860 KF Thermoprep

Manual

8.860.8003EN

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Although all the information given in this documentation has been checked with great care, errors cannot be entirely excluded. Should you notice any mistakes please send us your comments using the address given above.

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1 Introduction

1.1 Instrument description

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

One of its decisive advantages is the reduction of sample preparation to a minimum. Thanks to the use of hermetically sealed sample vessels ("headspace vials"), the filling of the samples can be accomplished directly on-site. The PTFE-coated septa guarantee a constant, non-falsified 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 for the purpose of generating the gas flow. An inlet valve is available for nitrogen or other inert gases. The determination of the moisture can be accomplished in the measuring cell either coulometrically or volumetrically in accordance with Karl Fischer.

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 heating**
  Heating tube for preventing the condensation of moisture.
- **Stand 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 moisture determination according to Karl Fischer (coulometric or volumetric). The 860 KF Thermoprep enables the application of thermal gas extraction technology.

The present instrument is suitable for processing chemicals and flammable samples. The usage of the 860 KF Thermoprep therefore requires that the user has basic knowledge and experience in the handling of poisonous and caustic substances. Knowledge with respect to the application of the 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 have to be followed by the user in order to ensure safe operation of the instrument.

1.2.1 Symbols and conventions

The following symbols and styles are used in this documentation:

*(5-**12**)*	**Cross-reference to figure legend** The first number refers to the figure number, the second to the instrument part in the figure.
1	**Instruction step** Carry out these steps in the sequence shown.
Method	**Dialog text**, **parameter** in the software
File ▸ New	Menu or menu item
[Next]	**Button** or **key**
	**Warning** This symbol draws attention to a possible life hazard or risk of injury.
	**Warning** 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.
	**Caution** This symbol draws attention to a possible damage of instruments or instrument parts.
	**Note** This symbol marks additional information and tips.

1.3 Safety instructions

1.3.1 General notes on safety

Warning

This instrument may only be operated in accordance with the specifications in this documentation.

This instrument has 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.

Mains voltage

Warning

An incorrect mains voltage can damage the instrument.

Only operate this instrument with a mains 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.

Always pull the mains cable out of the mains connection socket before connecting or disconnecting electrical appliances on the rear panel of the instrument.

1.3.3 Personnel safety

Warning

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

Warning

The **oven and the sample vessels** can be at a temperature of up to **250 °C**! Avoid direct skin contact. Use the septum-sealing tongs supplied or another suitable holding device for inserting the sample vessels in the oven or removing them. Wear heat-insulated gloves if necessary.

Warning

There is a **considerable risk of injury** connected with the needles. Grasp the handle of the needle adapter with both hands when you guide the needle into the sample vessel. Take care to ensure that no other person reaches into the danger zone during this procedure.

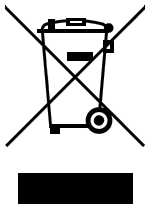
1.3.4 Flammable samples and solvents

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.
- Keep all sources of flame far from the workplace.
- Clean up spilled fluids and solids immediately.
- Follow the safety instructions of the chemical manufacturer.

1.3.5 Recycling and disposal



This product is covered by European Directive 2002/96/EC, WEEE – Waste from Electrical and Electronic Equipment.

