

# 860 KF Thermoprep



## Manual

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# **860 KF Thermoprep**

## **Manual**

Technical Communication  
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This documentation is an original document.

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### **Disclaimer**

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

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# 1 Introduction

## 1.1 Instrument description

The 860 KF Thermoprep is used wherever the heating of a sample and/or the thermal expulsion of moisture in solids or liquids is required. In combination with a coulometric or volumetric KF titrator, the 860 KF Thermoprep is the ideal analysis system for water content determination in samples that contain disruptive components or from which moisture can be removed only with difficulty.

One of its decisive advantages is reducing sample preparation to a minimum. Thanks to the use of hermetically sealed sample vessels ("headspace vials"), samples can be filled directly on-site. The PTFE-coated septa guarantee a constant, unaltered water content, even after prolonged downtimes.

The sample heated in the oven module releases its moisture in the form of water vapor, which is conveyed into a measuring cell with the aid of a gas flow. An air pump is installed to generate the gas flow. An inlet valve is available for nitrogen or other inert gases. The moisture can be determined in the measuring cell either coulometrically or volumetrically using Karl Fischer titration.

### 1.1.1 Device components

The 860 KF Thermoprep has the following components:

- **Oven**  
Oven module made of aluminum with software-operated temperature control for heating the sample vessel.
- **Fan**  
Propeller fan for cooling the oven module.
- **Inlet valve**  
Valve for switching over the source of the gas flow.
- **Air pump**  
Pump for generating the gas flow.
- **Outlet heater**  
Heating tubing for preventing the condensation of moisture.
- **Support rods**  
Guidance device with needle adapter and tubing for the gas flow.
- **Operating unit**  
Monochrome LCD display and keyboard.

### 1.1.2 Intended use

The 860 KF Thermoprep is designed for usage as an auxiliary device for sample preparation in analytical laboratories. Its main area of application is water content determination using Karl Fischer titration (coulometric or volumetric). The 860 KF Thermoprep enables the application of thermal gas extraction technology.

This device is suitable for processing chemicals and flammable samples. Therefore, the use of the 860 KF Thermoprep requires the user to have basic knowledge and experience in handling toxic and caustic substances. Knowledge regarding the application of fire prevention measures prescribed for laboratories is also mandatory.

## 1.2 About the documentation





### CAUTION

Please read through this documentation carefully before putting the device into operation. The documentation contains information and warnings which the user must follow in order to ensure safe operation of the device.

### 1.2.1 Symbols and conventions

The following symbols and formatting may appear in this documentation:

(5-12)	<b>Cross-reference to figure legend</b> The first number refers to the figure number, the second to the device part in the figure.
<b>1</b>	<b>Instruction step</b> Perform the steps one after the other.
<b>Method</b>	<b>Dialog text, parameter</b> in the software
<b>File ► New</b>	Menu or menu item
<b>[Continue]</b>	<b>Button</b> or <b>key</b>
	<b>WARNING</b> This symbol draws attention to a possible life-threatening hazard or risk of injury.
	<b>WARNING</b> This symbol draws attention to a possible hazard due to electrical current.

**WARNING**

This symbol draws attention to a possible hazard due to heat or hot instrument parts.

**WARNING**

This symbol draws attention to a possible biological hazard.

**WARNING**

Warning of optical radiation

**CAUTION**

This symbol draws attention to possible damage to devices or device parts.


**NOTICE**

This symbol highlights additional information and tips.

## 1.3 Displaying accessories

Up-to-date information on the scope of delivery and on optional accessories can be found on the Metrohm website.

### 1 Searching for a product on the website

- Go to <https://www.metrohm.com>.
- Click on .
- Enter the article number of the product (e.g. **2.1001.0010**) into the search field and press **[Enter]**.

The search result is displayed.

### 2 Displaying product information

- To display the products matching the search term, click on **Product models**.
- Click on the desired product.

Detailed information regarding the product is displayed.

### 3 Displaying accessories and downloading the accessories list

- To display the accessories, scroll down to **Accessories and more**.
  - The **scope of delivery** is displayed.
  - Click on **[Optional parts]** for the optional accessories.



- To download the accessories list, click on **[Download accessories PDF]** under **Accessories and more**.



**NOTE**

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Metrohm recommends keeping the accessories list for reference purposes.

## 2 Safety

### 2.1 Safety instructions

#### 2.1.1 General notes on safety



##### **WARNING**

---

Operate this device only according to the information contained in this documentation.

This device left the factory in a flawless state in terms of technical safety. The following instructions must be observed carefully to preserve this status and ensure non-hazardous operation of the device.

#### 2.1.2 Electrical safety

Electrical safety when working with the device is ensured as part of the international standard IEC 61010.



##### **WARNING**

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Only personnel qualified by Metrohm are authorized to carry out service work on electronic components.



##### **WARNING**

---

Never open the housing of the device. The device could become damaged. There is a considerable risk of injury if live components are touched.

There are no parts inside the housing which can be serviced or replaced by the user.

#### **Supply voltage**



##### **WARNING**

---

An incorrect supply voltage can damage the device.

Operate this device only with a supply voltage specified for it (refer to the rear of the device).



### 2.1.4 Flammable samples and solvents



#### WARNING

---

All relevant safety measures are to be observed when working with flammable samples or when adding solvents.

- Use nitrogen or another inert gas for conveying the moisture.
- Set up the instrument in a well-ventilated location (e.g., fume cupboard).
- Keep all sources of flame far from the workplace.
- Clean up spilled liquids and solids immediately.
- Follow the safety instructions of the chemical manufacturer.

