  |
| Text          | Defines the text for the header. Only for <b>Header = Yes</b> .        | Arbitrary   |
| Footer        | Activates the footer on print outs.                                    | Yes I No  |
| Text          | Defines the text for the footer. Only for <b>Footer</b> = <b>Yes</b> . | Arbitrary   |
| End of report | Select the information to be printed at the end of a report.           | Created by   Modified<br>by   Checked by  <br>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.                                   |          |

# 6.2.2 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 <b>Enforce password/fingerprint = Yes</b> is selected in <b>Account Policies</b> . | Yes I No               |
| Lock user      | If activated, the user account is locked.  Only appears if <b>Enforce password/fingerprint = Yes</b> is selected in <b>Account Policies</b> .   | Yes I No               |

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



- 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].
  - ⇒ The two fixed defined groups are listed in this window.
- 2 Select one of these groups to access the **Group parameters** dialog.
- ⇒ 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.

#### **Account Policies**

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 I 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).

| Mi  | n. no. of char- | Specifies the minimum number of characters required for user    | Yes   No |
|-----|-----------------|---|----------|
| act | ters            | 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.      |          |

## 6.2.3 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.

<sup>\*</sup>Basic functions: Starting drift or blank determination.

## **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 appropriate error message is issued. | Yes I No |
| Show SOP                         | If an SOP (standard operating procedure) has been defined in the <b>Title</b> 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 when not con-<br>nected           | If "yes" is selected then a warning is issued when the analysis is started if there is no connection to LabX.   | Yes I No          |
|---|---|-------------------|
| 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 <b>Auto stand</b> 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<br>printer connec-<br>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 <b>Record</b> is skipped. If a printer is connected, the method function <b>Record</b> 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 I 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 <b>Check USB-Stick connection and wait = Yes</b> . | To USB-Stick   No |
| Check USB-Stick connection and wait             | If this parameter is activated, the presence of a USB - Stick is validated 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.       |                        |

#### 6.2.3.1 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.

You can define the following parameters:

| 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: <b>Time period</b> ).   | <b>Days</b> : 11000<br><b>Hours</b> : 110 <sup>4</sup> |
| 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 <b>Usable life</b> .  Only if <b>Time period = Days</b> and <b>Reminder = Yes</b> . | 01000  |

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 28)").

#### 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 28)").

# 6.2.4 Reagent control

Navigation: Setup>Global>Reagent control

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

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

- The time interval for use of the reagent solution.
- 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 reagent solution.

To monitor the reagent solution, the time, water volume, and number of samples is recorded and added 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 reagent solution. The Solvent manager is started to pump the reagent solution away. The cell is then filled with new solution. All counters are reset to zero (see Manual operations: Pump).

For sample analysis using the Stromboli oven sample changer, the reagent solution can only be replaced in Standby mode before analysis of the first sample, or at the end of the series, if the titrator returns to Standby mode.

You can choose the Reagent control button in the Global settings to open the Reagent control dialog.

In this window, you can define the following parameters for solvent replacement:

| Parameters  | Description  | Values   |
|---|--|----------|
| Monitoring usable life of reagent                         | Specifies whether the reagent solution is to be monitored.                             | Yes   No |
| Usable life   | Defines the time interval in days for the use of the reagent solution.                 | 110000   |
| Enforce replace-<br>ment when<br>exceeding<br>usable life | Forces the user to replace the reagent solution immediately after the prompt to do so. | Yes I No |
| Monitoring capacity of reagent                            | Specifies whether the capacity is to be monitored.                                     | Yes I No |

| Stir   | Enables the stirrer during solvent exchange.   | Yes I No |
|--|--|----------|
| Enforce replace-<br>ment at max.<br>no. of samples     | Forces the user to replace the reagent solution immediately after the prompt to do so.                   | Yes I No |
| Max. no. of samples                                    | Maximum number of samples (not including the blank value) after which the reagent is to be replaced.     | 0120     |
| Monitoring no. of samples                              | Specifies whether the system should monitor the number of samples.                                       | Yes I No |
| Enforce replace-<br>ment when<br>exceeding<br>capacity | Forces the user to replace the reagent solution immediately after the prompt to do so.                   | Yes I No |
| Max. amount of water                                   | The maximum volume of water in [mg] for a reagent. Only if <b>Monitoring capacity of reagent = Yes</b> . | 01000000 |

#### See also

Monitoring the expiration date of a resource (page 28)

# 6.3 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.

# 6.3.1 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   Italian   Span-  |
|             |  | ish I Chinese I Russian I |
|             |  | Polish I Korean           |
| Record      | Defines the language in which the protocols are to be printed out. | German   English          |
|             |  | French   Italian   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.

## 6.3.2 Screen

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  <br>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 I 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                               |

## 6.3.3 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   No |
| ton               |  |          |

# 6.3.4 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 immediately. This enables you to start the analysis without any interfering dialog. | Yes I No  |
| Homescreen position | You can select the free position for the shortcut on the Homescreen.   | 112       |

# 6.3.5 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      |

#### 6.4 Maintenance & Service

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

# 6.4.2 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   Import |
| Method ID  | Here you can select the Method ID for the relevant method.   | Method list     |

### 6.4.3 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.

# 6.4.4 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.

# 6.4.5 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.

#### 6.4.6 Terminal

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

#### 6.4.7 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.

# 6.4.8 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:

i

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.

#### **6.4.9** Update

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

# 6.4.10 Delete Mettler method template

You can easily delete Mettler method template from the titrator.

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

## 6.5 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.              |

#### 6.5.1 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].
- ⇒ 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.  | -108108   |
| 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 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.

# 6.5.2 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].
- ⇒ 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.  | -108108   |
| 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.

# 7 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 components | Possible manual operations | Possible usages                  |
|---------------------|----------------------------|----------------------------------|
| Stirrer             | Stir                       | Dissolve a solid sample          |
| Pump                | Pump                       | Fill, empty, or replace liquids. |
| Sensor              | Measure                    | Voltametric indication           |



- 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.

#### 7.1 Stirrer

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.
- ⇒ 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-<br>rer2   AB1/Stirrer  <br>Internal stirrer   More<br>depending on configu-<br>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. | 010⁴ I ∞   |



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

# **7.2** 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 reagent                     |
| 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 samples). The fill date for the cell is also reset.                                 | Yes I No   |
| Stirrer         | A stirrer can be switched on. Only for <b>Action = Fill</b> or <b>Drain</b> .   | Yes I 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 configuration |
| Speed           | Speed in [%]. Only if stirrer is activated.   | 0100   |

## 7.3 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:

### 7.3.1 Polarized sensor

| Parameters      | Description  | Values   |
|-----------------|--|--|
| Sensor          | Select a sensor from the list. The list depends on the sensor type selected in <b>Type</b> . | List of available sensors  |
| ipoi            | Ipol is the polarization current, in [ $\mu A$ ], 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-<br>rer2   AB1/Stirrer  <br>Internal stirrer   More<br>depending on configu-<br>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 "\infty" for unlimited measurement time. | 010⁴   ∞   |

| Record | If activated, the measured values will be printed out.            | Yes I No |
|--------|---|----------|
| dt     | Defines the time interval in [sec] for outputting measured values | 16000    |
|        | to the printer.   |          |
|        | Only appears if <b>Record</b> = <b>Yes</b> was selected.          |          |

i

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.

## 8 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 coul

Method for coulometric water content determination with the Karl Fischer method (according ASTM D1492).

#### Bromine Index

Method for coulometric Bromine Index (BI) determination.