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

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

2 Overview of the instrument

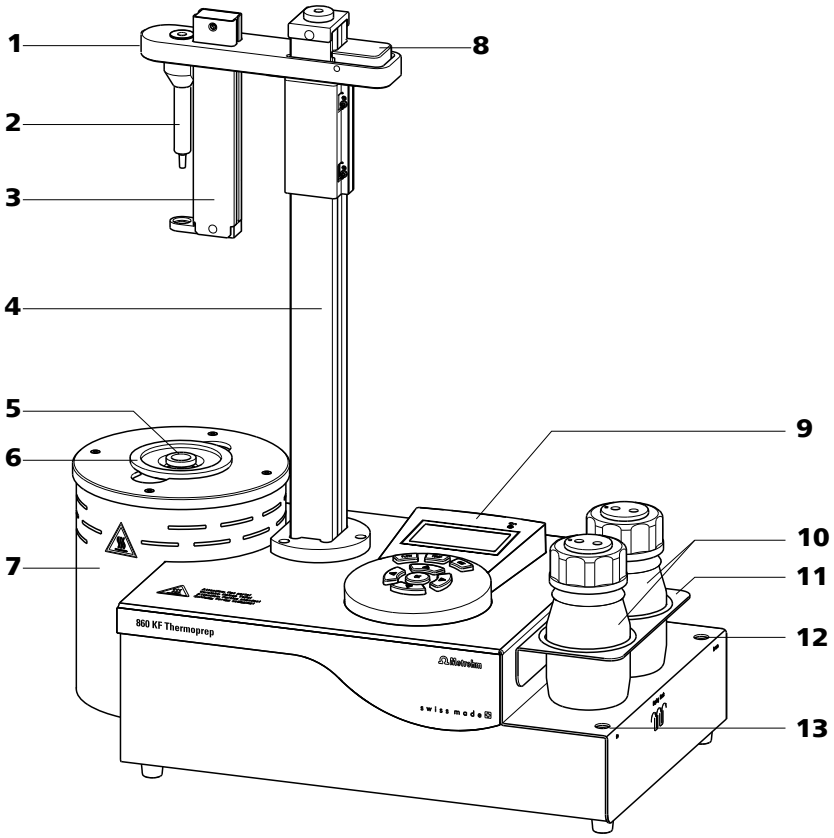


Figure 1 Front 860 KF Thermoprep

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

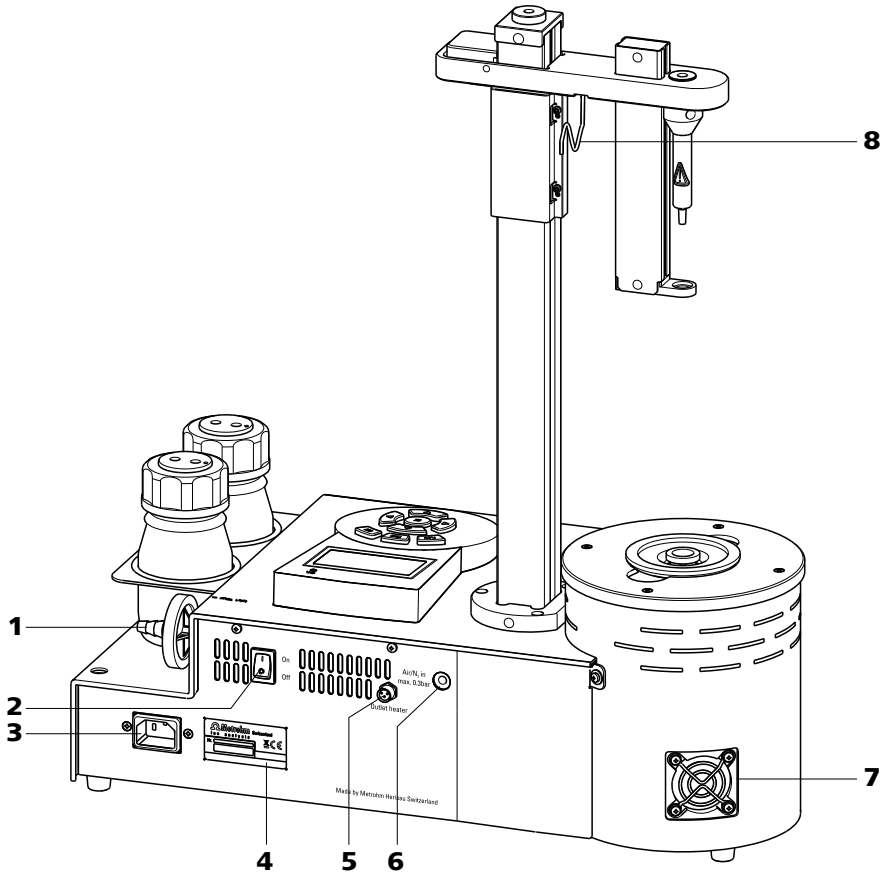


Figure 2 Rear 860 KF Thermoprep

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

3 Installation

3.1 Setting up the instrument

3.1.1 Packaging

The instrument is supplied in highly protective special packaging together with the separately packed accessories. Keep this packaging, as only this ensures safe transportation of the instrument.

3.1.2 Checks

Immediately after receipt, check whether the shipment has arrived complete and without damage by comparing it with the delivery note.

3.1.3 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, free of vibrations, protected from corrosive atmosphere, and contamination by chemicals.

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

3.2 Connecting the mains cable

Warning

This instrument must not be operated except with the mains voltage specified for it (see rear of the instrument).

Protect the connection sockets against moisture.

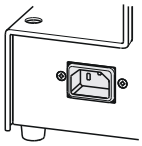


Figure 3 Connecting the mains cable

3.3 Mounting the guidance rod

The guidance 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 guidance rod a bit downwards by pressing the fixing lever.

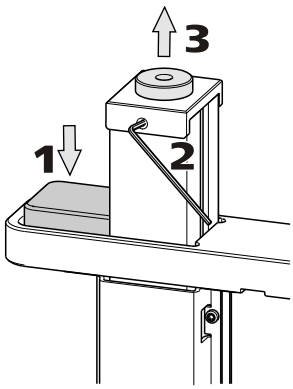


Figure 4 Loosen the adapter

2 Loosen the adapter on the head of the guidance rod with a hexagon key.

3 Remove the adapter.

4 Insert the tubing jutting out of the housing from below into the guidance rod.

Note

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

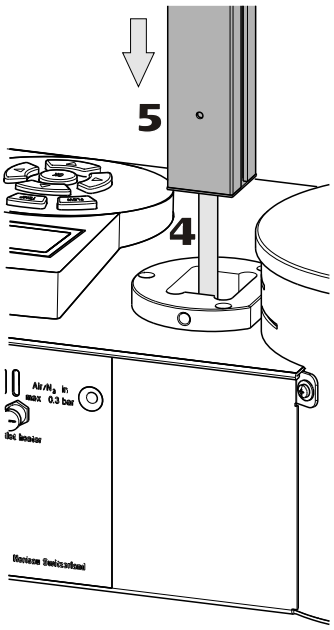


Figure 5 Mounting the guidance rod

5 Insert the guidance rod into the opening intended for this purpose from above and push it downwards until it hits the stop. Observe the orientation of the guidance rod.

6 Pull out the tubing inside the guidance rod as far as possible. Eventually use a piece of sand paper in order to be able to better grasp the tubing.