### 3 Overview of the instrument

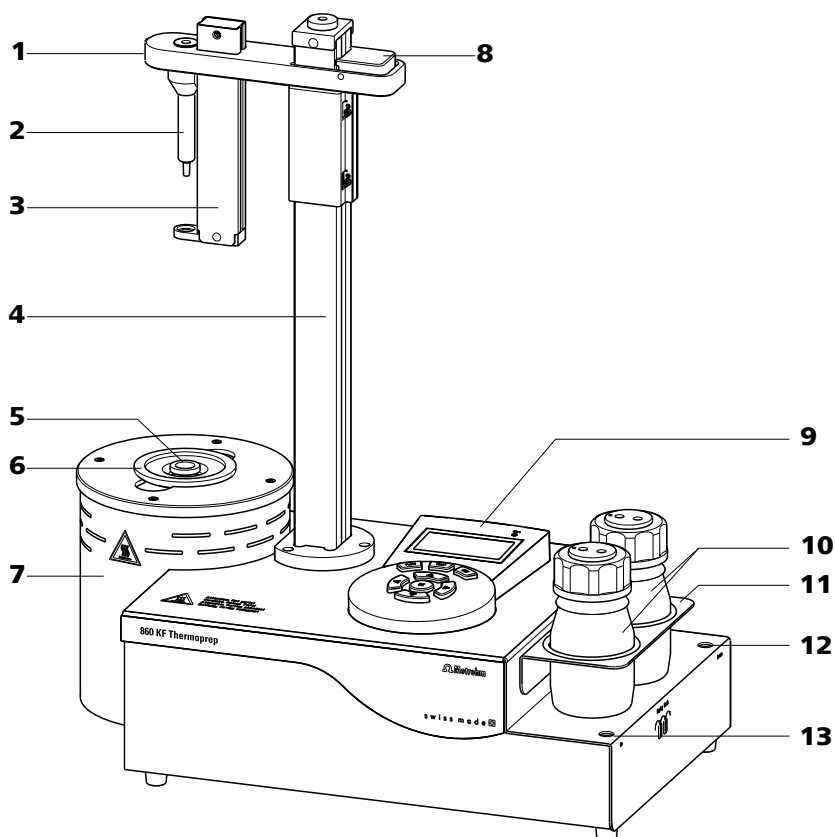


Figure 1 Front 860 KF Thermoprep

<b>1</b> Guide head	<b>2</b> Needle holder (6.2049.040) With needle adapter.
<b>3</b> Spring sleeve With vessel stripper.	<b>4</b> Guidance rod
<b>5</b> Sample vessel (6.2419.007) With 6 ml content.	<b>6</b> Sample holder (6.2063.010)
<b>7</b> Oven module With sheet metal housing.	<b>8</b> Fixing lever
<b>9</b> Operating unit With display and keyboard.	<b>10</b> Drying flasks (6.1608.050) With 6.1602.145 drying flask insert.
<b>11</b> Drying flask holder	<b>12</b> Gas inlet With M6 thread.
<b>13</b> Gas outlet With M6 thread.	

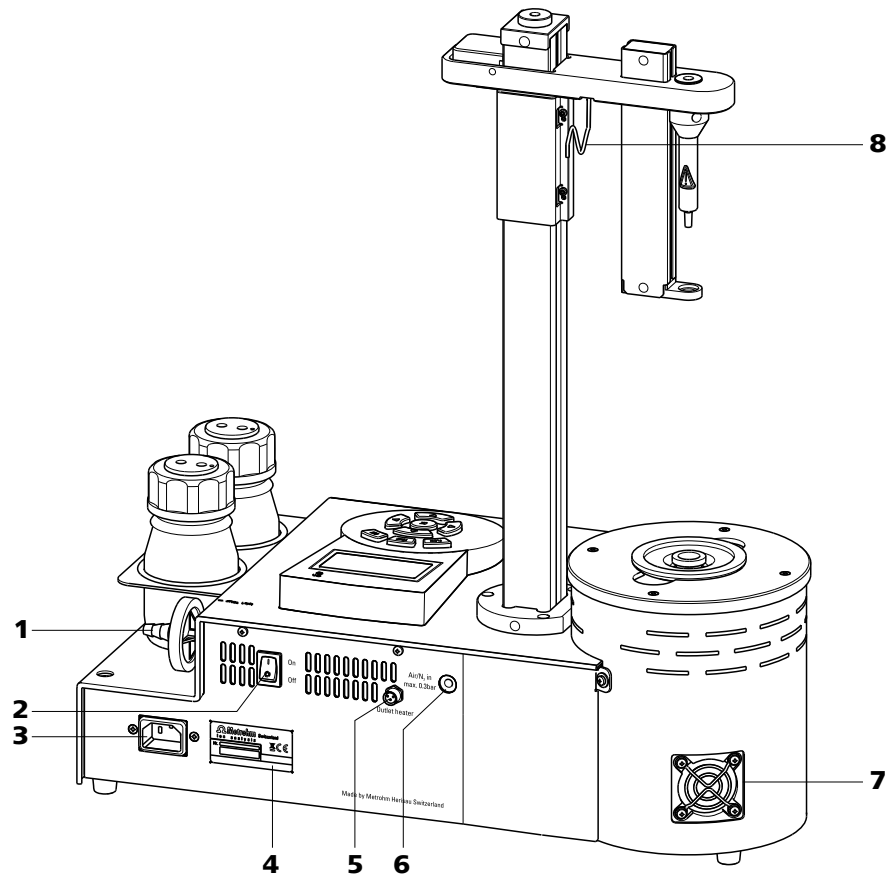


Figure 2 Rear 860 KF Thermoprep

<p><b>1 Air pump inlet</b> With 6.2724.010 dust filter.</p>	<p><b>2 Mains switch</b></p>
<p><b>3 Mains connection socket</b></p>	<p><b>4 Type plate</b> Contains specifications concerning mains voltage and serial number.</p>
<p><b>5 Outlet heating connection</b> For the heating tube.</p>	<p><b>6 Air/nitrogen connector</b> With M6 interior thread. Inlet for external gassing.</p>
<p><b>7 Fan</b> For cooling the oven module.</p>	<p><b>8 Cable holder (6.2060.010)</b></p>



## 4 Installation

### 4.1 Setting up the device

#### 4.1.1 Packaging

The product and accessories are supplied in protective special packaging. Keep this packaging to ensure safe transportation of the product. If a transport lock is present, keep this as well for future reuse.

#### 4.1.2 Checks

Inspect the delivery immediately upon receipt:

- Check the delivery against the delivery note to ensure completeness.
- Check the product for damage.
- If the delivery is incomplete or damaged, contact your regional Metrohm representative.

#### 4.1.3 Location

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

Place the instrument in a location of the laboratory which is suitable for operation and free of vibrations and which provides protection against corrosive atmosphere and contamination by chemicals.

The instrument should be protected against excessive temperature fluctuations and direct sunlight.

## 4.2 Connecting the instrument to the power grid



### WARNING

#### Electric shock from electrical potential

Risk of injury by touching live components or through moisture on live parts.

- Never open the housing of the instrument while the power cord is still connected.
- Protect live parts (e.g. power supply unit, power cord, connection sockets) against moisture.
- Unplug the power plug immediately if you suspect that moisture has gotten inside the instrument.
- Only personnel who have been issued Metrohm qualifications may perform service and repair work on electrical and electronic parts.

#### Connecting the power cord

#### Accessories

Power cord with the following specifications:

- Length: max. 2 m
- Number of cores: 3, with protective conductor
- Instrument plug: IEC 60320 type C13
- Conductor cross-section 3x min. 0.75 mm<sup>2</sup> / 18 AWG
- Power plug:
  - according to customer requirement (6.2122.XX0)
  - min. 10 A



### NOTE

Do not use a not permitted power cord!

#### 1 Plugging in the power cord

- Plug the power cord into the instrument's power socket.
- Connect the power cord to the power grid.



## 4.3 Mounting the support rod

The support rod with the guide head is enclosed separately with the 860 KF Thermoprep and must first be mounted.

Proceed as follows:

- 1 Move the guide head of the support rod a bit downwards by pressing the locking lever.

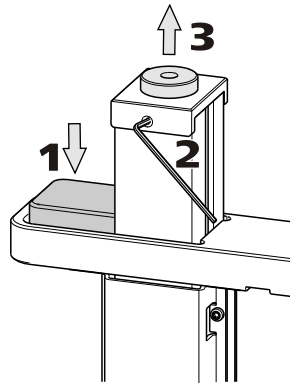


Figure 3 Loosening the adapter

- 2 Loosen the adapter on the head of the support rod with a hex key.
- 3 Removing the adapter.
- 4 Insert the tubing jutting out of the housing from below into the support rod.



### NOTE

This tubing conveys the gas from the flow controller upward through the support rod to the guide head. From there, the gas is guided to the double hollow needle (see next chapter).