#### 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 coulometric Karl Fischer titration, consisting of "KFC" followed by a sequential number (KFC01, KFC02...).
- You can enter a name of your choice for the method ID for a user method, however it cannot begin with "KFC" followed by a number.

## 8.1 METTLER TOLEDO Methods

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

| ID   | Туре                 | Title                         | Description   |
|------|----------------------|-------------------------------|---|
| M314 | KF coul              | Water standard 1.0 mg/g       | Check with a standard 1 ppm water in toluene  |
| M315 | KF coul<br>Stromboli | Oven stand. 1%<br>(Stromboli) | Automatic gas phase extraction with an EMD standard oven with a water content of 1.0%.  Note: This method includes one sample loop for the blank determination and one for the water content determination. |

| ID   | Туре                        | Title                        | Description  |
|------|-----------------------------|------------------------------|--|
| M391 | KF coul                     | Toluene dry                  | Water content determination of a sample with a water content in the ppm range  |
| M392 | KF coul                     | Nitrogen gas                 | Water content determination of a gaseous sample with a water content in the ppm range  |
| M393 | KF coul                     | Acetone dry                  | Water content determination of a ketone-containing sample with a water content in the ppm range  |
| M394 | KF coul external extraction | Sugar (external extraction)  | External extraction of a sample with a water content in the ppm range  |
| M395 | KF coul                     | PET granulates (manual oven) | Manual gas phase extraction of a sample with a water content in the ppm range, using the DO308 oven  |
| M396 | KF coul Stromboli           | Temp.ramp (Stromboli)        | Automatic gas phase extraction with one blank loop and 13 sample loops with different temperatures: From 120 °C to 300 °C in 15° increments. Proposal: Polycarbonate or polyamide as sample. |
| M397 | Bromine index (1 loop)      | Bromine index                | Bromine index determination in mg/100g using a blank value   |
| M398 | Bromine index blank         | Bromine index                | Determining the blank value of an electrolyte  |
| M399 | Bromine index (2 loops)     | Bromine index                | Bromine index determination with one conditioning loop and one loop for determining the bromine index of the sample  |

The polarizable electrode DM143-SC is used as a sensor.

# 8.2 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.
- 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**].
  - ⇒ The method function disappears from the method.

- 11 After inserting all required method functions, tap [Save].
- ⇒ 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.

## 8.2.1 Method Templates

When you create a new method, the method templates prescribe the sequence of the method functions. These method templates are not application-specific. They allow the user to establish user methods quickly and easily. Most of the parameters 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                           | Description  | Method type                           | Device type |
|---------------------------------|--|---------------------------------------|-------------|
| KF coul                         | Determining the water content in ppm (coulometric standard KF method)  | Coulometric Karl<br>Fischer titration | C20 / C30   |
| External extraction/dissolution | When calculating the function, it is possible to select the calculation formula that corresponds to the extraction method. Furthermore, this standard method is used to determine the blank value of the solvent. (Coulometric standard KF method) | External extraction/dissolution       | C30         |
| Stromboli                       | Gas phase extraction with Stromboli (one blank and one sample loop) (Coulometric standard KF method)   | Stromboli                             | C30         |
| EP coul / EP coul               | 2 loop method (conditioner loop and sample determination loop) (Coulometric standard bromine index method)   | BI                                    | C30         |
| Blank with EP coul              | Determining the blank value of the solvent in [mg/100g] (Coulometric standard bromine index method)  | ВІ                                    | C30         |
| EP coul using blank             | Weight as sample entry, determining the bromine index using the blank value in mg (Coulometric standard bromine index method)  | ВІ                                    | C30         |

# 8.3 Modifying or Deleting Methods

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



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.
- ⇒ 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.

## 8.4 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**.

#### 8.5 Terminate method

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") 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(Coul) titrations (page 15)").

## 8.6 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:

#### 8.6.1 Possible Number of Method Functions

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

## Number of functions per method for KF coul, external extraction, and Stromboli

| Method function     |     | per per method for<br>hod types except | Max. number for Stromboli methods (for C30 only) |  |
|---------------------|-----|--|--|--|
|                     | C20 | C30                                    |  |  |
| Title               | 1   | 1                                      | 1  |  |
| Sample (KF)         | 1   | 1                                      | 14   |  |
| Titration stand     | 1   | 1                                      | 14   |  |
| Titration (KF coul) | 1   | 1                                      | 14   |  |
| Calculation         | 3   | 40                                     | 40   |  |
| End of sample       | 1   | 1                                      | 14   |  |
| Auxiliary value     | -   | 30                                     | 30   |  |
| Blank               | -   | -                                      | 10   |  |
| Instruction         | 1   | 10                                     | 10   |  |
| Report              | 1   | 10                                     | 14   |  |
| Drift determination | -   | -                                      | 14   |  |
| Mix time            | 1   | 1                                      | 15   |  |
| Homogenizer         | -   | -                                      | -  |  |
| Standby (Stromboli) | -   | -                                      | 1  |  |

#### Number of functions for Bromine Index (BI)

| Number of functions | Max. number for BI |
|---------------------|--------------------|
| Title               | 1                  |
| Sample              | 3                  |
| Titration stand     | 3                  |
| Titration (EP coul) | 3                  |
| Calculation         | 40                 |
| End of sample       | 3                  |
| Auxiliary value     | 30                 |
| Blank               | 10                 |
| Instruction         | 10                 |
| Report              | 10                 |
| Mix time            | 3                  |

## 8.6.2 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 coul             | 1                                  |  |
| External extraction | 1 (for C30 only)                   |  |
| Stromboli           | 14 (for C30 only)                  |  |
| Bromine index       | 3 (for C30 only)                   |  |

## 8.6.3 Sample loops

#### Stromboli

Two templates exist for the "Stromboli (KF coul)" 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 C30 only):

| Sample loop (KF): | Sample (KF)                 |  |
|-------------------|-----------------------------|--|
|                   | Titration stand (Stromboli) |  |
|                   | Mix time                    |  |
|                   | Titration (KF Coul)         |  |
|                   | Calculation                 |  |
|                   | Report                      |  |
|                   | End of sample               |  |
|                   | Blank                       |  |

#### **Bromine Index**

Three templates exist for the "Bromine index" method type:

| Sample loop: | Sample               |
|--------------|----------------------|
|              | Titration stand (KF) |
|              | Mix time             |
|              | Titration (EP Coul)  |
|              | Calculation          |
|              | Report               |
|              | End of sample        |

## 8.6.4 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.

#### **KF Method**

- Titration Stand (KFStand | Stromboli | External KF stand)
- Mix time
- Titration (KF coul)
- Auxiliary value
- Instruction
- Record
- Calculation

The following method functions are only available for the "Stromboli" method type (for C30 only):

- Blank
- Drift determination

#### **Bromine index**

The following method functions are permitted within a bromine index method:

- Titration stand (KF stand | External KF stand)
- Mix time
- Titration (EP coul)
- Auxiliary value
- Instruction
- Record
- Calculation
- Blank



The selection option depends on the sequence of the KF method (see above) .

## 8.6.5 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:

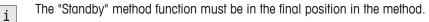
#### Method type "KF coul"

For the method type "KF coul", no selection options are available outside the sample loop.

