

# 860 KF Thermoprep



## Manual

8.860.8003EN / 2019-12-02





Metrohm AG

CH-9100 Herisau

Switzerland

Phone +41 71 353 85 85

Fax +41 71 353 89 01

[info@metrohm.com](mailto:info@metrohm.com)

[www.metrohm.com](http://www.metrohm.com)

# **860 KF Thermoprep**

## **Manual**

Technical Communication  
Metrohm AG  
CH-9100 Herisau  
techcom@metrohm.com

This documentation is protected by copyright. All rights reserved.

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

# Table of contents

<b>1</b>	<b>Introduction</b>	<b>1</b>
1.1	Instrument description	1
1.1.1	Instrument components	1
1.1.2	Intended use	2
1.2	About the documentation	2
1.2.1	Symbols and conventions	2
1.3	Safety instructions	3
1.3.1	General notes on safety	3
1.3.2	Electrical safety	3
1.3.3	Personnel safety	4
1.3.4	Flammable samples and solvents	5
1.3.5	Recycling and disposal	5
<b>2</b>	<b>Overview of the instrument</b>	<b>6</b>
<b>3</b>	<b>Installation</b>	<b>8</b>
3.1	Setting up the instrument	8
3.1.1	Packaging	8
3.1.2	Checks	8
3.1.3	Location	8
3.2	Connecting the instrument to the power grid	8
3.3	Mounting the support rod	9
3.4	Mounting the sample holder	12
3.5	Mounting the needles	13
3.6	Mounting the heating tubing	15
3.7	Mounting the dust filter	16
3.8	Assembling drying flasks	17
3.9	Assembling the air/nitrogen connector	19
3.10	Inserting the heating tube into the KF titration cell	19
<b>4</b>	<b>Operation</b>	<b>22</b>
4.1	Switching the instrument on and off	22
4.2	The keypad	22
4.3	The display	23
4.3.1	Display elements	23
4.3.2	Status display	23
4.3.3	Error messages	24



## — 11 —

Figure 1	Front 860 KF Thermoprep .....	6
Figure 2	Rear 860 KF Thermoprep .....	7
Figure 3	Loosening the adapter .....	9
Figure 4	Mounting the support rod .....	10
Figure 5	Mounting the tubing .....	11
Figure 6	Attaching the adapter .....	11
Figure 7	Mounted support rod .....	12
Figure 8	Mounting the sample holder .....	12
Figure 9	Mounting the needles .....	14
Figure 10	Mounting the heating tubing (rear view) .....	15
Figure 11	Connecting the heating tubing .....	16
Figure 12	Mounting the dust filter .....	16
Figure 13	Preparing the drying flasks .....	17
Figure 14	Mounting the tubings .....	18
Figure 15	External gas connector .....	19
Figure 16	Coulometric KF titration cell .....	20
Figure 17	Volumetric KF titration cell .....	21
Figure 18	Power switch .....	22
Figure 19	Keypad of the operating unit .....	22
Figure 20	Main dialog .....	23
Figure 21	Insert the sample vessel into the oven .....	30
Figure 22	Move the guide head down .....	30
Figure 23	Insert the needle .....	31





# 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 holding times.

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 Instrument 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 instrument 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 instrument into operation. The documentation contains information and warnings which the user must follow in order to ensure safe operation of the instrument.

### 1.2.1 Symbols and conventions

The following symbols and formatting may appear in this documentation:

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



## 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.



### CAUTION

This symbol draws attention to possible damage to instruments or instrument parts.



## NOTE

This symbol highlights additional information and tips.

### 1.3 Safety instructions

### 1.3.1 General notes on safety



## WARNING

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

This instrument left the factory in a flawless state in terms of technical safety. To maintain this state and ensure non-hazardous operation of the instrument, the following instructions must be observed carefully.

### 1.3.2 Electrical safety

The electrical safety when working with the instrument is ensured as part of the international standard IEC 61010.



## WARNING

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



## WARNING

Never open the housing of the instrument. The instrument could be damaged by this. There is also a risk of serious 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 instrument.

Only operate this instrument with a supply voltage specified for it (see rear panel of the instrument).

### Protection against electrostatic charges



## WARNING

Electronic components are sensitive to electrostatic charges and can be destroyed by discharges.

Do not fail to pull the power cord out of the power socket before you set up or disconnect electrical plug connections at the rear of the instrument.

### 1.3.3 Personnel safety



## WARNING

Wear protective glasses and working clothes suitable for laboratory work while operating the 860 KF Thermoprep.