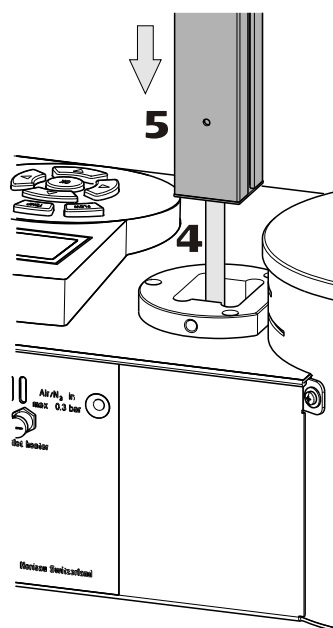


Figure 4 Mounting the support rod

- 5 Insert the support rod into the opening intended for this purpose from above and push it downwards until it hits the stop. Observe the orientation of the support rod.
- 6 Pull out the tubing inside the support rod as far as possible. You might want to use a piece of sand paper to be able to better grasp the tubing.

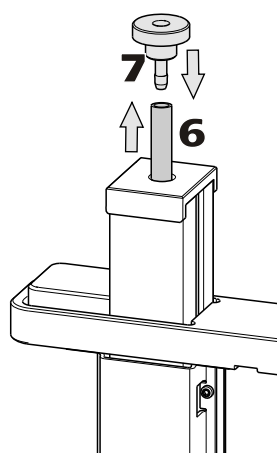


Figure 5 Mounting the tubing

- 7 Fasten the tubing on the olive of the 4.860.4260 adapter.
- 8 Pull out the support rod as far as possible so that the adapter rests closely on the support rod.

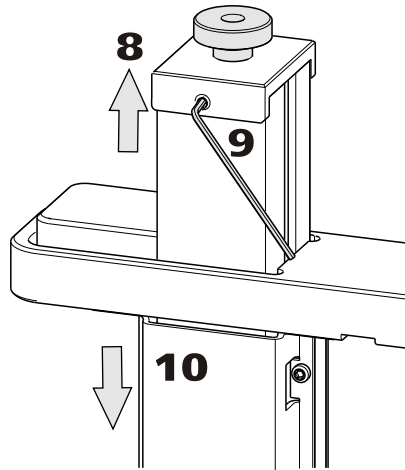


Figure 6 Attaching the adapter

- 9** Attach the adapter to the support rod. Fasten the screw into the support rod with the aid of a hex key.
- 10** Carefully push the support rod downwards.

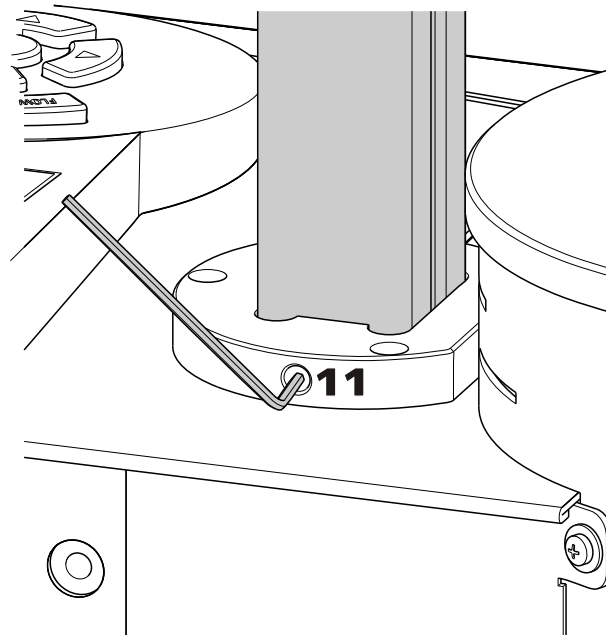


Figure 7 Mounted support rod

- 11** Fix the support rod with the V.0434.008 hex screw following the illustration. The screw and the corresponding hex key are enclosed separately with the instrument.

## 4.4 Mounting the sample holder

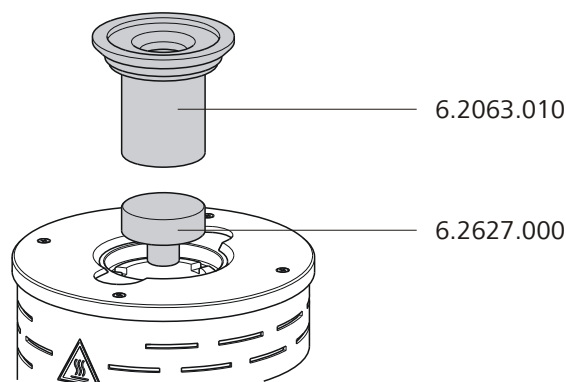


Figure 8 Mounting the sample holder



### NOTE

The sample holder may not be inserted or removed unless it has cooled down.

The dimensions of the supplied sample holder are optimized for the usage of 6.2419.007 6-mL sample vials by Metrohm. This ensures the best possible transfer of heat between oven and sample.

- 1 Place the **6.2627.000 oven insert** into the oven from above.
- 2 Place the **6.2063.010 sample holder** into the oven from above.

If vessels with other dimensions are to be used, then individually modified sample holders can be ordered. The precise vessel dimensions (including tolerances) are required. Non-standard sample vessels may only have dimensions within the following limit values:

- Diameter 10.0 - 32.0 mm
- Immersion depth 20.0 - 45.0 mm

## 4.5 Mounting the needles

The length of the needle holder defines how deeply the needle for the gas inlet penetrates the sample vessel. The **6.2049.040 needle holder** with a length of 58 mm ensures that the needle penetrates the liquid or powdery sample. The carrier gas flows through the sample and efficiently removes the contained moisture.

If there is a risk that the heated sample may block the needle, then use the **6.2049.050 needle holder** with a length of 73 mm. In this case, the needle for the gas inlet penetrates the sample vessel slightly more than the outlet needle and has no contact with the sample. The 6.2049.050 needle holder can be ordered from Metrohm if required.

Needle holders with the dimensions required for situations calling for special sample vessels can be supplied by Metrohm upon request.

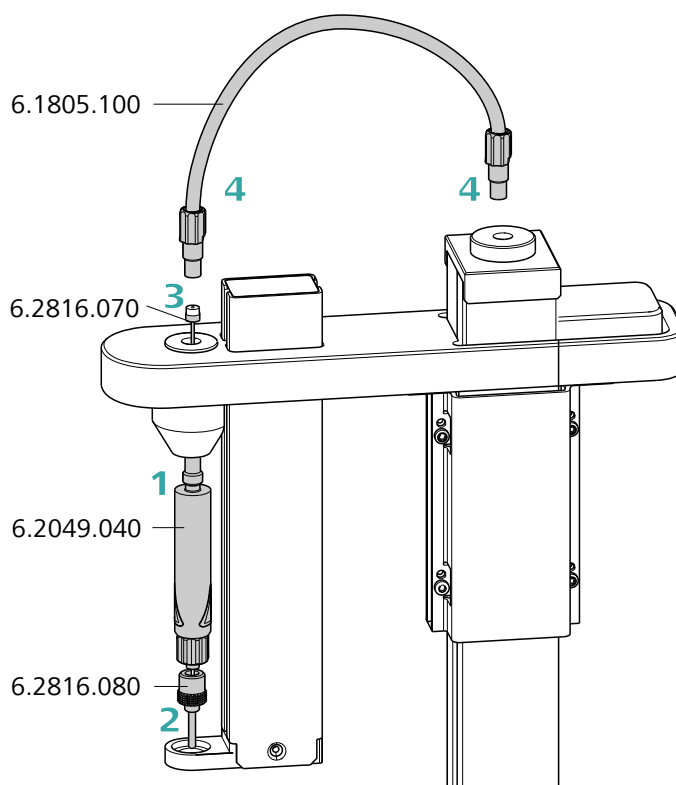


Figure 9 Mounting the needles

Mount the needles as follows:

- 1** Screw the **6.2049.040 needle holder** into the distributor on the guide head.