#### Method type "Stromboli KF coul"

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

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



#### Method type "Bromine index"

For the method type "Bromine index", you can use the following method functions:

- Calculation
- Auxiliary value
- Blank
- Instruction
- Record

# 8.7 Overview of method functions

| Functions           | Explanation  | Within loop   | Outside loop |
|---------------------|--|---------------|--------------|
| Title               | Title and characteristics of the method.   | No            | Yes          |
| Sample (KF)         | Start of a sample loop.  | Start of loop |              |
| Titration stand     | Select a titration stand (KF stand or Stromboli TTL)   | 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           |
| Titration (KF Coul) | Conducts a coulometric Karl Fischer titration.   | Yes           | No           |

| Functions           | Explanation  | Within loop | Outside loop |
|---------------------|--|-------------|--------------|
| Auxiliary value     | 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 instructions 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<br>Karl Fischer titrations<br>(only for method type<br>"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          |

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

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

Functions marked with \* are optional.

## 8.8 Method functions

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

## 8.8.1 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<br>with "M" followed by a<br>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. | 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          |

## 8.8.2 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 method function "Drift determination" applies only for methods of the type "Stromboli".

| 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 determination 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   |

# 8.8.3 Sample (KF)

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

# **Subfunction: Sample**

| 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 <b>Number of IDs</b> .   | 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. <b>Fixed volume</b> or <b>Fixed pieces</b> : 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  <br>Volume   Fixed<br>volume   Pieces   Fixed<br>pieces |
| Lower limit         | Defines the lower limit for the variable entry of data. The unit will depend on the setting for the <b>Entry type</b> parameter.  Only appears if for <b>Entry type</b> no "Fixed" values are selected.  | [g]: 0 1000<br>[mL]: 0 10 <sup>4</sup><br>[pcs.]: 010 <sup>6</sup>             |
| Upper limit         | Defines the upper limit for the variable entry of data. The unit will depend on the setting for the <b>Entry type</b> parameter.  Only appears if for <b>Entry type</b> no "Fixed" values are selected.  | [g]: 0 10 <sup>3</sup><br>[mL]: 0 10 <sup>3</sup><br>[pcs.]: 0 10 <sup>6</sup> |
| Weight              | Weight in [g]. Appears only if <b>Entry type</b> = <b>Fixed weight</b> was selected.   | 01000  |
| Value               | Volume in [mL]. Only appears if for <b>Entry type</b> "Fixed" values are selected.   | 0104   |
| Pieces              | The number of sample(s). Appears only if <b>Entry type</b> = <b>Fixed pieces</b> was selected.   | 0106   |
| Weight per<br>piece | The weight in [g] per pirece.  Appears only if <b>Entry type</b> = <b>Pieces</b> or <b>Fixed pieces</b> 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 = <b>Ext. Extraction</b> .  | 01000                      |
|--------------------------|--|----------------------------|
| Wt. extracted sample     | Total weight of sample in [g] which was extracted or dissolved in the solvent.  Only for method type = <b>Ext. Extraction</b> .  | 01000                      |
| <b>Correction factor</b> | Any correction factor that can be used in calculations.  | 0.0001106                  |
| Temperature              | The temperature in [°C] during the analysis.   | -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 <b>Automatic</b> is selected, the analysis begins without any user confirmation if the value falls below the maximum start drift and the defined stability criterion <b>Drift stability</b> /dt and the set temperature are reached.  If the standby is executed by the method function <b>Standby</b> (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 <b>Analysis start = Automatic</b> is selected.   | 01000                      |
| dt                       | dt in [sec] is the time taken to determine the drift stability. The time recording can begin before the set temperature is reached and before the value falls below the maximum start drift.  Only for "Stromboli" method type and if <b>Analysis start = Automatic</b> is selected.   | 11000                      |
| 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 addition |

## Subfunction: Blank value (for method type: external extraction)

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.

You can determine the following parameters:

| Parameters       | Description  | Values                                     |
|------------------|--|--|
| Source for blank | <b>Setup</b> : After the blank value is determined, the value and the unit of the blank are transferred to the settings. | Setup   Fix value [%]  <br>Fix value [ppm] |
|                  | Fix: The value defined in the method is used.  | Request [%]   Request   [ppm]              |
|                  | <b>Request</b> : The blank value in the relevant unit is requested before each sample.                                   | [bb]                                       |
|                  | The specified blank value is labeled with "B" in the method function <b>Calculation</b> .                                |  |
| Value            | Here you can enter a numerical value. Only appears if for <b>Entry type</b> "Fixed" values are selected.                 | 0106                                       |
| 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 <b>Setup</b> 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 <b>Fixed weight</b> or <b>Fixed volume</b> , 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  <br>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 <b>Entry type</b> parameter.  Applies only for <b>Entry type</b> = <b>Weight</b> and <b>Volume</b> .   | 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 <b>Entry type</b> parameter.  Applies only for <b>Entry type</b> = <b>Weight</b> and <b>Volume</b> .   | 01000  |
| Weight      | Weight in [g]. Appears only if <b>Entry type = Fixed weight</b> was selected.  | 01000  |
| Volume      | Volume in [mL]. Appears only if <b>Entry type = Fixed volume</b> was selected.   | 01000  |
| Density     | The density of the liquid sample in [g/mL] for <b>Entry type = Volume</b> or <b>Fixed volume</b> .   | 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   |
| 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 addition                       |
| 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   |
| 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 <sup>6</sup>                                 |

## See also

• Blanks (page 33)

# 8.8.4 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 drift      | 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-<br>ture | Temperature setting in [°C] for the "Stromboli" oven sample changer. Only for <b>Titration stand = Stromboli TTL</b> .   | 50300  |

## 8.8.5 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   |

## 8.8.6 Titration (KF Coul)

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

You can determine the relevant parameters for the following subfunctions:

#### **Subfunction: Sensor**

For coulometric KF titrations, only polarized sensors are used.

| Parameters | Description  | Values                    |
|------------|--|---------------------------|
| Sensor     | Select a sensor from the list. The list depends on the sensor type selected in <b>Type</b> . | List of available sensors |
| Ipol       | lpol is the polarization current, in [ $\mu A$ ], for the voltametric indication.            | 0.024.0                   |

#### **Subfunction: Stir**

| Parameters | Description                        | Values |
|------------|------------------------------------|--------|
| Speed      | Defines the stirring speed in [%]. | 0100   |

## **Subfunction: Control**

The titrant generation is controlled by the generator current. Defined current increments can be defined user-specifically or automatically. You can also set the titration end point (recommended value: 100 mV). You can determine the rate of titrant generation - whether it is generated normally or slowly (cautiously). The "Cautious" mode is used to avoid overtitration for smaller sample volumes.

| Parameters   | Description   | Values            |
|--------------|---|-------------------|
| End point    | End point in [mV] of the Karl Fischer titration and the standby titration.  | -2000 2000        |
| Rate         | Cautious or normal current regulation.  | Cautious   Normal |
| Control band | The value in [mV] defines the width of the control band. Outside the control band, the system will titrate with the maximum dispensing 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 addition of titrant to approach the end point cautiously. | 0.12000           |

| Generator cur- | You can select whether the pulse strength is regulated automati-  | Automatic   Fix       |
|----------------|---|-----------------------|
| rent           | cally or whether the user enters a required fixed pulse strength. |                       |
| Current        | The current can be selected with fixed generator currents.        | 400   300   200   100 |

#### **Subfunction: Termination**

| Parameters | Description   | Values   |
|------------|---|--|
| Туре       | Termination of titration following defined drift and if the value falls below the end-point value (EP). <b>Drift stop relative</b> : Actual drift stop value = online drift + drift <b>Drift stop absolute</b> : Actual drift stop value = drift <b>Delay time</b> : Termination after a delay time below the EP. | Drift stop relative  <br>Drift stop absolute  <br>Delay time |
| Drift      | The drift value in [µg/min] for the termination criterion drift stop relative or drift stop absolute.   | 1.0 106  |
| 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 <sup>8</sup>   Auxiliary value                           |
| Max. time  | Maximum duration of the titration (without post-consumption measurement).   | 010 <sup>8</sup> I ∞ I Auxiliary value                       |

## 8.8.7 Titration (EP Coul)

The Karl Fischer titration is performed using the "Titration (EP Coul)" method function. Especially, this function is used to determine the Bromine Index (BI).