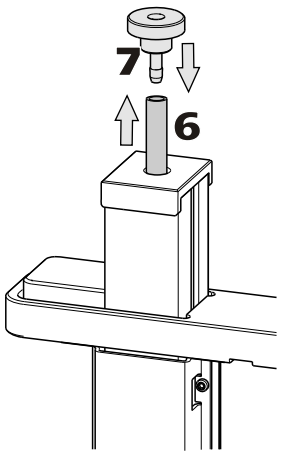


Figure 6 Mounting tubing

7 Fasten the tubing on the olive of the 4.860.4260 adapter.

8 Pull out the guidance rod as far as possible in order that the adapter rests close on the guidance rod.

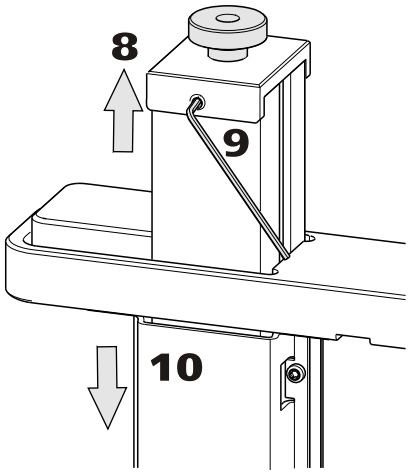


Figure 7 Fix the adapter

9 Fix the adapter in the guidance rod. Fasten the screw in the guidance rod with the aid of a hexagon key.

10 Carefully push the guidance rod downwards.

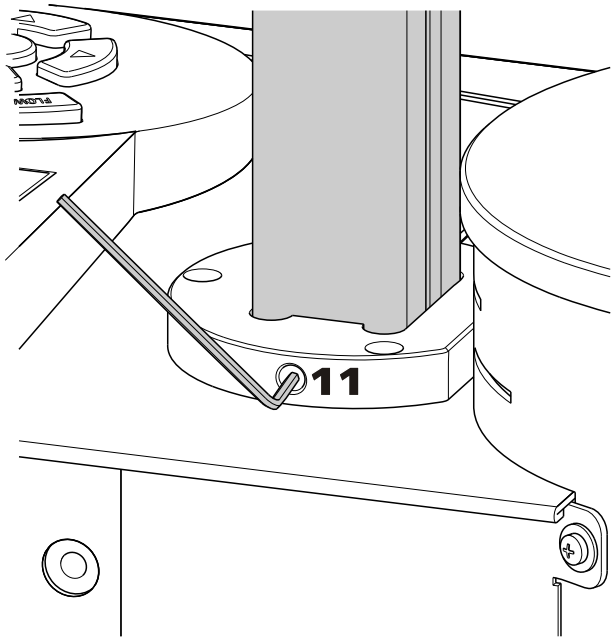


Figure 8 Guidance rod mounted

11 Fix the guidance rod with the V.0434.008 hexagon screw according to the drawing. The screw and the associated hexagon key are packaged separately with the instrument.

3.4 Mounting the sample insert

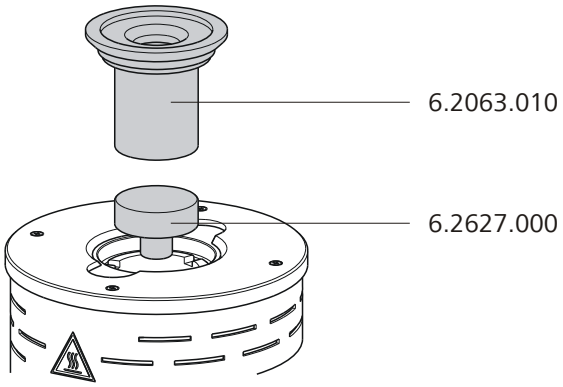


Figure 9 Mounting the sample insert

Note

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

The dimensions of the supplied sample insert are optimized for the usage of 6 mL sample vials 6.2419.007 from 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 insert** into the oven from above.

If vessels with other dimensions are to be used, then individually modified sample inserts can be ordered. The precise vessel dimensions (including tolerances) will be required when ordering. 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 deeply the injection needle penetrates the sample vessel. The **6.2049.040 needle holder**, which is 58 mm in length, ensures that the needle penetrates the liquid or powdery sample. The carrier gas can flow through the sample and effect an efficient expulsion of the moisture it contains.

If there is a danger that the heated sample could clog the needle, then use the **6.2049.050 needle holder** with 73 mm length. In this case the injection needle penetrates the sample vessel only slightly deeper than the outlet needle and has no contact with the sample itself. 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.

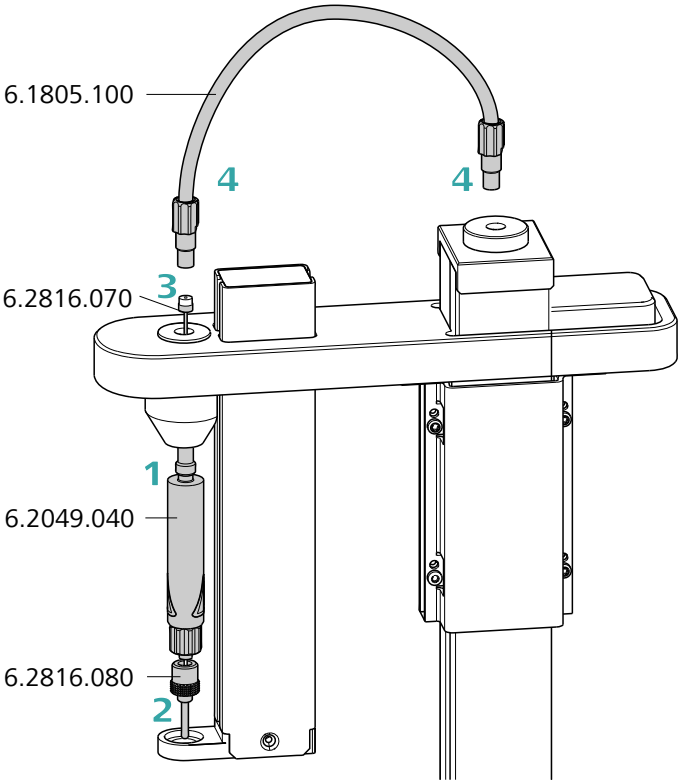


Figure 10 Mounting the needles

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 injection needle** into the opening of the distributor from above and allow it to drop down.

Note

Take care to ensure that the white PTFE seal is positioned securely on the needle.

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

3.6 Mounting the heating tubing

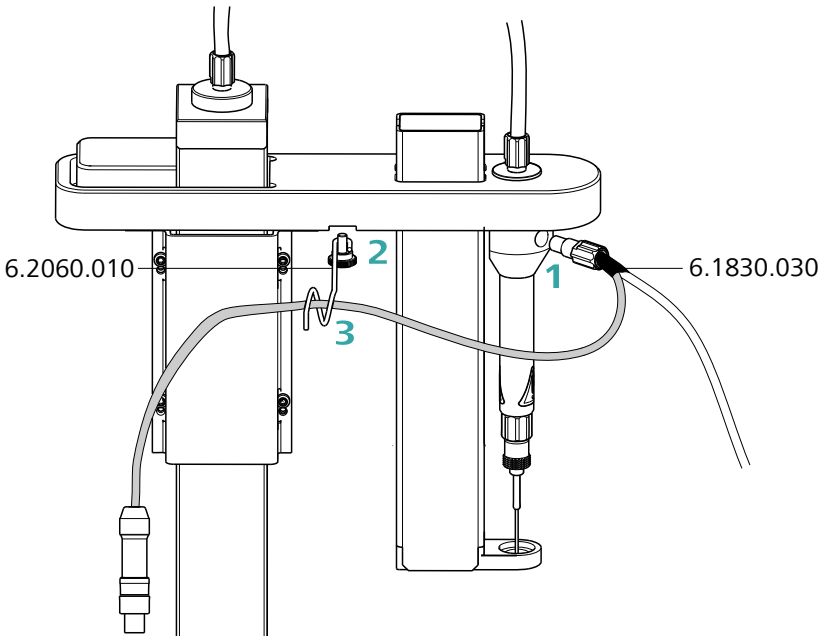


Figure 11 Mounting the heating tubing (view from rear)

Proceed as follows:

1 Screw the M6 connector of the **6.1830.030 heating tubing** into the side 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 panel of the instrument.