- 2 Screw the **6.2816.080 outlet needle** onto the Luer connector of the needle holder.
- 3 Carefully guide the **6.2816.070 needle for gas inlet** into the opening of the distributor from above and allow it to drop down.

**NOTE**

Ensure that the white PTFE seal sits firmly on the needle.

- 4 Screw the **6.1805.100 FEP tubing** onto the opening of the distributor by hand. Screw the other end of the tubing onto the opening on the upper side of the support rod.

## 4.6 Mounting the heating tubing

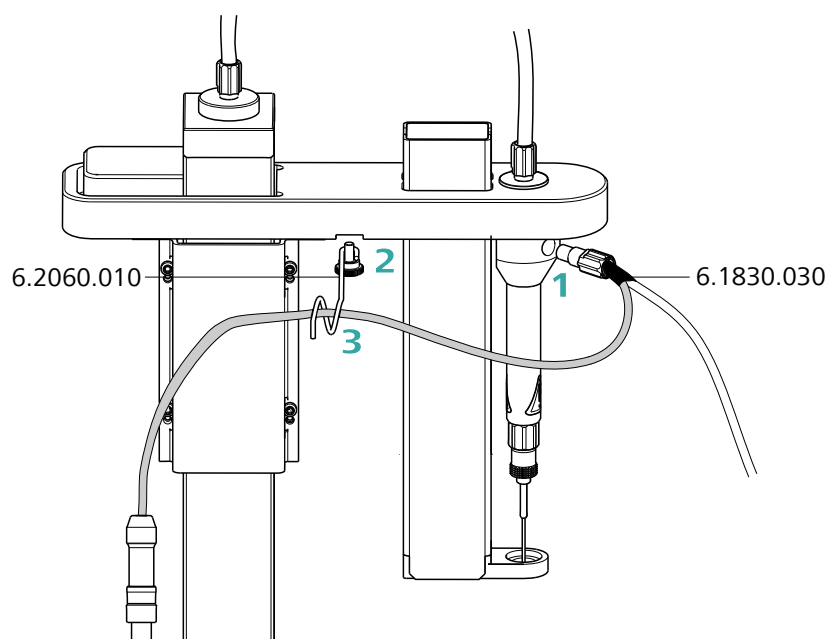


Figure 10 Mounting the heating tubing (rear view)

Proceed as follows:

- 1 Screw the M6 connector of the **6.1830.030 heating tubing** into the lateral opening of the distributor on the guide head.
- 2 Use the accompanying knurled screw to screw the **6.2060.010 cable holder** tightly onto the guide head from below.



- 3 Guide the heating tubing cable through the eyelet of the cable holder.

**NOTE**

The cable holder prevents the cable of the heating tubing from coming into contact with parts of the housing that are hot.

- 4 Connect the heating tubing cable to the **Outlet heater** connector on the rear of the instrument.

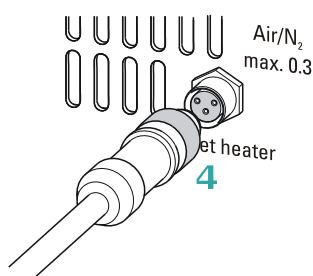


Figure 11 Connecting the heating tubing

Rotate the plug in such a way that the three contact pins correspond to the sequence of the corresponding socket openings. Press the plug towards the socket and rotate the front knurled screw clockwise.

**NOTE**

The heating jacket of the heating tubing is heated up to approx. 40 to 50 °C as soon as the instrument is switched on. This prevents condensation of moisture in the tubing when it is expelled from the sample and transferred into a KF titration cell with a carrier gas.

## 4.7 Mounting the dust filter

The installed air pump must be protected from dust. For this reason, a **6.2724.010 dust filter** must be placed on the air inlet (**Inlet filter**) on the right-hand side of the housing..

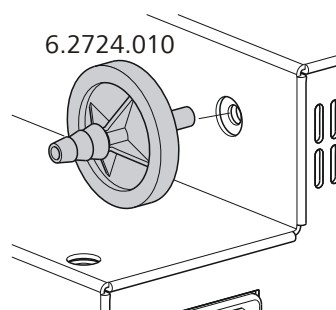


Figure 12 Mounting the dust filter

The dust filter should be replaced once a year.

## 4.8 Assembling drying flasks

Two drying flasks with desiccant are integrated into the gas flow to dry the supplied gas. Dust (e.g. from the desiccant) must be prevented from finding its way into the sample vessel.

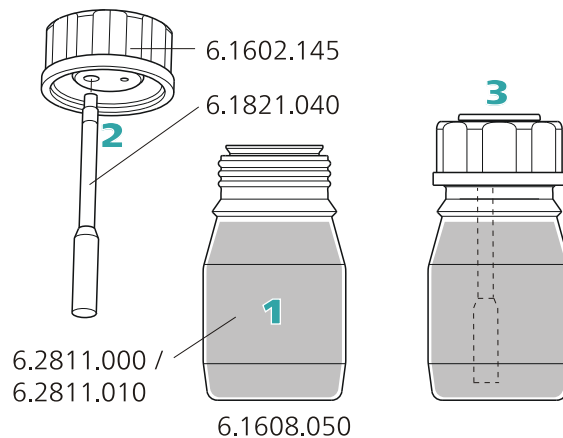


Figure 13 Preparing the drying flasks

Prepare the two drying flasks as follows:

- 1** Fill both **6.1608.050 drying flasks** with **6.2811.000/6.2811.010 molecular sieve**.

- 2 Screw one **6.1821.040 filter tube** into each of the **6.1602.145 drying flask caps** from below. Tighten the filter tubes by hand.
- 3 Screw the two drying flask caps with the filter tubes onto the drying flasks. Tighten the caps by hand.

**NOTE**

If drying flask caps or filter tubes are not tightened well, the gas flow may not be precise and regular. The error message "Flow rate error" usually occurs when screw connections are not tight.