"Titration (EP Coul)" contains subfunctions which each have their own parameters.

You can determine the relevant parameters for the following subfunctions:

#### **Subfunction: Sensor**

For coulometric titrations, only polarized sensors are used.

| Parameters | Description  | Values                    |
|------------|--|---------------------------|
| Sensor     | Select a sensor from the list. The list depends on the sensor type selected in <b>Type</b> . | List of available sensors |
| Ipol       | lpol is the polarization current, in [ $\mu A$ ], for the voltametric indication.            | 0.024.0                   |

#### **Subfunction: Stir**

| Parameters | Description                        | Values |
|------------|------------------------------------|--------|
| Speed      | Defines the stirring speed in [%]. | 0100   |

## **Subfunction: Control**

The titrant generation is controlled by the generator current. Defined current increments can be defined user-specifically or automatically. You can also set the titration end point (recommended value:100 mV). You can determine the rate of titrant generation - whether it is generated normally or slowly (cautiously). The "Cautious" mode is used to avoid overtitration for smaller sample volumes.

| Parameters             | Description   | Values                |
|------------------------|---|-----------------------|
| End point              | End point in [mV] of the Karl Fischer titration and the standby titration.  | -2000 2000            |
| Rate                   | Cautious or normal current regulation.  | Cautious   Normal     |
| Control band           | The value in [mV] defines the width of the control band. Outside the control band, the system will titrate with the maximum dispensing 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 addition of titrant to approach the end point cautiously. | 0.12000               |
| Generator cur-<br>rent | You can select whether the pulse strength is regulated automatically or whether the user enters a required fixed pulse strength.  | Automatic   Fix       |
| Current                | The current can be selected with fixed generator currents.  | 400   300   200   100 |

## **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  <br>Drift stop absolute  <br>Delay time |
| Drift      | The drift value in [µg/min] for the termination criterion drift stop relative or drift stop absolute.  | 1.0 106  |
| 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 <sup>8</sup>   Auxiliary value                           |
| Max. time  | Maximum duration of the titration (without post-consumption measurement).  | 0108   ∞   Auxiliary value                                   |

# 8.8.8 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-<br>tion and calculation<br>(page 69)")   Auxiliary<br>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 outside limits | Determines whether the method should be interrupted if a value lies outside the defined limits (only appears if the "Limits" parameter 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 <sup>8</sup> 10 <sup>8</sup>   |
| Upper limit                 | Appears only if "limits" = "yes" was selected.  | -108 108   |

## 8.8.9 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-<br>tion and Calculation<br>(page 69)")   Auxiliary<br>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 I No   |
| Interruption outside limits | Determines whether the method should be interrupted if a value lies outside the defined limits (only appears if the "Limits" parameter 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.  | -108 108   |

| Upper limit | Appears only if "limits" = "yes" was selected. | -10 <sup>8</sup> 10 <sup>8</sup> |
|-------------|--|----------------------------------|

## 8.8.10 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 (control 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 <b>Continue after = Time interval</b> is selected. | 0106  |
| Print          | If selected, the instructions will be output to a connected printer.   | Yes I No  |

## 8.8.11 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", "Formula", 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 | Automatic   User defined |
|             | list.   |                          |



- 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".

| Result         | After selecting a result from the dropdown list, the system will 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 parameters. You can also enter any number. | Results list I Arbitrary         |
|----------------|---|----------------------------------|
| Result unit    | The unit of the result. (Is not automatically adjusted after changes to "Formula" or "Constant".) You can also use " <b>Proposal</b> " to select from a predefined suggestion list.   | Device list   Arbitrary          |
| Formula        | The formula for calculating the result. You can use " <b>Results proposals</b> " to select from a predefined suggestion list.   | Formula list   Arbitrary         |
| Constant       | 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 <sup>8</sup> 10 <sup>8</sup> |
| Upper limit    | Defines the upper result limit.  Appears only if "result limit" = "yes" was selected.   | -108108                          |

| Interruption outside limits  | Determines whether the method should be interrupted if a value lies outside the defined limits (only appears if the "Limits" parameter 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 functions. For example, this will allow you to define a maximum value 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 "Calculation" method function is used within a sample loop. | Yes I No |
| Multiple determination       | 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 samples            | 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 determination" = "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 determination" = "Yes" were selected.  | Yes I No |
| Record                       | If "yes" is selected for "Record", the multiple determination function will create a record listing the groups after a double determination whose relative standard deviation lies above a "Max. srel" number defined in the method.  Appears only if "Extra statistical functions" and "Multiple determination" = "Yes" were selected.  | Yes I No |

# 8.8.12 Report

This method function defines the type and scope of the data to be output for a report using the printer (see "Hardware>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<br>series out of loop: No I<br>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-<br>ple I Per Series I No Out<br>of Loop: No I Yes |

| Raw results          | The raw results produced during the determination   | within loop: Per Sam-<br>ple   Per Series   No Out<br>of Loop: No   Yes |
|----------------------|---|---|
| Measured value table | The table of measured values of the current sample (not available 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<br>Series   Non Per Sample<br>  Per Series   No        |
| E-C                  | Titration curve of the current sample. The potential is plotted against the load (not available out of loop).     | Yes I No  |
| E – †                | Titration curve of the current sample. The potential is plotted versus the time (not available outside of loop).  | Yes I No  |
| C-t                  | Titration curve of the current sample. The load is plotted against 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 "Driff" 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  |
| C - t & Drift - t    | Two overlaid titration curves "C-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  |

## 8.8.13 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         | Yes I No |
|             | processed after the method function "End of sample", or if the |          |
|             | titrator returns to Standby mode.                              |          |



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.

## 8.8.14 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.

## 8.8.15 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.

# 9 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 blank samples.

# 9.1 Sample series

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                | You can enter a brief comment about the series.   | Arbitrary   |
| Number of sam-<br>ples | Defines the number of samples to be analyzed.   | 1120        |
| Continuous run         | After each termination of the analysis (using series or methods) the analysis is automatically restarted (this is done until the process is canceled manually). | Yes I No    |

(Only for method type "Bromine index")



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.

# 9.2 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"). 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 automatically assigned when you create a new list entry.

| ID               | A user-defined name for the ID of the sample, in accordance with the "Sample" method function.                        | Arbitrary                                   |
|------------------|---|---|
| Sample size      | You can enter the sample size here. For fixed entry types, this field only appears as an info field.                  | 01000 [g]   [mL]<br>010 <sup>6</sup> [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 <b>Entry type</b> = <b>Pieces</b> or <b>Fixed pieces</b> was selected. | 0 1000                                      |

| Solvent weight       | Quantity of solvent in [g] in which the sample was extracted or dissolved.  Only for method type = <b>Ext. Extraction</b> .   | 01000     |
|----------------------|---|-----------|
| Wt. extracted sample | Total weight of sample in [g] which was extracted or dissolved in the solvent.  Only for method type = <b>Ext. Extraction</b> .   | 01000     |
| ID 2ID 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 for <b>Number of IDs</b> . | Arbitrary |
| Comment              | You can enter a brief comment about the series.   | Arbitrary |
| Correction factor    | Any correction factor that can be used in calculations.   | 0.0001106 |
| 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:





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

## 10 Results

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



- In the C20, only the results of the last analysis are saved, in the C30, 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 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 (C20) or the last two individual samples or series (C30).
- 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 stored 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 determination types: Sample 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") is started for a second time, existing data is 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: Changes to the results are marked by an asterisk in the record, e.g., VEQ\*.