## WARNING

The **oven and the sample vessels** can reach temperatures of up to **250 °C!** Avoid direct skin contact. Use the provided septum crimping tongs or another suitable holding device to insert the sample vessels into or remove them from the oven. Wear heat-insulating gloves, if necessary.

**WARNING**

The needles pose a **considerable risk of injury**. When inserting the needle into the sample vessel, hold the handle of the needle adapter with both hands. Ensure that no other person reaches into the danger zone during this procedure.

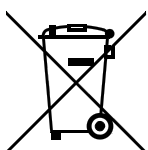
### 1.3.4 Flammable samples and solvents

**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.

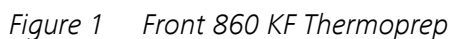
### 1.3.5 Recycling and disposal



This product is covered by European Directive 2012/19/EU, WEEE – Waste Electrical and Electronic Equipment.

The correct disposal of your old instrument will help to prevent negative effects on the environment and public health.

More details about the disposal of your old instrument can be obtained from your local authorities, from waste disposal companies or from your local dealer.



6 ■■■■■■



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





## Accessories

- 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



Do not use a not permitted power cord!

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

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

Proceed as follows:

- 

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.



## NOTICE

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).

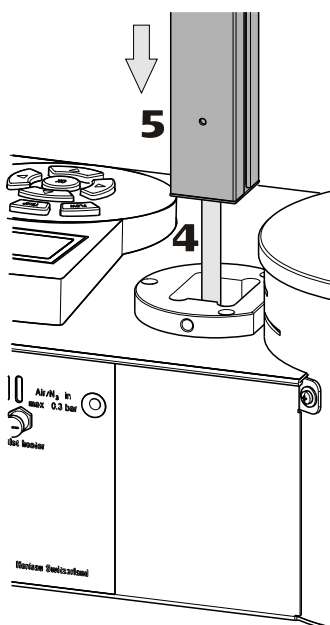
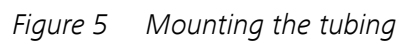


Figure 4 Mounting the support rod

- 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.



- 

- 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.



- ### 3.4 Mounting the sample holder



12 ■■■■■■

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 not exhibit dimensions outside of the following limit values:

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

## 3.5 Mounting the needles

The length of the needle holder defines how deep the needle for the gas inlet enters 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 the heated sample may block the needle, use the **6.2049.050 needle holder** with a length of 73 mm. In this case, the needle for the gas inlet enters the sample vessel slightly more than the outlet needle and it does not have any contact with the sample. The 6.2049.050 needle holder can be ordered at Metrohm if required.

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



- 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.



## NOTICE

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.

### 3.6 Mounting the heating tubing

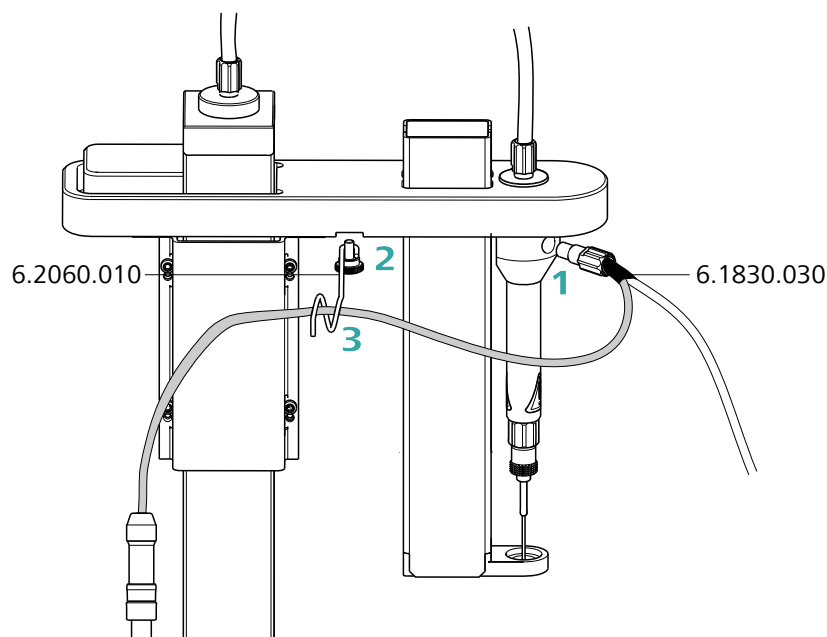


Figure 10 Mounting the heating tubing (rear view)

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.



#### NOTICE

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.