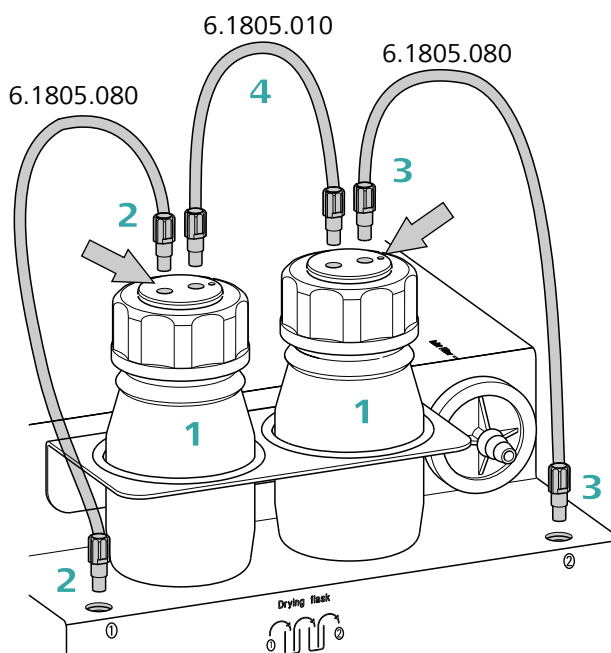


Figure 14 Mounting the tubings

Mount the FEP tubings as follows:

- 1 Insert the two drying flasks that have been prepared into the holders, see figure.
- 2 Screw one 6.1805.080 FEP tubing (25 cm length) to the gas outlet (at the front, labeled **1**). Screw the other tubing end on the front drying flask to the M6 connector **without** point marking (see left-hand arrow).
- 3 Screw the second 6.1805.080 FEP tubing (25 cm length) to the gas inlet (at the rear, labeled **2**). Screw the other tubing end on the rear

drying flask to the M6 connector **with** point marking (see right-hand arrow).

- 4 Screw the 6.1805.010 FEP tubing (13 cm length) tightly onto the remaining M6 connectors of the drying flasks.

The **Drying flask** marking on the right-hand side of the instrument displays the diagram for the tubing.



#### NOTE

Tighten the screw connections well by hand.

## 4.9 Assembling the air/nitrogen connector

If compressed air, nitrogen or another gas is to be used for transferring the expelled moisture, then a separate connector is available at the rear of the instrument.

A tube with M6 thread can be connected directly to the **Air/N<sub>2</sub> in** connector. Enclosed with the instrument is the **6.1808.040 M6/M8 tubing adapter** for a tubing with an M8 thread. The **6.1808.050 M8/tubing olive** can additionally be put in place in order to connect a simple tubing.

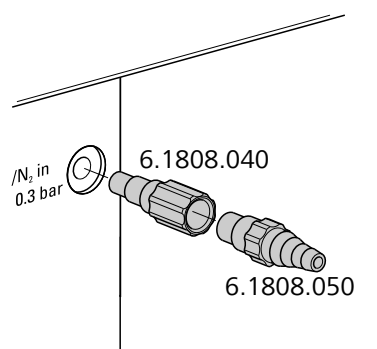


Figure 15 External gas connector



#### WARNING

If gas is fed from a pressure line or a pressure vessel, then an upstream pressure reduction valve must be installed. The gas pressure must not exceed 0.3 bar of overpressure.

## 4.10 Inserting the heating tube into the KF titration cell

The 860 KF Thermoprep can be used in combination with a KF coulometer or a volumetric KF titrator. The mounting of the tip of the heating tube is accomplished in different ways for the respective KF titration cells.

### Coulometric KF titration cell

- Remove the protective cover from the tip of the heating tube and the E.3010.032 O-ring.
- Disassemble the accompanying **6.1446.170 heating tube stopper** into three parts.

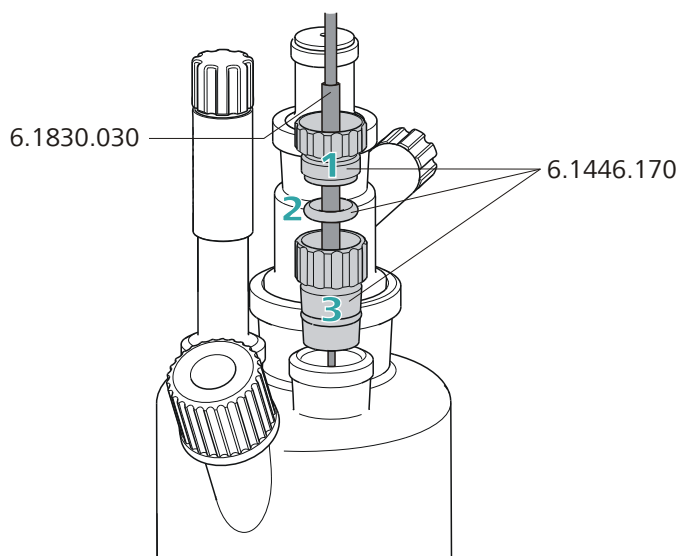


Figure 16 Coulometric KF titration cell

Proceed as follows:

- 1** Guide the upper part of the heating tube stopper over the tip of the **6.1830.030 heating tube** as shown in the illustration.
- 2** Guide the O-ring of the heating tube stopper over the heating tube.
- 3** Guide the lower part of the heating tube stopper over the heating tube and screw the three parts together (not too tightly).
- 4** Insert the stopper with the tip of the heating tube into one of the two SGJ openings of the KF titration cell

- 5 Shift the tip vertically in such a way that the outlet opening of the tubing is immersed as deeply as possible. The tip must not however be permitted to get in the way of the stirring bar in the KF titration cell. Afterwards give the heating tube stopper its final tightening.

### Volumetric KF titration cell

- Remove the protective cover from the tip of the heating tube and the E.3010.032 O-ring. The latter you will still need.
- A **6.2730.020 septum stopper** is enclosed with every KF titrator made by Metrohm. Disassemble this septum stopper into three parts and remove the septum. It will not be required.

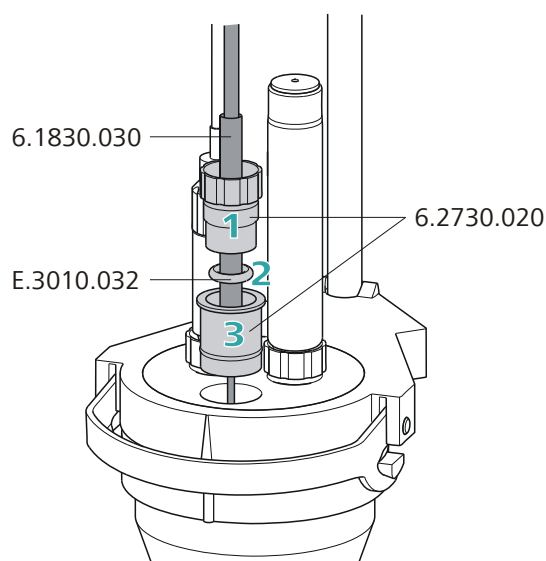


Figure 17 Volumetric KF titration cell

Proceed as follows:

- 1 Guide the upper part of the septum stopper over the tip of the **6.1830.030 heating tube** as shown in the illustration.
- 2 Guide the **E.3010.032** O-ring over the heating tube.  
This O-ring is also part of the 6.1244.040 set of seals that is enclosed with each KF titrator. It cannot be reordered individually.
- 3 Guide the lower part of the septum stopper over the heating tube and screw the three parts together (not too tightly).
- 4 Insert the stopper with the tip of the heating tube into the front opening of the KF titration cell



- 5 Shift the height of the tip of the heating tube vertically in such a way that the outlet opening of the tubing is immersed as deeply as possible. The tip must not however be permitted to get in the way of the stirring bar in the KF titration cell. Afterwards give the septum stopper its final tightening.

## 5 Operation

### 5.1 Switching the instrument on and off

The on/off switch is located on the rear of the instrument.

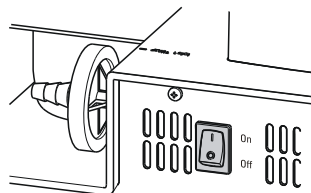


Figure 18 Power switch

The oven is switched on automatically after the instrument has been switched on and it is heated to the temperature that was set last. At the same time, the outlet heating of the heating tubing is also switched on.