# 10.1 Results 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.

#### Method type KF coul

#### Results proposal list for KF coul

| Result                  | Unit   | Replace "m"<br>according to entry<br>type | Formula R =          | Constant |
|-------------------------|--------|---|----------------------|----------|
| Coulometric consumption | mC     |   | R=ICEQ               | C = 1    |
| Mean consumption        | μg/min |   | R=(ICEQ/10.712)/TIME | C = 1    |
| Titration duration      | min    |   | R=TIME               | C = 1    |
| Total water content     | μg     |   | R=CW                 | C = 1    |

| Result  | Unit  | Replace "m"<br>according to entry<br>type | Formula R =                | Constant      |
|---------|-------|---|----------------------------|---------------|
| Content | mg    |   | R=                         | C = 1,000     |
|         | μg    |   | (ICEQ/10.712-TIME*DRIFT)/C | C = 1         |
|         | %     | g   | R=(ICEQ/10.712-            | C = 10,000    |
|         | ppm   | ml: m*d                                   | TIME*DRIFT)/(C*m)          | C = 1         |
|         | mg/g  | pc: m*wp                                  |                            | C = 1,000     |
|         | g/kg  |   |                            | C = 1,000     |
|         | μg/ml | ml  |                            | C = 1         |
|         | mg/ml | g: m/d                                    |                            | C = 1,000     |
|         | mg/pc | рс  |                            | C = 1,000     |
|         | μg/l  | ml  |                            | C = 0.001     |
|         |       | g: m/d                                    |                            |               |
|         | g/ml  | ml<br>g: m/d                              |                            | C = 1,000,000 |

# Ext. Extraction method type / KF coul. solution

# Results proposal list for external dissolution / extraction

| Result                          | Unit | Replace "m" according to entry type | Formula R =   | Constant                                  |
|---------------------------------|------|-------------------------------------|---|---|
| External dissolution (B in %)   | %    | g<br>ml: m*d<br>pc: m*wp            | R=C*[(msol+mext)/mext]-<br>B*msol/mext              | C=(ICEQ/10.712-<br>TIME*DRIFT)/(10,000*m) |
| External dissolution (B in ppm) | ppm  | g<br>ml: m*d<br>pc: m*wp            | R=C*[(msol+mext)/mext]-<br>B*msol/mext              | C=(ICEQ/10.712-<br>TIME*DRIFT)/m          |
| External extraction (B in %)    | %    | g<br>ml: m*d<br>pc: m*wp            | R=100/(100-C)*<br>(C*msol/mext-<br>B*msol/mext)     | C=(ICEQ/10.712-<br>TIME*DRIFT)/(10,000*m) |
| External extraction (B in ppm)  | ppm  | g<br>ml: m*d<br>pc: m*wp            | R=pw(6)/[pw(6)-<br>C]*(C*msol/mext-<br>B*msol/mext) | C=(ICEQ/10.712-<br>TIME*DRIFT)/m          |

# Method type Stromboli KF coul

# Results proposal list for Stromboli KF coul

| Result                     | Unit   | Replace "m"<br>according to<br>entry type | Formula R =                  | Constant |
|----------------------------|--------|---|------------------------------|----------|
| Coulometric consumption    | mC     |   | R=ICEQ                       | C = 1    |
| Mean consumption           | μg/min |   | R=(ICEQ/10.712)/TIME         | C = 1    |
| Titration duration         | min    |   | R=TIME                       | C = 1    |
| Total water content        | μg     |   | R=CW                         | C = 1    |
| Stromboli blank val-<br>ue | μg     |   | R=(ICEQ/10.712-TIME*DRIFT)/C | C = 1    |

| Result              | Unit  | Replace "m"<br>according to<br>entry type | Formula R =                              | Constant      |
|---------------------|-------|---|--|---------------|
| Content blank value | mg    |   | R=(ICEQ/10.712-TIME*DRIFT-               | C = 1,000     |
| compensated (B in   | μg    |   | B[Blank Stromboli])/C                    | C = 1         |
| µg)                 | %     | g   | R=(ICEQ/10.712-                          | C = 10,000    |
|                     | ppm   | ml: m*d<br>pc: m*wp                       | TIME*DRIFT-<br>B[Blank Stromboli])/(C*m) | C = 1         |
|                     | mg/g  |   |  | C = 1,000     |
|                     | g/kg  |   |  | C = 1,000     |
|                     | μg/ml | ml  |  | C = 1         |
|                     | mg/ml | g: m/d<br>pc                              |  | C = 1,000     |
|                     | mg/pc |   |  | C = 1,000     |
|                     | μg/l  | ml  |  | C = 0.001     |
|                     |       | g: m/d                                    |  |               |
|                     | g/ml  | ml<br>g: m/d                              |  | C = 1,000,000 |

## Method type bromine index

## Results proposals bromine index

| Result                  | Unit     | Replace "m"<br>according to<br>entry type | Formula R=                                    | Constant C=   |
|-------------------------|----------|---|---|---------------|
| Blank                   | mC       |   | R=ICEQ  | C = 1         |
| Blank                   | mg       |   | R=0.000828147*ICEQ                            | C = 1         |
| Bromine index           | mg/100g  | g<br>mL: m*d<br>pc: m*wp                  | R=ICEQ*C/m                                    | C = 0.0828147 |
| Bromine index (B in mC) | mg/100 g | g<br>mL: m*d<br>pc: m*wp                  | R=(ICEQ-B[Bromine index])*C/m                 | C = 0.0828147 |
| Bromine index (B in mg) | mg/100 g | g<br>mL: m*d<br>pc: m*wp                  | R=(0.000828147*ICEQ-<br>B[Bromine index])*C/m | C = 100       |

## 10.1.1 Internal calculations

#### Internal calculations for blank determinations

| Result | Unit | Replace "m" according to entry type | Formula R =                 | Constant  |
|--------|------|-------------------------------------|-----------------------------|-----------|
| Blank  | %    | g ml: m*d                           | R=(ICEQ/10.712-TIME*DRIFT)/ | C = 10000 |
|        | ppm  |                                     | (C*m)                       | C = 1     |

## More internal calculations

| Result | Unit | Replace "m" according to entry type | Formula R =    | Constant |
|--------|------|-------------------------------------|----------------|----------|
| CW     | μg   | -                                   | CW=ICEQ/10.712 | 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.

#### 10.4 Statistics

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 [x] and the standard deviation [s]:

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

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 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.

# 10.6 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.

# 10.7 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 Sequences

## 11.1 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 by choosing **AddToHome** (see "Description of Functions > The User Interface > Shortcuts and Direct Shortcuts"), 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 immediately. This enables you to start the analysis without any interfering dialog. | Yes I No  |
| Homescreen position | You can select the free position for the shortcut on the Homescreen.   | 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]<br>010 <sup>6</sup> [pcs.] |
| Density          | You can enter the sample's density, in [g/mL], here.  Does not appear for the <b>Entry type = Pieces</b> and <b>Fixed pieces</b> . | 0100  |
| Weight per piece | The weight in [g] per pirece.  Appears only if <b>Entry type = Pieces</b> or <b>Fixed pieces</b> was selected.                     | 0 1000                                      |
| Solvent weight   | Quantity of solvent in [g] in which the sample was extracted or dissolved.  Only for method type = <b>Ext. Extraction</b> .        | 01000                                       |

| ID 2ID 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 for <b>Number of IDs</b> . | Arbitrary |
|-------------------|---|-----------|
| Comment           | You can enter a brief comment about the series.   | Arbitrary |
| Correction factor | Any correction factor that can be used in calculations.   | 0.0001106 |
| Temperature       | The temperature in [°C] during the analysis. If temperature monitoring 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-<br>ples | Defines the number of samples to be analyzed.   | 1120  |
| 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]<br>010 <sup>6</sup> [pcs.] |
| Continuous run         | After each termination of the analysis (using series or methods) the analysis is automatically restarted (this is done until the process is canceled manually). | Yes I No                                    |

(Only for method type "Bromine index")



- 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.