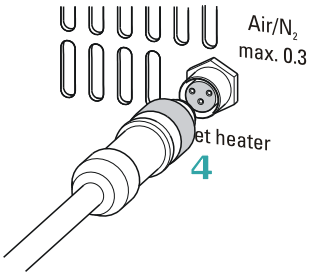


Figure 12 Connecting the heating tubing

Rotate the plug in such a way that the three contact pins match the alignment of the corresponding openings on the socket. Press the plug against the socket and rotate the front knurled screw in clockwise direction.

Note

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

3.7 Mounting the dust filter

The built-in air pump must be protected against dust. A **6.2724.010 dust filter** must be placed on the air inlet (**Inlet filter**) on the right-hand side of the housing for this reason.

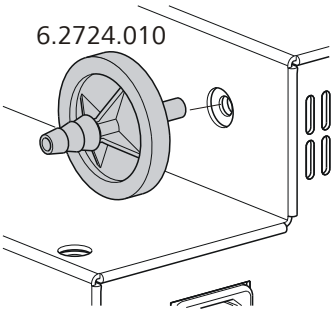


Figure 13 Mounting the dust filter

The dust filter should be replaced once a year.

3.8 Assembling the drying flask

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

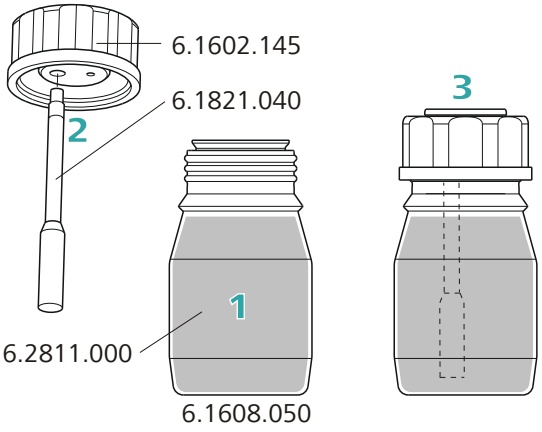


Figure 14 Preparing the drying flasks

Prepare both 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 covers** from below. Tighten the filter tubes well by hand.

3 Screw the two drying flask covers with the filter tubes onto the drying flasks. Tighten the covers well by hand.

Note

If drying flask covers or filter tubes are not sufficiently tightly screwed on, then this will prevent a precise, regular flow of gas. The error message "Flow rate error" will appear as a rule when there are leaks in the threaded connections.

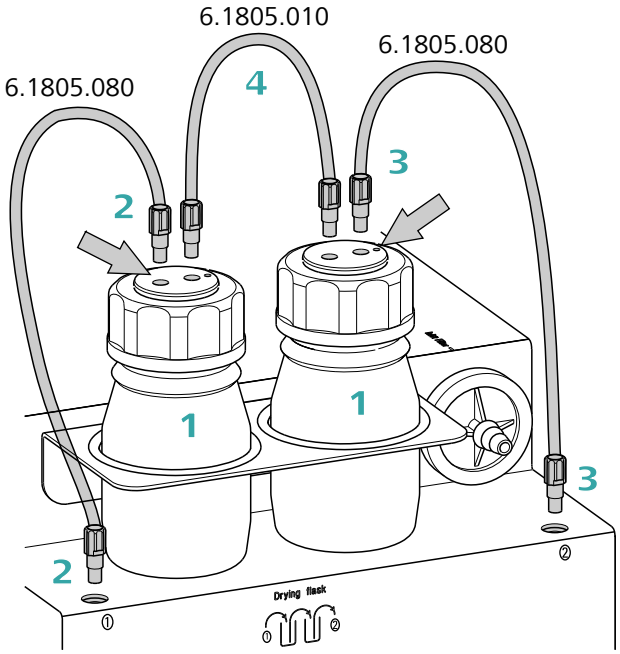


Figure 15 Mounting the tubings

Mount the FEP tubings as follows:

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

Note

Tighten the screw connections well by hand.

3.9 Air/nitrogen connector

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

A tube with M6 thread can be connected directly to the connector **Air/N_2 in**. Enclosed with the instrument is the **6.1808.040 M6/M8 tubing adapter** for tubing with an M8 thread. The **6.1808.050 M8/tubing olive** can also be put in place in order to connect a simple tubing.

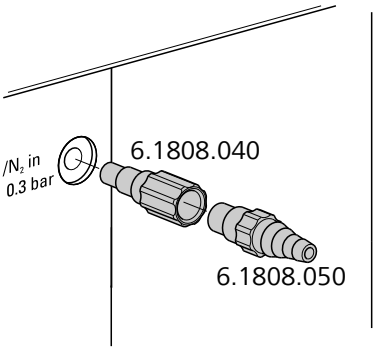


Figure 16 External gas supply connection

Warning

If gas is supplied from a pressure line or a pressure vessel, then it is imperative that a pressure reduction valve be placed upstream. The gas pressure may not exceed a maximum overpressure level of 0.3 bar.

3.10 Inserting the heating tube into the KF titration cell

The 860 KF Thermoprep can be used for either coulometric or volumetric water determinations. 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.