### 5.2 The keypad

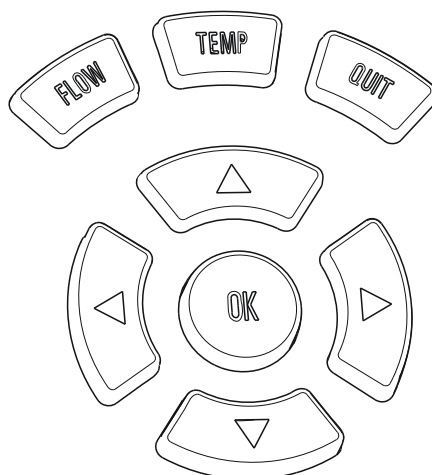


Figure 19 Keypad of the operating unit

#### Key functions in the main dialog

[FLOW]	Switches the gas flow on or off.
[TEMP]	Switches between the two target temperatures.
[QUIT]	No function.
[OK]	Opens parameter menu.
[◀]	Reduces the gas flow by 1 mL/min during operation. Press the key shortly.



- [▶] Increases the gas flow by 1 mL/min during operation. Press the key shortly.
- [▲] Reduces the brightness of the display.
- [▼] Increases the brightness of the display.

## 5.3 The display

### 5.3.1 Display elements







Figure 20 Main dialog

<b>1 Temperature display</b> Shows the measured oven temperature.	<b>2 Icon of the status display</b>
<b>3 Gas flow display</b> Shows the set gas supply and the flow rate.	<b>4 Target temperature</b> Shows the set specification (temp. 1 or temp. 2).
<b>5 Status display</b> Shows status and error messages.	

### 5.3.2 Status display

Table 1 Icons of the status display

Icon	Meaning
	The oven is heated.
	The oven is cooled.
	The predefined temperature has been reached.
	An error has occurred. The error type is shown in the status display.

### 5.3.3 Error messages

Table 2 Error messages

Error message	Cause	Remedy
<b>Flow rate error</b>	<i>The tubing system is leaking or the flow rate is set too high.</i>	Tighten all tubing connections and screws of the drying flasks.  Check the function of the pump.
	<i>The pressure is too low or too high during external gas supply.</i>	Regulate the pressure of the external gas to 0.1 - 0.3 bar.
	<i>The flow sensor is defective.</i>	Contact a service engineer.
<b>Max. temp. reached</b>	<i>The oven temperature is higher than 265 °C.</i>	Switch off the instrument immediately and let the oven cool down.
	<i>The temperature control might be defective.</i>	If the error reoccurs again when switching the instrument on, contact a service engineer.
<b>Adj. data missing</b>	<i>The adjustment data of the instrument is not correct.</i>	The instrument must be adjusted again. Contact a service engineer.

## 5.4 Modifying numerical values and settings

The settings of the instrument can be modified in two ways:

- Selection of predefined values from a list. Parameters with selection lists are marked with a colon (:).
- Modifying numerical values.

The various parameters can be selected using the [▼] and [▲] arrow keys. The current line will be displayed reversed.

### 5.4.1 Selecting from a selection list

You can modify parameters with selection lists (e.g. **Flow source:**) using the **[▶]** or **[◀]** arrow keys. The next value in the selection list is usually displayed.

Apply the modified value with the **[OK]** key.

### 5.4.2 Modifying numerical values

Modify parameters with numerical values using the arrow keys:

- 1 Use the **[▶]** or **[◀]** arrow keys to select a digit of the numerical value.

The selected digit is displayed in black on white. The colors for the rest of the line remain reversed.

**Temp. 1 100 °C**

- 2 Modify the digit using the **[▲]** or **[▼]** arrow keys.

- 3 Select the next digit and modify it, etc.

- 4 Apply the modified value with the **[OK]** key.

Press **[QUIT]** if the previous value is to be restored.

## 5.5 Parameters

**[OK]**

The instrument parameters can be set in a menu which can be opened with the **[OK]** key. Quit the parameter menu with **[QUIT]**.



#### NOTE

Modified values are applied by pressing **[OK]**. A modification is discarded with **[QUIT]**.

### Temp. 1

Default value for temperature 1.

Input range	<b>50 to 250 °C</b> (Increment: <b>1</b> )
Default value	<b>100 °C</b>

### Temp. 2

Default value for temperature 2.

Input range	<b>50 to 250 °C</b> (Increment: <b>1</b> )
Default value	<b>100 °C</b>

**Gas flow**

Default value for the gas flow.

Input range	<b>10 to 150 mL/min</b> (Increment: <b>1</b> )
Default value	<b>50 mL/min</b>

**Gas type**

Selection of the gas.

Selection	<b>air   nitrogen   other</b>
Default value	<b>air</b>

**other**

A measuring factor must be specified for the gas used when selecting **other**. The measuring factor is used for the correct measurement of the flow rate.

**Meas. factor**

Measuring factor for measuring the flow rate of the gas. This setting is only possible if **other** was selected as gas. For air and nitrogen, the corresponding measuring factor is inserted automatically.

*Only if 'gas type' = 'other'.*

Input range	<b>0.001 to 2.000</b> (Increment: <b>0.001</b> )
Default value	<b>1.000</b>

**Flow source**

Selection of the gas supply.

Selection	<b>pump   valve</b>
Default value	<b>pump</b>

**pump**

Built-in air pump.

**valve**

Inlet valve for inert gas.

## 5.6 Configuration

### [TEMP]

The basic settings of the instrument can be configured in the setup menu. You can open the menu by pressing the **[TEMP]** key while switching the instrument on. Press **[QUIT]** to close the menu.



#### NOTE

The setup menu can be opened even if the key lock is on.

### Lock keys

Key lock. The **[OK]**, **[◀]** and **[▶]** keys can be locked. With this, you can prevent that changes are made in the parameter menu or that the gas flow is changed during a determination. If this function is enabled (**on**), you can only switch between temperature 1 and temperature 2 and switch the gas flow on or off. The brightness of the display can still be adjusted.

Selection	<b>off   on</b>
Default value	<b>off</b>

### Temp.corr

Temperature correction. The default value for the temperature control of the oven can be influenced by this correction value. In this way, you can adjust a temperature difference between the oven module and the sample in the vessel if required.

Input range	<b>-10 to 10 °C (Increment: 1)</b>
Default value	<b>0 °C</b>

The determination of the necessary temperature correction can be accomplished with a special oven insert and should be performed by a service engineer. Contact your responsible Metrohm supplier.

## 6 Performing a determination

Moisture determinations according to the heating method require the conditioning of the entire tubing system and of the KF titration cell before a determination can be carried out. Because the sample vials and the septum seals may contain small amounts of moisture, three to five blank value determinations must be carried out with sealed, empty vessels.

Solid or liquid samples are weighed out into sample vials, which are then sealed. We recommend the use of 6.2419.007 sample vials with 6.1448.057 septum seals. The silicone septum of the aluminum cap resists temperatures of up to 250 °C and has proven itself to be an optimum solution.

### 6.1 Conditioning the system

The entire system must be conditioned prior to use, i. e. it must be absolutely water-free. The oven must be heated to the desired working temperature for this purpose. The needle must be inserted into a sealed sample vessel (conditioning vessel) in order to flush the entire tubing system with the carrier gas. The gas supply is thus connected with the heating tube.