# 11.2 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.

is not set, the series is automatically ended after the final sample.

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 max-

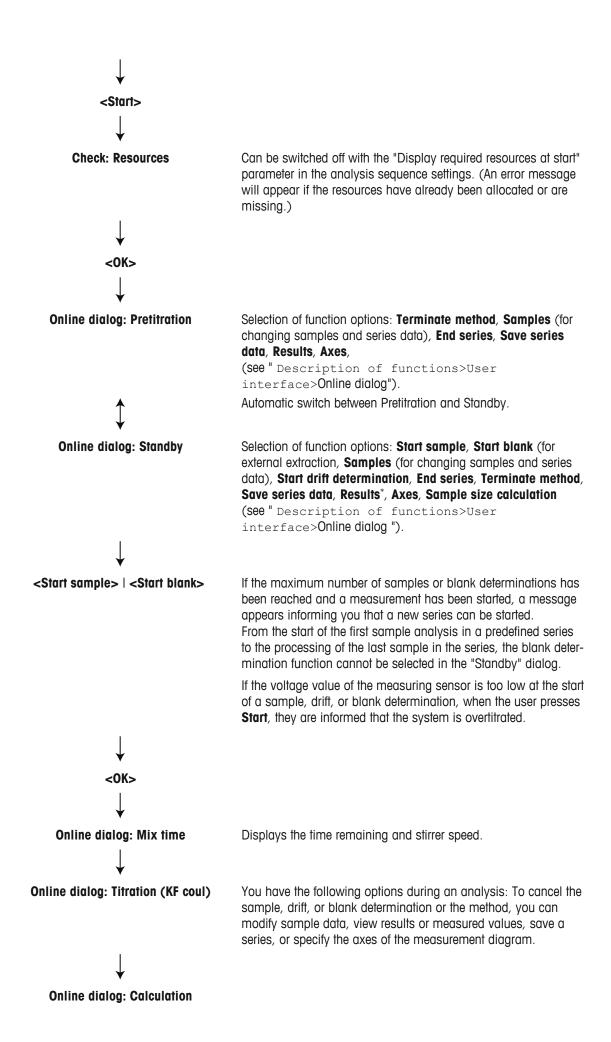
imum start driff is exceeded, you can end data entry and save the data by choosing **OK**.

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

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



This dialog can be switched off for a start using the "Shortcut parameters" dialog and the "Immediate start" parameter when creating or managing a shortcut.





#### Online dialog: Standby

\*During Standby or Pretitration mode, you can access the results of the current determination type (sample or 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)<sup>1</sup>
- Exclude samples1
- Perform outlier test1
- Results
- Display statistics
- Undo changes

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

#### **Drift determination**

There are several different ways to determine the drift:

- 1. 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").
- 2. Spontaneous drift determinations: The drift can be determined from Standby mode of any KF method. The driff determined here is used if the "Source for driff" 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").

## 11.2.1 Series analyses with the "Stromboli" oven sample changer

Before starting a Stromboli method, the pump must be switched on and the set temperature must be set. Every Stromboli method begins in the Start position (beaker is in the drift position). In this position, the pretitrations, manual and automatic drift determinations 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 Start analysis = "automatic" is set, the series is processed automatically. After each sample is processed, the next sample is analyzed without prompting. To enable automatic Start analysis, 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 system is switched to Pretitration mode during this time, the sample changer returns to the start position (beaker in the drift position) and performs the pretitration followed by the standby titration. The analysis of the next sample 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:

#### Start analysis

Each analysis starts in position 1, immediately after the "Drift" 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

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

i

Before actually canceling 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.

#### Canceling the drift 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 driff 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.

#### 11.2.2 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...

## 11.2.3 Switching between determination types

You can determine statistics for sample and blank 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. For example: If you carry out multiple sample determinations and then carry out a blank determination, the sample statistics are terminated. If you restart a sample determination, the memory for the samples is deleted and filled with new sample data.



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

## 11.2.4 Replacing the reagent solution

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 reagent 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.

# 12 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.

The system differentiates between the following types of analysis data:

| Displaying raw data      | Raw data is defined when you create a method or series. It is automatically 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 number of samples.   |
| Sample data              | All data from the analyzed samples, such as e.g. the sample size, sample density and sample ID.   |
| Resource data            | Data for all resources used during the execution of an analysis (for example, titration stand, blank value). 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, such as the drift value, or coulometric consumption to the end point (ICEQ).  |
|                          | 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.   |

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, blank)
- Results (the results of a calculation can then be used in a subsequent calculation.)

## 13 Evaluate and calculate

# 13.1 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 coul)         | 1           |        |
| Calculation                 |             | R1     |
| Calculation                 |             | R2     |
| Calculation                 |             | R3     |
| End of sample               |             |        |
| Sample (KF)                 |             |        |
| Titration stand (Stromboli) |             |        |
| Mix time                    |             |        |
| Titration (KF coul)         | 2           |        |
| Calculation                 |             | R4     |
| End of sample               |             |        |
| Calculation                 |             | R5     |

# 13.2 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.



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

| Basic symbol | Unit | Possible symbol additions | •           | Meaning  |
|--------------|------|---------------------------|-------------|--|
| IC           | [mC] | EQ                        | ICEQ (=CEQ) | Coulometric consumption to the end point of the titration method function. |

| Basic symbol | Unit  | Possible symbol additions | Symbol     | Meaning  |
|--------------|---|---------------------------|------------|--|
| TIME         | [min:s]   |                           | TIME       | Duration of a sample analysis<br>from the end of Standby mode<br>to the end of the Titration<br>method function (incl. waiting<br>for sample addition) |
| t            | [min:s]   |                           | t          | Duration of a sample analysis<br>(without waiting for sample<br>addition)  |
| E            | [mV]  | EQ                        | EEQ        | Potential at the end point of the titration method function.   |
| EST          | [mV]  |                           | EST        | Measured potential at the start of the titration method function   |
| DRIFT        | [µg/min]  |                           | DRIFT      | Consumption (mass) per minute for the titration method function (water quantity per time unit that penetrates the titration stand).                    |
| cw           | [ha]  |                           | CW         | Volume of water titrated up to EP (without drift or blank value correction).   |
| CWPOST       | [ha]  |                           | CWPOST     | Titrated volume of water during<br>a post-consumption measure-<br>ment (without drift or blank val-<br>ue correction).                                 |
| CWPOSTMean   | [µg/min]  |                           | CWPOSTMean | Determined quantity of titrated water per unit of time during the post-consumption measurement (without drift or blank value correction).              |
| В            | Arbitrary for KF stand, e.g. [%] and [ppm] [µg] for Stromboli |                           | B[Name]    | Blank value (water content of the solvent)   |
| Н            | Arbitrary   |                           | H[Name]    | Auxiliary value  |
| m            | [ml]<br>[g]<br>[pcs]  |                           | m          | 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          | Density of a sample or a standard  |
| wp           | [g/pcs]   |                           | wp         | Weight per piece (can be changed using the "Sample" method function)   |
| f            |   |                           | f          | Correction factor (as defined in the "Sample" method function)   |
| Ts           |   |                           | Ts         | Specified temperature: [°C, K, °F]   |

| Basic symbol | Unit                             | Possible symbol additions | Symbol   | Meaning  |
|--------------|----------------------------------|---------------------------|----------|--|
| CONT         | [mg/g] [mg/ml] [mg/pc] [%] [ppm] |                           | CONT     | Concentration of a liquid KF standard  |
| Rx           | Arbitrary                        |                           | Rx       | Result x.  |
| С            |                                  |                           | С        | 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] | Mean value of a result Rx  |
| S            | Arbitrary                        | Rx                        | s[Rx]    | Standard deviation of a result Rx  |
| srel         | [%]                              | Rx                        | srel[Rx] | The relative standard deviation of the result Rx.  |
| n            |                                  |                           | n        | Sample number  |

## 13.3 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

A typical example of a formula within the "Calculation" method function would be the expression **R=ICEQ** in the "Formula" parameter, where R represents the coulometric consumption in [mC] until the end point is reached. 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.