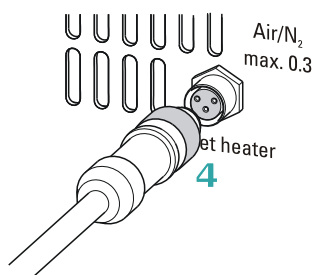


Figure 11 Connecting the heating tubing

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.



## NOTICE

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 removed from the sample and transferred to a KF titration cell with a carrier gas.

### 3.7 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..

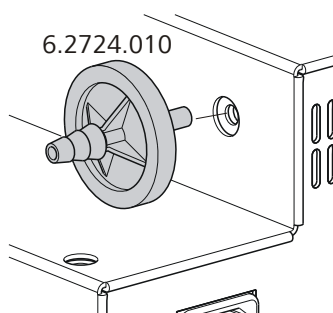


Figure 12 Mounting the dust filter

The dust filter should be replaced once a year.



### 3.8 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.

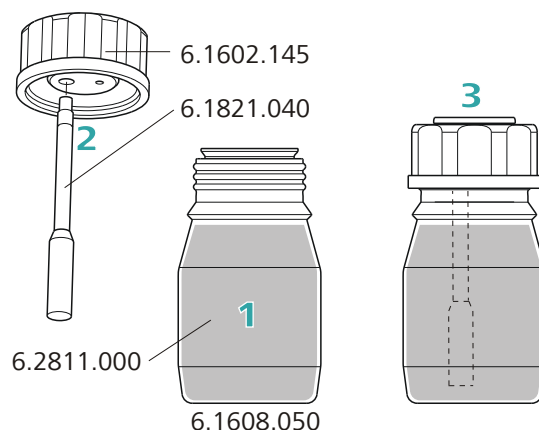


Figure 13 Preparing the drying flasks

Prepare the two drying flasks as follows:

- 1** Fill both **6.1608.050 drying flasks** with **6.2811.000 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.



#### NOTICE

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.

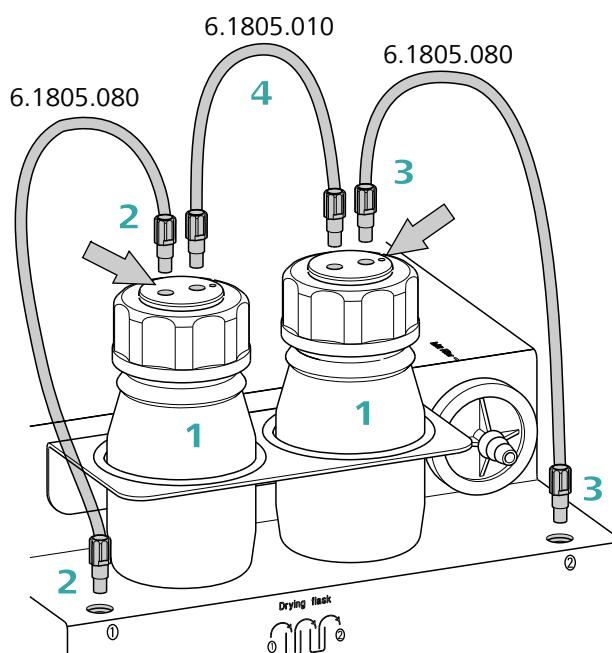


Figure 14 Mounting the tubings

Mount the FEP tubings as follows:

- 1 Insert the two drying flasks that have been prepared into the holders, see illustration.
- 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.



## NOTICE

Tighten the screw connections well by hand.

### 3.9 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.

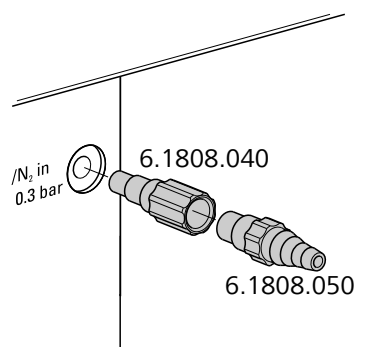


Figure 15 External gas connector



#### WARNING

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

### 3.10 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.

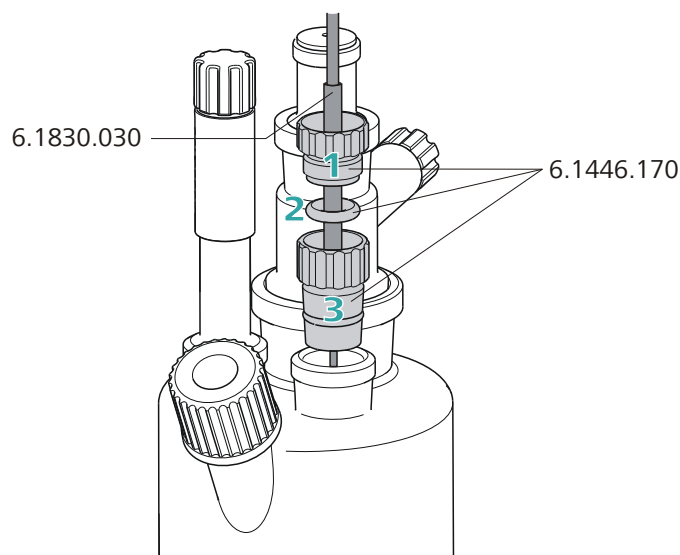


Figure 16 Coulometric KF titration cell

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.