#### 6.1.1 Preparing the KF titration cell

##### Filling the KF titration cell

The KF titration cell must remain filled with conditioned working medium when not in use. The working medium must be replaced from time to time, even though the sample is not transferred into the working medium during the heating-out process.

Prepare the KF titration cell as follows:

- 1 Fill the KF titration cell with working medium. We recommend approximately 150 mL of working medium when using a Coulometer cell and approximately 35 mL with a volumetric KF titration cell. The shaft of the heating tube should, however, not be immersed.
- 2 Align the tip of the heating tube against the vessel wall.
- 3 Switch on the stirrer. Set the stirring rate to high.



It is advantageous to have air bubbles stirred into the working medium and forcefully broken up in the process. The air in the KF titration cell must also be dried.

### Conditioning the working medium

- 1 Load a conditioning method on the KF titrator and begin the conditioning by pressing the **[Start]** key.

You will find the necessary settings in the KF titrator manual.



#### NOTE

It is imperative that an **extraction time** of **at least 300 seconds** be set for a determination using the heating method.

## 6.1.2 Preparing the 860 KF Thermoprep

### Switch on the 860 KF Thermoprep and select the temperature

The necessary settings on the 860 KF Thermoprep:

- 1 Switch on the instrument.
- 2 Select one of the preset temperatures using the **[TEMP]** key.  
If you would like to change **Temp. 1** or **Temp. 2**, press **[OK]**.

### Inserting the conditioning vessel

- 1 Seal an empty sample vessel tightly with a septum seal. To do this, use the crimping tongs.
- 2 Insert the sample vessel into the oven.



#### NOTE

**Caution!** The oven is hot. Use the crimping tongs to insert the sample vessel.

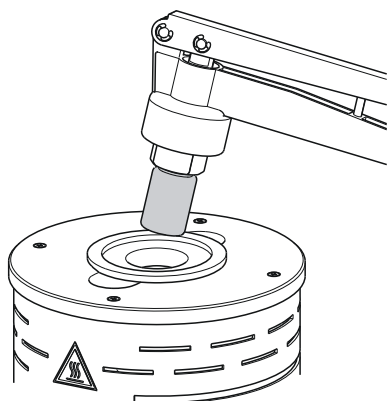


Figure 21 Insert the sample vessel into the oven

### Inserting the needle

- 1 Grip the guide head with both hands and press the fixing lever.

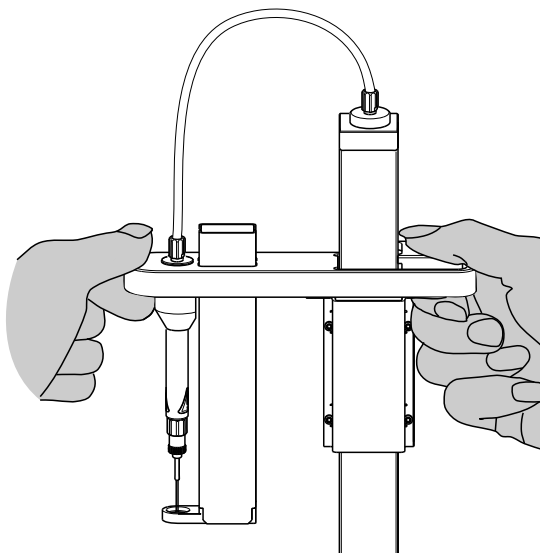


Figure 22 Move the guide head down

- 2 Move the guide head down and insert the needle into the sample vessel.

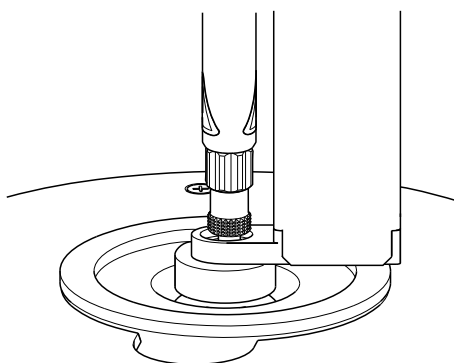


Figure 23 Insert the needle

### Switching on the gas flow

- 1 Press the **[OK]** key and adjust the flow rate (**Gas flow**), e. g. 50 mL/min. Use the arrow keys **[▶]** or **[◀]** to select the number of decimal places, and then **[▲]** or **[▼]** to change the value.
- 2 Confirm the entry by pressing **[OK]** and select **Flow source**.
- 3 Using the arrow keys **[▶]** or **[◀]**, select **pump** or **valve** (if you are using an external carrier gas) and confirm by pressing **[OK]**.
- 4 Close the Parameters menu by pressing **[QUIT]**.
- 5 Press the **[FLOW]** key.  
The gas flow is switched on. The flow rate gradually approaches the level set.



#### NOTE

The carrier gas should emerge from the tip of the heating tube into the working medium as tiny bubbles and be spun there forcefully. Alter the stirring rate if necessary. The tip of the tubing should be immersed until it reaches the bottom of the KF titration cell and be rotated against the vessel wall.

Allow the system to condition for a few minutes and swirl the KF titration cell now and again in order to remove the last traces of moisture. Check whether the set oven temperature has been reached.

**NOTE**

The signal drift on the KF titrator should be less than 20 µg/min (or µL/min) and remain stable for at least 2 minutes before the blank value determination can be started.

## 6.2 Determining the blank value

Because the surface of the sample vessels and the air enclosed in the vessel could contain moisture, it is imperative that three to five blank value determinations be carried out. One determination with an empty, tightly sealed sample vessel is carried out in each case. The result is calculated as the quantity of water expelled from the sample vessel. The titration parameters used for a regular sample must also be applied to blank value determinations. Select a relative stop drift of 5 µg/min (or µL/min) as the stop criterion.

### Preparing the blank

The system should already be conditioned. A conditioning vessel must be in the oven at this stage and the needle must already be inserted.

- 1 Seal three to five sample vessels tightly with the crimping tongs.
- 2 Check that the oven temperature and the gas flow rate correspond to the values which have been set.
- 3 Wait until the working medium in the KF titration vessel has been conditioned and the signal drift is stable.

### Starting the blank value determination

- 1 Start the determination by pressing the **[Start]** key on the KF titrator.
- 2 Use both hands to move the guide head of the 860 KF Thermoprep upward while pressing the fixing lever.  
Refer to the illustrations on *pages 33 ff.*
- 3 Remove the conditioning vessel from the oven with the crimping tongs.

**NOTE**

**Caution!** The vessel is hot.

- 4 Place the blank into the oven using the crimping tongs.
- 5 Use both hands to move the guide head downward and insert the needle into the blank.

**Waiting for the determination**

- 1 Wait until the determination is finished. Conditioning will then be resumed automatically.
- 2 Repeat the blank value determination with the next blank.

## 6.3 Determining samples

The blank value must be subtracted from the calculated consumption when determining a sample.

**Preparing the KF titrator**

- 1 Load a suitable method.
- 2 Start the conditioning by pressing the **[Start]** key.

**Preparing the sample**

- 1 Weigh the sample into a sample vessel and seal it tightly with the crimping tongs.
- 2 Check that the oven temperature and the gas flow rate correspond to the values which have been set.
- 3 Wait until the working medium in the KF titration vessel has been conditioned and the signal drift is stable.