#### **Conditions**

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!)

#### 13.3.1 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.

## **Simplification**

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

|                  | Explanation                         | Examples                                   |
|------------------|-------------------------------------|--|
| Basic symbol and | Taken together, they serve as an    | ICEQ corresponds to the coulometric con-   |
| symbol extension | identifier for the analysis data.   | sumption in [mC] to the end point of the   |
|                  |                                     | titration.                                 |
| Group index      | Specifies which method function     | ICEQ[3] represents the titration end point |
|                  | within a method function group gen- | of the third loop of the method of type    |
|                  | erates the analysis data.           | Stromboli (model C30 only).                |

# 13.3.2 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                 | /                            |                          |       |

Logical operators are only permitted within the formulas of "Condition" subfunctions (or parameters).

i

# Index

|   |                                       |          |   | Results, KF                           | 57       |
|---|---------------------------------------|----------|---|---------------------------------------|----------|
| _ |                                       |          |   | Switch                                | 67       |
| A |                                       |          |   | Drift determination                   | 16, 66   |
|   | Account policies                      |          | E |                                       |          |
|   | Defining                              | 26       |   | Emergency stop (reset)                | 13       |
|   | Action When Exceeding Usable          | 28       |   | End of sample                         | 54       |
|   | Life                                  | 00       |   | End series                            | 16       |
|   | Add Result All Results                | 60<br>59 |   | Evaluation and calculation            | 68       |
|   | Analysis and Resources Behav-         | 59       |   | Exceeding Usable Life                 | 28       |
|   | ior                                   |          |   | Experts                               | 26       |
|   | Action When Exceeding                 | 28       |   | Expired Resources External Extraction | 18<br>67 |
|   | Life Span                             |          |   | Analysis Sequence                     | 67       |
|   | Action When Exceeding                 | 28       | _ | Analysis ocquence                     |          |
|   | Usable Life                           |          | F |                                       |          |
|   | Analysis Sequence Set-                | 27       |   | Fingerprint reader                    | 22, 22   |
|   | tings                                 | 07       |   | Registration                          | 22       |
|   | Resource Behavior<br>Analysis Data    | 27<br>68 |   | Firmware History                      | 32       |
|   | Naming conventions                    | 69       |   | Formula examples Bromine index        | 59       |
|   | Using                                 | 71       |   | Ext. extraction                       | 58       |
|   | Analysis Data in Formulas             | 7.<br>71 |   | KF coul                               | 57       |
|   | Analysis sequence                     | 64       |   | Stromboli KF coul                     | 58       |
|   | Auxiliary values                      | 33, 51   |   | Formula simplification                | 71       |
| В |                                       |          |   | Formulas                              | 71       |
| _ | Balance                               | 19       |   | Fundamentals                          |          |
|   | Barcode reader                        | 20       | _ | Bromine index                         | 11       |
|   | Beep                                  | 31       | G |                                       |          |
|   | Blank determinations                  | •        |   | General safety information            | 8        |
|   | Internal calculations                 | 59       |   | Generator electrodes                  |          |
|   | Blank value                           | 51       |   | with diaphragm                        | 11       |
|   | Dialog                                | 33       |   | without diaphragm                     | 11       |
|   | External extraction                   | 47       |   | Global settings                       |          |
|   | Starting determination                | 17       |   | Analysis and Resources                | 26       |
|   | Board data<br>Board firmware          | 32<br>32 |   | Behavior                              | 20       |
|   | Bromine index                         | 32       |   | Reagent control<br>System             | 29<br>24 |
|   | ASTM D1492                            | 38       |   | User Management                       | 25       |
|   | Formula examples                      | 59       | _ | Goor Managornom                       |          |
|   | Fundamentals                          | 11       | Н |                                       |          |
|   | Number of loops                       | 42       |   | Hardware                              |          |
|   | Outside loop                          | 44       |   | Balance                               | 19       |
| _ | Within loop                           | 44       |   | Barcode reader                        | 20       |
| C |                                       |          |   | Fingerprint reader<br>LevelSens       | 22<br>23 |
|   | Cancel determination                  |          |   | Network settings                      | 22       |
|   | Online dialog                         | 17       |   | PC settings                           | 22       |
|   | Configure barcode reader              | 20       |   | Peripherals                           | 19, 19   |
|   | Configure USB stick                   | 20       |   | Printer                               | 20       |
|   | Configuring a balance                 | 19       |   | Pumps                                 | 19       |
|   | Control                               | 50       |   | Sensors                               | 18       |
|   | Titration EP(Coul) Titration KF(Coul) | 50<br>49 |   | Titration stands                      | 24       |
|   | Current increments                    | 49       |   | USB-Stick<br>Hidden method functions  | 20<br>54 |
|   | Titration EP(Coul)                    | 50       |   | Homescreen                            | 13       |
|   | Titration KF(Coul)                    | 49       | _ | Homescreen                            | 13       |
| D |                                       |          | ı | Identification, titrator              | 24       |
|   | Date and time                         | 24       |   | Import/Export, data                   | 31       |
|   | Defining pumps                        | 19       |   | Increments                            | 49, 50   |
|   | Determination types                   |          |   | Indexing of method functions          | 69       |
|   |                                       |          |   |                                       |          |