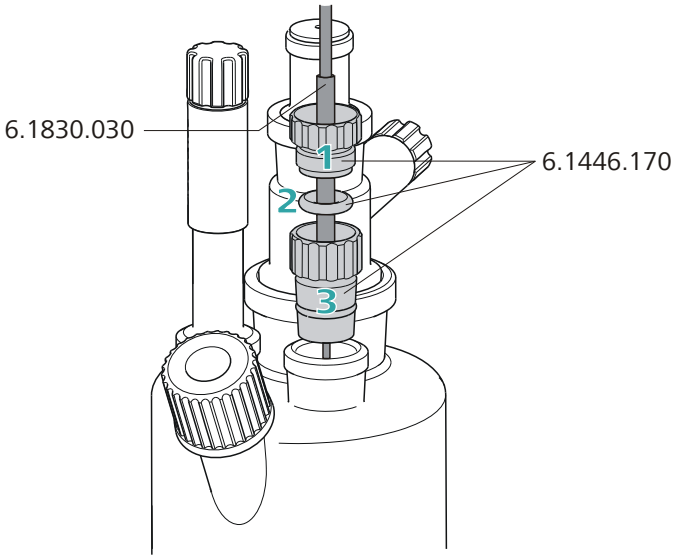


Figure 17 Coulometric KF titration cell

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.

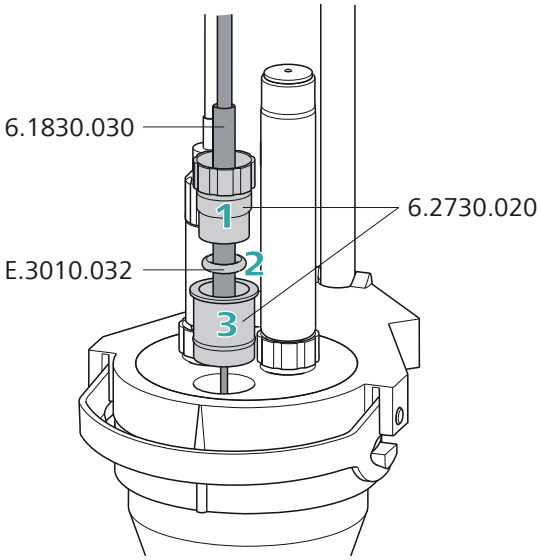


Figure 18 Volumetric KF titration cell

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.

4 Operation

4.1 Switching the instrument on and off

The rocker switch for switching on and off is located at the rear of the instrument.

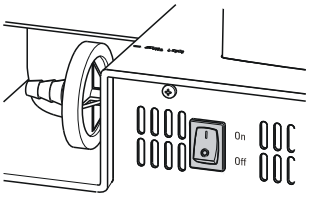


Figure 19 Mains switch

The oven switches on automatically after the instrument is switched on and is heated up to the temperature that was most recently set. The outlet heating of the heating tube is also switched on at the same time.

4.2 The keypad

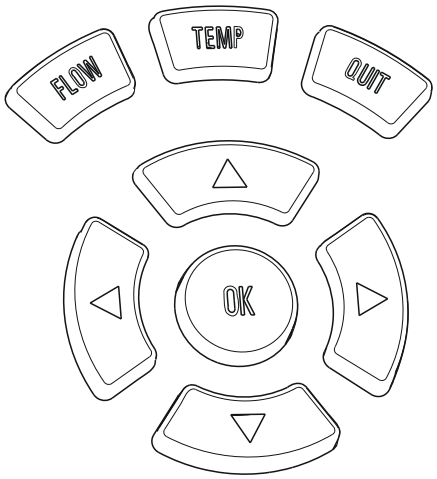


Figure 20 Keypad and operating unit

The key functions in the main dialog

[FLOW]	Switches the gas flow on and off.
[TEMP]	Switches between the two target temperatures.
[QUIT]	No function.
[OK]	Opens the parameters menu.

[◂]	Reduces gas flow by 1 mL/min during ongoing operation. Keep the key pressed down briefly for this purpose.
[▸]	Increases gas flow by 1 mL/min during ongoing operation. Keep the key pressed down briefly for this purpose.
[▴]	Reduces the brightness of the display.
[▾]	Increases the brightness of the display.

4.3 The display

4.3.1 Display elements

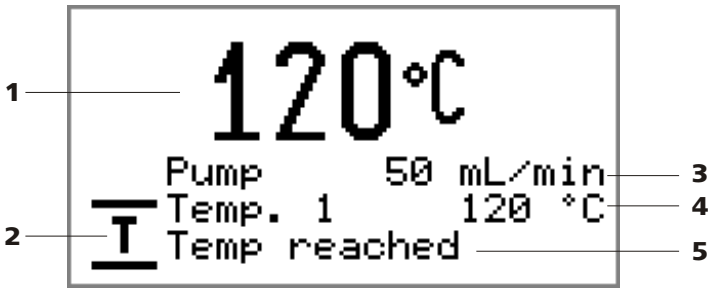


Figure 21 Main dialog

1 Temperature display Shows the measured oven temperature.	**2 Symbol of the status display**
3 Gas flow display Displays the set gas supply and the flow rate.	**4 Target temperature** Displays the set specification (Temp. 1 or Temp. 2).
5 Status display Shows status and error messages.	

4.3.2 Status display

Table 1 Symbols of the status display

Symbol	Significance
	The oven is being heated.
	The oven is being cooled.
	The pre-selected temperature was reached.

Symbol	Significance
	An error has occurred. The type of the error will be identified in the status display.

4.3.3 Error messages

Table 2 Error messages

Error message	Cause	Remedy
Flow rate error	*The tubing system is leaking or the flow rate is set too high.*	Screw all of the tubing connections and the screw connectors of the drying flasks tightly. Check the functioning of the pump.
	In cases of external gas supply, the pressure is either too low or too high.	Adjust the pressure of the external gas to 0.1…0.3 bar.
	The flow sensor is defective.	Inform a service technician.
Max. temp. reached	*The oven temperature is greater than 265 °C. There is presumably a defect in the temperature control.*	Switch off the instrument without delay and allow the oven to cool down. Inform a service technician of the problem if the error appears again after switching back on.
Adj. data missing	*The adjustment data of the instrument are not correct.*	The instrument must be readjusted. Inform a service technician.