### Starting the sample determination

- 1 Start the determination by pressing the **[Start]** key on the KF titrator.
- 2 Use both hands to move the guide head of the 860 KF Thermoprep upward while pressing the fixing lever.  
Refer to the illustrations on *pages 33 ff.*
- 3 Remove the blank from the oven with the crimping tongs.



#### NOTE

**Caution!** The vessel is hot.

- 4 Place the sample vessel into the oven using the crimping tongs.
- 5 Use both hands to move the guide head downward and insert the needle into the sample vessel.

### Waiting for the determination

- 1 Wait until the determination is finished. Conditioning will then be resumed automatically.
- 2 Repeat the determination with the next blank.

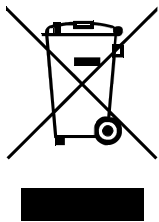


## 8 Troubleshooting

### 8.1 Problems and their solutions

<b>Problem</b>	<b>Cause</b>	<b>Remedy</b>
<b>The drift is very high during conditioning.</b>	<i>Molecular sieve of drying flasks and/or the titration cell exhausted.</i>	Replace molecular sieve.
	<i>The titration cell is leaking</i>	Check seals. If necessary, replace.
<b>The titration time is too long.</b>	<i>The sample is non-homogenous</i>	Reduce the size of the sample before weighing in.
	<i>Not the entirety of the moisture expelled.</i>	Select more stringent switch-off criteria on the KF titrator: lower stop drift, higher switch-off delay time.
	<i>Tubing connections leaking</i>	Inspect tubing and replace as necessary.
<b>The results are spread widely.</b>	<i>Molecular sieve of drying flasks exhausted.</i>	Replace molecular sieve.
	<i>Condensate in the heating tube.</i>	<ul style="list-style-type: none"> <li>▪ Dry the tubing.</li> <li>▪ Reduce the gas flow.</li> <li>▪ Perhaps lower the oven temperature.</li> </ul>
	<i>Gas flow too high.</i>	Reduce the gas flow.
	<i>The sample is non-homogenous</i>	Reduce the size of the sample before weighing in.
<b>Selected gas flow is not achieved.</b>	<i>System is leaking</i>	Check all tubing connections and drying flasks for leaks.
	<i>Pressure from external gas supply either too low or too high.</i>	Adjust gas pressure (max. 0.3 bar).
	<i>Pump defective</i>	Contact a service technician.

# 9 Recycling and disposal



Properly dispose of chemicals and of the product to reduce negative effects on the environment and public health. Local authorities, waste disposal companies or dealers provide more detailed information on disposal. Observe the WEEE EU directive (WEEE = Waste Electrical and Electronic Equipment) for the proper disposal of waste electronic equipment within the European Union.

## 10 Appendix

### 10.1 Practical notes

#### Carrier gas selection

If the hot sample is sensitive to air or oxygen (decomposition) and if it releases substances that disrupt the KF reaction, then nitrogen (N<sub>2</sub>) should be used as the carrier gas.

#### Temperature setting

The temperature selected should be as high as the sample allows (high temperature = shorter analysis time). The sample may not, however, decompose. It must only release water and not any oxidizable substances.

The temperature displayed refers to the temperature in the heating block and not to the sample temperature. Depending on the vial size used, the gas flow and the temperature setting, the actual temperature of the sample can deviate up to 10%.

#### Gas flow

Once the needle is inserted into the sample vessel until it stops, the gas flow moves through the sample and then through the outlet needle into the titration vessel where the moisture is titrated. If the needle is not permitted to immerse into the sample, then a longer needle holder can be used.

The gas flow should be kept as low as possible. Particularly in the case of very moist samples, care must be taken to ensure that water is not released too quickly and in excessively large quantities into the titration vessel. In addition, there is the danger of condensate formation in the heating tubing. The working medium in the cell must be able to absorb the moisture that is expelled without delay. Normally, a flow rate of 40 to 60 mL/min is optimal.

The tolerances for gas flow measurement depend on the system: They depend on the pressure, temperature, measuring system and the gas itself, and a deviation of 20% is possible.

The gas flow measurement is calibrated with air; other gases deviate more or less strongly from that.

If required, the flow rate of the gas can be checked using a flow measuring instrument at the local pressure and current temperature. An individual measuring factor can be entered with the **Meas. factor** parameter so that the corrected gas flow is displayed. At the same time, however, **other** must be selected for the **Gas type** parameter.

**Extraction time**

An extraction time of min. 5 minutes should be set on the titrator in order to prevent the titration from being canceled before the sample has released its water.

**Conditioning the system**

The system must be conditioned with an empty, sealed sample vial (conditioning vial) before a determination is made.

**10.2 Literature**

- E. Scholz, *Hydranal® Manual*, Riedel-de Haën
- P. Bruttel, R. Schlink, *Water Determination by Karl Fischer Titration* Monograph, Metrohm, 2006, Order no. 8.026.5013

## 11 Technical specifications

### 11.1 Oven

<i>Temperature range</i>	50 - 250 °C
<i>Accuracy</i>	±3 °C
<i>Correction range</i>	-10 - +10 °C
<i>Heating cartridges performance</i>	165 W (typical) Dependent on supply voltage
<i>Heating rate</i>	Typically 15 °C/min (at 80 - 180 °C, 230 V) Dependent on temperature, supply voltage, sample amount and vessel dimensions
<i>Cooling rate</i>	Typically 9 °C/min (at 180 - 80 °C) Dependent on temperature, sample amount and vessel dimensions

### 11.2 Gas flow

<i>Flow range</i>	10 - 150 mL/min Under normal conditions, calibrated with air at approx. +25 °C and 1013 mbar. Deviations of gas amount per time unit of ±20% are possible.
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### 11.3 Outlet heater

<i>Socket connection</i>	U = 16 ±1 V I ≤ 0.8 A
<i>Typical tubing temperature</i>	approx. 50 °C



## 11.4 Ambient conditions

<i>Nominal function range</i>	+5 °C - +45 °C, at max. 80% humidity, non-condensing
<i>Storage</i>	+5 - +45 °C
<i>Altitude / Pressure range</i>	Max. 2,000 m.a.s.l. / min. 700 mbar
<i>Pollution degree</i>	2

## 11.5 Energy supply

<i>Nominal voltage range</i>	100 - 120 / 220 - 240 V ( $\pm 10\%$ )
<i>Nominal frequency</i>	50 / 60 Hz
<i>Power consumption</i>	200 W
<i>Protection</i>	2 ATH
<i>Overvoltage category</i>	II

## 11.6 Reference conditions

<i>Ambient temperature</i>	25 °C ( $\pm 3$ °C)
<i>Relative humidity</i>	$\leq 60\%$
<i>Supply voltage</i>	230 V

## 11.7 Dimensions

<i>Width</i>	0.44 m
<i>Height</i>	0.18 m (without support rod) 0.50 m (with support rod)
<i>Depth</i>	0.23 m
<i>Weight (without accessories)</i>	8.17 kg
<i>Material</i>	
<i>Housing</i>	Cover and bottom: Steel sheet, stove-enameled
<i>Oven covering</i>	PTFE
<i>Operating unit</i>	Polycarbonate / Acrylonitrile butadiene styrene (PC/ABS)