|   | Info button                          | 13       |   | Method syntax                            | 41           |
|---|--------------------------------------|----------|---|--|--------------|
|   | Instruction                          | 52       |   | Method Templates                         | 40           |
|   | Internal calculations                |          |   | Method types                             | 38           |
|   | Blank determinations                 | 59       |   | Methods                                  |              |
|   | More                                 | 59       |   | Creating                                 | 39           |
| K |                                      |          |   | Modify                                   | 40<br>38     |
|   | Karl Fischer stand                   |          |   | Preprogrammed METTLER TOLEDO balances    | 19           |
|   | Setting up                           | 24       |   | METTLER TOLEDO Methods                   | 38           |
|   | Karl Fischer titration               |          |   | Mix time                                 | 49           |
|   | Measurement principle                | 10       |   | Monitoring                               |              |
|   | Keyboard layout                      | 0.1      |   | Usable life and life span                | 28           |
|   | Define                               | 31       | N |  |              |
| L |                                      |          |   | Naming conventions for calcula-          | 69           |
|   | Language, setting                    | 30       |   | tions                                    | 00           |
|   | Layout of terminal                   | 13       |   | Network settings                         | 22           |
|   | Level sensor                         | 23       | _ | -  | -            |
|   | LevelSens                            | 23       | 0 |  |              |
|   | Activate                             | 23       |   | Online Dialog                            | 15           |
|   | Life span Life span monitoring       | 28<br>29 |   | Open series Operation of the touchscreen | 64<br>13     |
|   | Loops                                | 42       |   | Outlier test                             | 60           |
| _ | 20000                                |          | _ | Cumor 1001                               |              |
| M | Managina                             | 00       | P | DO#i                                     | 00           |
|   | Managing groups Manual operations    | 26       |   | PC settings                              | 22<br>19, 19 |
|   | Pump                                 | 35       |   | Peripherals Balance                      | 19, 19       |
|   | Sensor                               | 36       |   | Balances                                 | 20           |
|   | Solvent manager                      | 35       |   | Barcode reader                           | 20           |
|   | Stirrer                              | 35       |   | Fingerprint reader                       | 22           |
|   | Mathematical functions and           | 72       |   | Printer                                  | 20           |
|   | operators                            |          |   | USB data export                          | 20           |
|   | Measured Values                      | 17       |   | USB stick                                | 20           |
|   | Method<br>Delete                     | 41       |   | Polarized sensors Preloaded Methods      | 18<br>38     |
|   | Start                                | 41       |   | Pretitration                             | 30           |
|   | Method editor                        | 38       |   | Automatic switch                         | 16           |
|   | Method functions                     |          |   | Online dialog                            | 16           |
|   | Hidden method functions              | 54       |   | Printer                                  | 20           |
|   | Out of loop                          | 44       |   | Setup                                    | 20           |
|   | Overview                             | 44       |   | USB data export                          | 20, 20       |
|   | Possible number                      | 42<br>43 |   | Protective clothing                      | 9            |
|   | Within loop Method functions, hidden | 43       |   | Pump<br>Manual operations                | 35           |
|   | Pretitration                         | 54       | _ | Mariaar operations                       |              |
|   | Standby                              | 54       | R |  |              |
|   | Method functions, Parameters         |          |   | Reagent manager                          | 29           |
|   | Auxiliary value                      | 51       |   | Setup                                    | 19           |
|   | Blank value                          | 51       |   | Reagents                                 | 00           |
|   | Calculation                          | 52       |   | Monitoring<br>Registration               | 29           |
|   | Drift determination                  | 46<br>54 |   | Fingerprint reader                       | 22           |
|   | End of sample<br>Instruction         | 54<br>52 |   | Reset button                             | 13           |
|   | Mix time                             | 49       |   | Reset to factory settings                | 32           |
|   | Record                               | 53       |   | Results                                  |              |
|   | Sample (KF)                          | 46       |   | Deleting                                 | 62           |
|   | Title                                | 45       |   | Display                                  | 15-16        |
|   | Titration (EP Coul)                  | 50       |   | Results proposals                        | 60           |
|   | Titration (KF Coul)                  | 49       |   | Routine                                  | 26           |
|   | Titration stand                      | 48       | _ | Running tasks                            | 14           |
|   | Method ID                            | 38       |   |  |              |

| S |                                 |          |    | Time and date             | 24     |
|---|---------------------------------|----------|----|---------------------------|--------|
|   | Safety Information              |          |    | Title                     |        |
|   | General                         | 8        |    | Parameter                 | 45     |
|   | Protective clothing             | 9        |    | Titrant _                 |        |
|   | Signal words                    | 8        |    | Replacement               | 67     |
|   | Warning Symbols                 | 8        |    | Titrant replacement       | 67     |
|   | Sample (KF)                     | 46       |    | Titration                 |        |
|   | Sample analysis                 | 16       |    | Network settings          | 22     |
|   | Sample data                     |          |    | PC settings               | 22     |
|   | Change                          | 16       |    | Titration (EP Coul)       | 50     |
|   | Sample Results                  | 61       |    | Titration (KF Coul)       | 49     |
|   | Sample series                   |          |    | Titration stand           | 48     |
|   | Sample parameters               | 55       |    | Setting up                | 24     |
|   | Sample size                     | 16       |    | Titrator identification   | 24     |
|   | Standby                         | 17       | U  |                           |        |
|   | Screen, setting                 | 30       | U  |                           |        |
|   | Sensor                          |          |    | Undo Changes              | 61     |
|   | Polarized                       | 37       |    | Updating                  | 33     |
|   | Sensors                         |          |    | Upgrading                 | 33     |
|   | Configure                       | 18       |    | Usable life               | 28     |
|   | Series analysis                 | 66       |    | Usable life and life span |        |
|   | Series data                     | 30       |    | Monitoring                | 28     |
|   | Save                            | 16       |    | USB compact printer       | 20     |
|   | Series templates                | 55       |    | USB data export           | 20, 20 |
|   | Service & Maintenance           | 00       |    | USB data export box       | 20     |
|   | Board data                      | 32       |    | USB-Stick                 | 20     |
|   | Board firmware                  | 32       |    | User Interface            | 13     |
|   | Data Import/Export              | 31       |    | Input Fields              | 14     |
|   | Reset to factory settings       | 32       |    | User management           |        |
|   | Terminal                        | 32       |    | Managing groups           | 26     |
|   | Titrator Firmware History       | 32       |    | User rights               |        |
|   | Update                          | 33       |    | Experts                   | 26     |
|   | Upgrade                         | 33       |    | Routine                   | 26     |
|   | Settings                        | 33       |    | User settings             |        |
|   | global                          | 24       |    | Keyboards                 | 31     |
|   | Shortcuts                       | 14       |    | Language                  | 30     |
|   | Manage                          | 31       |    | Screen                    | 30     |
|   | Signal words                    | 8        |    | Shortcuts                 | 31     |
|   | Solvent capacity                | 29       |    |                           |        |
|   | Solvent manager                 | 23       | V  |                           |        |
|   | Manual operations               | 35       |    | Values                    |        |
|   | Sorting lists                   | 14       |    | Auxiliary                 | 33     |
|   | Standby                         | 16       |    | Blank                     | 33     |
|   | Automatic switch                | 16       | W  |                           |        |
|   | Online dialog                   | 16       | VV |                           |        |
|   |                                 |          |    | Warning Symbols           | 8      |
|   | Start analysis dialog           | 15<br>63 |    |                           |        |
|   | Starting an Analysis            |          |    |                           |        |
|   | Starting analysis<br>Statistics | 64<br>60 |    |                           |        |
|   |                                 |          |    |                           |        |
|   | Stirrer<br>Stop method          | 35<br>16 |    |                           |        |
|   | Stop method                     |          |    |                           |        |
|   | Stopping methods                | 41       |    |                           |        |
|   | Stromboli Anglysis asguspes     | 60       |    |                           |        |
|   | Analysis sequence               | 66       |    |                           |        |
|   | Stromboli TTL                   | 0.4      |    |                           |        |
|   | Setting up                      | 24       |    |                           |        |
|   | Symbol list                     | 69<br>24 |    |                           |        |
|   | System settings                 | 24       |    |                           |        |
| T |                                 |          |    |                           |        |
|   | Tape printer                    | 20       |    |                           |        |
|   | Tasks menu                      | 14       |    |                           |        |
|   | I USAS IIIGIIU                  | 14       |    |                           |        |

# To protect your product's future:

METTLER TOLEDO Service assures the quality, measuring accuracy and preservation of value of this product for years to come.

Please request full details about our attractive terms of service.

www.mt.com/titration

For more information

Mettler-Toledo AG, Analytical

CH-8603 Schwerzenbach, Switzerland Tel. +41 (0)44 806 77 11 Fax +41 (0)44 806 73 50 www.mt.com

Subject to technical changes. © Mettler-Toledo AG 11/2013 51710632C