4.4 Modifying numerical values and settings

The instrument settings can be modified in two different ways:

- Selection of specified values from a list. Parameters with list boxes are indicated with a colon (**:**).
- Modifying numerical values.

The different parameters can be selected with the arrow keys **[▼]** and **[▲]**. The current line is displayed in inverted fashion.

4.4.1 Selecting from a selection list

To modify parameters with list boxes (e.g. **Flow source:**), use the arrow keys **[►]** or **[◄]**. The respective next value in the list box will be displayed.

Confirm a modified value with the **[OK]** key.

4.4.2 Modifying numerical values

Modify parameters with numerical values using the arrow keys:

1 Use the arrow keys **[►]** or **[◄]** to select one of the digits of the numerical value.

The selected digit is displayed black on white. The rest of the line remains inverted.

2 Use the arrow keys **[▲]** or **[▼]** to modify the digits.

3 Select and modify the next digit, 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 adjusted in a menu that is opened by pressing the **[OK]** key. Exit the Parameters menu with **[QUIT]**.

Note

Modified values are applied after confirmation with the **[OK]** key. A modification is discarded with **[QUIT]**.

Temp. 1

Set value for temperature 1.

Range	**50 ... 250 °C** (Increment: **1**)
Default value	**100 °C**

Temp. 2

Set value for temperature 2.

Range	**50 ... 250 °C** (Increment: **1**)
Default value	**100 °C**

Gas flow

Set value for the gas flow.

Range	**10 ... 150 mL/min** (Increment: **1**)
Default value	**50 mL/min**

Gas type

Selection of the gas.

Selection	**air** | **nitrogen** | **other**
Default value	**air**

other
If **other** is selected, then a measurement factor for the gas that is used must be specified. The measuring factor is used for the correct measurement of the flow rate.

Meas. factor

The measuring factor for measuring the flow rate of the gas. This setting is only possible if **other** is selected for the gas. The corresponding measuring factor is automatically applied for air and nitrogen.

Only when 'gas type' = 'other'.

Range	**0.001 ... 2.000** (Increment: **0.001**)
Default value	**1.000**

Table 3 Measuring factors of different gases

Gas type	Measuring factor
Argon	0.950
Helium	0.300
Oxygen	1.000
Air	1.000
Nitrogen	1.000

Flow source

Selecting the flow source.

Selection	**pump** | **valve**
Default value	**pump**

pump
Built-in air pump.

valve
Inlet valve for inert gas.

4.6 Configuration

[TEMP]

The basic settings for the instrument can be made in the Setup menu. You can call it up during switch on by holding down the **[TEMP]** key. Press the **[QUIT]** key to exit the menu.

Note

The Setup menu can also be called up when the keylock is switched on.

Lock keys

Keylock. The **[OK]**,**[◂]** and **[▸]** keys can be locked. You can thus prevent changes from being made in the Parameters menu or of the gas flow rate during a determination. If the function is switched on (**on**), then the only switching that can be done is between Temperature 1 and Temperature 2,

in addition to switching the gas flow on and off. Furthermore it is still possible to modify the brightness of the display.

Selection	**off** | **on**
Default value	**off**

Temp.corr

Temperature correction. The target value for the temperature control of the oven can be influenced with this correction value. A temperature differential between the oven module and the sample in the vessel can thus be brought into line as needed.

Range	**-10 ... 10 °C** (Increment: **1**)
Default value	**0 °C**

The determination of the necessary temperature correction can be accomplished with a special oven insert and should be performed by a service technician. 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.

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.

5.1.2 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.

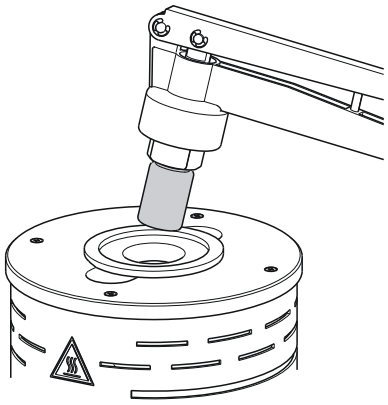


Figure 22 Insert the sample vessel into the oven

Inserting the needle

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

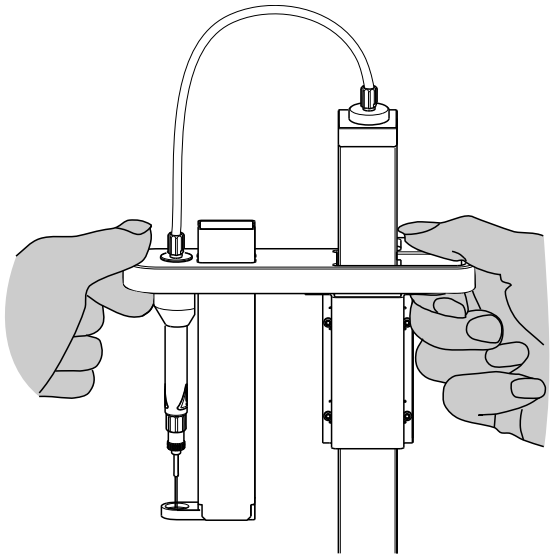


Figure 23 Move the guide head down

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

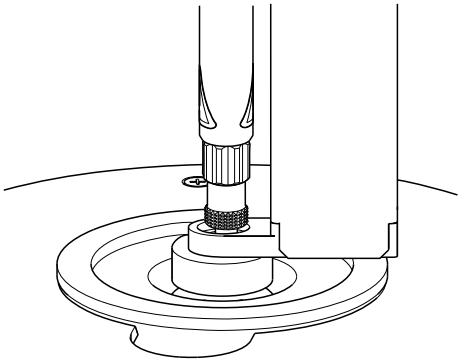


Figure 24 Insert the needle

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.

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 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 31* 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.

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 31* 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.

6 Handling and maintenance

6.1 General

The 860 KF Thermoprep requires appropriate care. Excess contamination of the instrument may result in functional disruptions and a reduction in the service life of the sturdy mechanics and electronics of the instrument.

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

Spilled chemicals and solvents must be removed immediately. In particular, the mains plug should be protected from contamination.