**1** Guide the upper part of the septum stopper over the tip of the **6.1830.030 heating tube** as shown in the illustration.

- 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.



- [▲]

[▼]

da



2

1

## 2

## Icon

## Icon

### 4.3.3 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. The temperature control might be defective.</i>	Switch off the instrument immediately and let the oven cool down.  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.

## 4.4 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.



#### 4.4.1 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.

#### 4.4.2 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. 60 °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.

## 4.5 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]**.



## NOTICE

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

**Temp. 1**

Default value for temperature 1.

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

## Temp. 2

Default value for temperature 2.

## Gas flow

**Gas type**

**other**

**Meas. factor**

### Flow source

**pump**

Built-in air pump.

**valve**

Inlet valve for inert gas.

## 4.6 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.



#### NOTICE

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 - 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.

## 5 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.

## 5.1 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.

### 5.1.1 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.

## Conditioning the working medium

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



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

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

- 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]**.

- 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.



■■■■■■■ 29

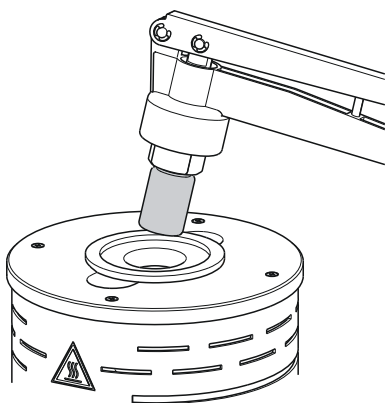


Figure 21 Insert the sample vessel into the oven

## Inserting the needle

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

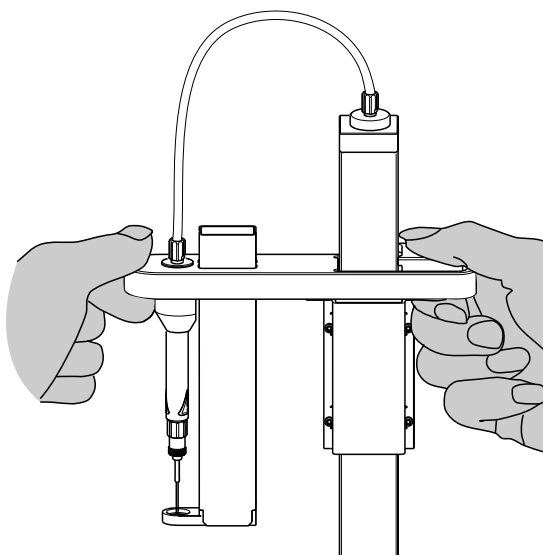


Figure 22 Move the guide head down

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

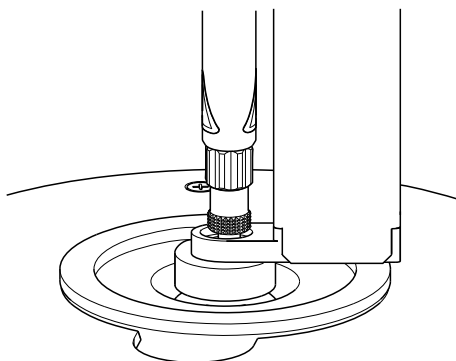


Figure 23 Insert the needle

### 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.



## NOTICE

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.



## NOTICE

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.

## 5.2 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  $\mu\text{g}/\text{min}$  (or  $\mu\text{L}/\text{min}$ ) 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 30 ff.*
- 3 Remove the conditioning vessel from the oven with the crimping tongs.





**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.

### 5.3 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 30 ff.*
- 3 Remove the blank from the oven with the crimping tongs.



## NOTICE

**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.

## 6.1 General

Severe contamination can also have an influence on the measured results. Regular cleaning of exposed parts can prevent this to a large extent.