6.2 Care

- Replace exhausted molecular sieves 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.
- Flush out 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 insert regularly.

Warning

Clean the oven only when it is switched off and cold.

6.3 Quality Management and validation with Metrohm

Quality Management

Metrohm offers you comprehensive support in implementing quality management measures for instruments and software. You can find information on this in the brochure available from your local Metrohm agent **«Quality Management with Metrohm»**.

Validation

Please contact your local Metrohm agent for support in validating instruments and software. Here you can also obtain validation documentation to provide help for carrying out the **Installation Qualification** (IQ = Installation Qualification) and the **Operational** (OQ = Operational Qualification). IQ and OQ are also offered as a service by the Metrohm agents. In

addition, various application bulletins are also available on the subject, which also contain **Standard Operating Procedures** (SOP = Standard Operating Procedure) for testing analytical measuring instruments for reproducibility and correctness.

Maintenance

Electronic and mechanical functional groups in Metrohm instruments can and should be checked as part of regular maintenance by specialist personnel from Metrohm. Please ask your local Metrohm agent regarding the precise terms and conditions involved in concluding a corresponding maintenance agreement.

Note

You can find information on the subjects of quality management, validation and maintenance as well as an overview of the documents currently available at *www.metrohm.com* under **Support/Quality Management**.

7 Troubleshooting

7.1 Problems and their solutions

Problem	Cause	Remedy
The drift is very high during conditioning.	*Molecular sieve of drying flasks and/or the titration cell exhausted.*	Replace molecular sieve.
	The titration cell is leaking	Check seals. If necessary, replace.
The titration time is too long.	*The sample is non-homogenous*	Reduce the size of the sample before weighing in.
	Not the entirety of the moisture expelled.	Select more stringent switch-off criteria on the KF titrator: lower stop drift, higher switch-off delay time.
	Tubing connections leaking	Inspect tubing and replace as necessary.
The results are spread widely.	*Molecular sieve of drying flasks exhausted.*	Replace molecular sieve.
	Condensate in the heating tube.	▪ Dry the tubing. ▪ Reduce the gas flow. ▪ Perhaps lower the oven temperature.
	Gas flow too high.	Reduce the gas flow.
	The sample is non-homogenous	Reduce the size of the sample before weighing in.
Selected gas flow is not achieved.	*System is leaking*	Check all tubing connections and drying flasks for leaks.
	Pressure from external gas supply either too low or too high.	Adjust gas pressure (max. 0.3 bar).
	Pump defective	Contact a service technician.

8 Appendix

8.1 Practical instructions

Carrier gas selection

If the hot sample is sensitive to air or oxygen (decomposition) and releases substances that disrupt the KF reaction, then nitrogen (N_2) should be used as the carrier gas.

Temperature settings

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

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

Gas flow

If the needle is inserted into the sample vessel until it stops, then 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 dip into the sample, then a longer needle holder can be used, see the chapter "Optional accessories".

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 excessively large quantities of water are not released too quickly into the titration vessel. In addition, there is the danger of condensate formation in the heating tube. The working medium in the cell must be able to absorb the moisture that is expelled without delay. Normally, a flow rate of 40...60 mL/min is optimal.

Extraction time

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

Conditioning the system

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

8.2 Literature

- E. Scholz, *Hydranal@-Manual*, Riedel-de Haën, 1996
- P. Bruttel, R. Schlink, *Water determination by Karl Fischer Titration.* Monograph, Metrohm, 2006, Best. Nr. 8.026.5013

9 Technical data

9.1 Oven

Temperature range	50…250 °C
Accuracy	±3 °C
Correction range	-10…+10 °C
Heating cartridge performance	165 W typical Depending on mains voltage
Heating rate	Typically 15 °C/min (with 80…180 °C, 230 V) Dependent on temperature, mains voltage, sample amount and vessel dimensions
Cooling rate	Typical. 9 °C/min (at 180…80 °C) Dependent on temperature, sample amount and vessel dimensions

9.2 Gas flow

Flow range	10…150 mL/min Under normal conditions

9.3 Outlet heating

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

9.4 Mains connection

Voltage	100…120 / 220…240 V
Frequency	50 / 60 Hz
Power consumption	200 W
Fuse	2.0 ATH

9.5 Safety specifications

Design and testing	According to EN/IEC 61010-1: 2001, UL 61010-1: 2004, CSA-C22.2 No. 61010-1: 2004, EN/IEC 61010-2-010: 2003, protection class I
Safety instructions	This document contains safety instructions which have to be followed by the user in order to ensure safe operation of the instrument.

9.6 Electromagnetic compatibility (EMC)

Emission

Standards fulfilled:

- EN/IEC 61326-1: 2006
- EN/IEC 61000-6-3: 2004
- EN 55022 / CISPR 22: 2006
- EN/IEC 61000-3-2: 2006
- EN/IEC 61000-3-3: 2005

Immunity

Standards fulfilled:

- EN/IEC 61326-1: 2006
- EN/IEC 61000-6-2: 2005
- EN/IEC 61000-4-2: 2001
- EN/IEC 61000-4-3: 2002
- EN/IEC 61000-4-4: 2004
- EN/IEC 61000-4-5: 2001
- EN/IEC 61000-4-6: 2001
- EN/IEC 61000-4-11: 2004
- EN/IEC 61000-4-14: 2004
- EN/IEC 61000-4-28: 2004
- NAMUR: 2004

9.7 Ambient temperature

Nominal function range	5…45 °C Humidity < 80 %
Storage	–20…70 °C
Transport	–40…70 °C

9.8 Reference conditions

Ambient temperature	25 °C (±3 °C)
Relative humidity	≤60 %
Mains voltage	230 V

9.9 Dimensions

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

10 Conformity and warranty

10.1 Declaration of Conformity

This is to certify the conformity to the standard specifications for electrical appliances and accessories, as well as to the standard specifications for security and to system validation issued by the manufacturing company.

Name of commodity

860 Thermoprep

Temperature-controlled oven unit for the sample treatment in coulometric or volumetric water determinations.