## 6.2 Care

- Replace exhausted molecular sieve promptly. You should replace the molecular sieve as soon as increased drift values appear in the Karl Fischer cell.
- Check all tubing connections regularly for leaks.
- Rinse the tubing connections from time to time. Carefully dry the tubing afterward. The tubing must be replaced after prolonged usage.
- Replace the dust filter once a year.
- Clean the sample holder regularly.



Clean the oven only with the instrument switched off and while it is cold.

## 7 Troubleshooting

## 7.1 Problems and their solutions

Problem	Cause	Remedy
The drift is very high during conditioning.	<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.
The titration time is too long.	<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.
The results are spread widely.	<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.
Selected gas flow is not achieved.	<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.

## 8 Appendix

### 8.1 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.

## 8.2 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

## 9 Technical specifications

### 9.1 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

### 9.2 Gas flow

<i>Flow range</i>	10 - 150 mL/min Under normal conditions, calibrated with air at approx. +25 °C and 1,013 mbar. Deviations of gas amount per time unit of ±20% are possible.
-------------------	---

### 9.3 Outlet heater

<i>Socket connection</i>	$U = 16 \pm 1 \text{ V}$ $I \leq 0.8 \text{ A}$
<i>Typical tubing temperature</i>	approx. 50 °C

## 9.4 Power connection

<i>Potential</i>	100 - 120 / 220 - 240 V
<i>Frequency</i>	50 / 60 Hz
<i>Power consumption</i>	200 W
<i>Fuse</i>	2.0 ATH

## 9.5 Ambient temperature

<i>Nominal function range</i>	5 - 45 °C Humidity < 80%
<i>Storage</i>	-20 - 70 °C
<i>Transport</i>	-40 - 70 °C

## 9.6 Reference conditions

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

## 9.7 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)



## 10 Accessories

Up-to-date information on the scope of delivery and optional accessories for your product can be found on the Internet. You can download this information using the article number as follows:

## Downloading the accessories list

- 1 Enter <https://www.metrohm.com/> into your Internet browser.
- 2 Enter the article number (e.g. **860**) into the search field.  
The search result is displayed.
- 3 Click on the product.  
Detailed information regarding the product is shown on various tabs.
- 4 On the **Included parts** tab, click on **Download the PDF**.  
The PDF file with the accessories data is created.



## NOTICE

Once you have received your new product, we recommend downloading the accessories list from the Internet, printing it out and keeping it together with the manual for reference purposes.

---

6.2627.000 oven insert ..... 13

Arrow button .....	22
Arrow key .....	24, 25
Assembling	
Drying flasks .....	17

Basic settings .....	27
Blank value .....	32
Brightness .....	23

Cable holder .....	7, 15
Carrier gas .....	37
Colon .....	24
Conditioning .....	28
Conditioning vessel .....	29
Connect	
Power grid .....	8
Connector	
Air/nitrogen .....	7, 19
Outlet heating .....	7
Crimping tongs .....	29

Display .....	23
Drying flask .....	6, 17, 18
Dust filter .....	7, 16

Electrostatic charge .....	4
Error message .....	24
External gas connector .....	19
Extraction time .....	38

Fan .....	7
Fixing lever .....	6, 30
Flow rate .....	23
Flow rate error .....	17
Flow source .....	26

Gas flow .....	23, 26, 37
Gas inlet .....	6
Gas outlet .....	6
Gas supply .....	26
Gas type .....	26
Guidance rod .....	6
Guide head .....	6, 30

Heating tube .....	19
Heating tubing .....	15

Icon .....	23
Injection needle .....	14
Inlet	
Air pump .....	7
Inlet filter .....	16

Keys .....	22
KF titration cell .....	19, 28
Coulometric .....	19
Volumetric .....	20

---

Lock keys ..... 27

Main dialog .....	22
Maintenance .....	35
Molecular sieve .....	17
Mount	
Dust filter .....	16
Heating tubing .....	15
Support rod .....	9
Tubings .....	18

## Mounting

KF titration cell .....	19
Needle .....	13
Sample holder .....	12

Needle .....	13
Needle holder .....	14
Numerical input .....	25

Outlet heater .....	15
outlet needle .....	14
Oven module .....	6
Oven temperature .....	23, 25

Parameters .....	25
Personnel safety .....	4
Power connection .....	8, 9

Safety instructions .....	3
Sample determination .....	33
Sample holder .....	6, 12
Sample vessel .....	6
Selection .....	25
Selection list .....	25
Serial number .....	7
Service .....	3
Setup .....	27
Signal drift .....	32
Solvents .....	5
Status .....	23
Supply voltage .....	4
Support rod .....	9

Temperature .....	23, 25, 37
Temperature correction .....	27
Tubings .....	18
Type plate .....	7

---

Valve ..... 26