This instrument has been built and has undergone final type testing according to the standards:

Electromagnetic compatibility

Emission:	EN/IEC 61326-1: 2006, EN/IEC 61000-6-3: 2004, EN 55022 / CISPR 22: 2006, EN/IEC 61000-3-2: 2006, EN/IEC 61000-3-3: 2005
Immunity:	EN/IEC 61326-1: 2006, EN/IEC 61000-6-2: 2005, EN/IEC 61000-4-2: 2001, EN/IEC 61000-4-3: 2002, EN/IEC 61000-4-4: 2004, EN/IEC 61000-4-5: 2001, EN/IEC 61000-4-6: 2001, EN/IEC 61000-4-11: 2004, EN/IEC 61000-4-14: 2004, EN/IEC 61000-4-28: 2004, NAMUR: 2004

Safety specifications

EN/IEC 61010-1: 2001, UL 61010-1: 2004,
CSA-C22.2 No. 61010-1: 2004, EN/IEC 61010-2-010: 2003, protection class I

CE

This instrument meets the requirements of the CE mark as contained in the EU directives 2006/95/EC (LVD), 2004/108/EC (EMC). It fulfils the following specifications:

EN 61326-1	Electrical equipment for measurement, control and laboratory use – EMC requirements
EN 61010-1	Safety requirements for electrical equipment for measurement, control and laboratory use

EN 61010-2-010	Particular requirements for laboratory equipment for the heating of materials

Manufacturer Metrohm Ltd., CH-9101 Herisau/Switzerland

Metrohm Ltd. is holder of the SQS-certificate ISO 9001:2000 Quality management system for development, production and sales of instruments and accessories for ion analysis.

Herisau, 27 November, 2007

D. Strohm

Vice President, Head of R&D

A. Dellenbach

Head of Quality Management

10.2 Warranty (guarantee)

Metrohm guarantees that the deliveries and services it provides are free from material, design or manufacturing errors. The warranty period is 36 months from the day of delivery; for day and night operation it is 18 months. The warranty remains valid on condition that the service is provided by an authorized Metrohm service organization.

Glass breakage is excluded from the warranty for electrodes and other glassware. The warranty for the accuracy corresponds to the technical specifications given in this manual. For components from third parties that make up a considerable part of our instrument, the manufacturer's warranty provisions apply. Warranty claims cannot be pursued if the Customer has not complied with the obligations to make payment on time.

During the warranty period Metrohm undertakes, at its own choice, to either repair at its own premises, free of charge, any instruments that can be shown to be faulty or to replace them. Transport costs are to the Customer's account.

Faults arising from circumstances that are not the responsibility of Metrohm, such as improper storage or improper use, etc. are expressly excluded from the warranty.

10.3 Quality Management Principles

Metrohm Ltd. holds the ISO 9001:2000 Certificate, registration number 10872-02, issued by SQS (Swiss Association for Quality and Management Systems). Internal and external audits are carried out periodically to assure that the standards defined by Metrohm's QM Manual are maintained.

The steps involved in the design, manufacture and servicing of instruments are fully documented and the resulting reports are archived for ten years. The development of software for PCs and instruments is also duly documented and the documents and source codes are archived. Both remain the possession of Metrohm. A non-disclosure agreement may be asked to be provided by those requiring access to them.

The implementation of the ISO 9001:2000 quality management system is described in Metrohm's QM Manual, which comprises detailed instructions on the following fields of activity:

Instrument development

The organization of the instrument design, its planning and the intermediate controls are fully documented and traceable. Laboratory testing accompanies all phases of instrument development.

Software development

Software development occurs in terms of the software life cycle. Tests are performed to detect programming errors and to assess the program's functionality in a laboratory environment.

Components

All components used in the Metrohm instruments have to satisfy the quality standards that are defined and implemented for our products. Suppliers of components are audited by Metrohm as the need arises.

Manufacture

The measures put into practice in the production of our instruments guarantee a constant quality standard. Production planning and manufacturing procedures, maintenance of production means and testing of components, intermediate and finished products are prescribed.

Customer support and service

Customer support involves all phases of instrument acquisition and use by the customer, i.e. consulting to define the adequate equipment for the analytical problem at hand, delivery of the equipment, user manuals, training, after-sales service and processing of customer complaints. The Metrohm service organization is equipped to support customers in implementing standards such as GLP, GMP, ISO 900X, in performing Opera-

tional Qualification and Performance Verification of the system components or in carrying out the System Validation for the quantitative determination of a substance in a given matrix.

11 Accessories

Note

Subject to change without notice.

11.1 Scope of delivery 2.860.0010

Qty.	Order no.	Description
1	1.860.0010	**860 KF Thermoprep** Instrument for the thermal extraction of humidity from solid and liquid samples.
1	6.1446.170	**Heating tube stopper** for coulometric KF vessels in connection with heatable tubing connection Material: PP Height (mm): 34 Outer diameter (mm): 13
1	6.1448.057	**Aluminum septum caps / 100 pieces** Including Al sealing. Material: Aluminum Material 2: Silicone Height (mm): 7.4 Outer diameter (mm): 20.7
2	6.1602.145	**Cap for drying bottle** Cap with GL45 thread for use with 6.1608.050

Qty.	Order no.	Description	
2	6.1608.050	**Drying bottle / 100 mL / GL 45**	
	Material:	Clear glass	
	Height (mm):	100	
	Outer diameter (mm):	56	
	Volume (mL):	100	
1	6.1805.010	**FEP tubing / M6 / 13 cm**	
	With light and kink protection		
	Material:	FEP	
	Inner diameter (mm):	2	
	Length (mm):	130	
2	6.1805.080	**FEP tubing / M6 / 25 cm**	
	With light and kink protection		
	Material:	FEP	
	Inner diameter (mm):	2	
	Length (mm):	250	

Qty.	Order no.	Description
1	6.1805.100	**FEP tubing / M6 / 40 cm**

With light and kink protection

Material:	FEP
Inner diameter (mm):	2
Length (mm):	400

Qty.	Order no.	Description
1	6.1808.040	**Adapter M6 outer / M8 inner**

Outer thread M6, inner thread M8.

Material:	PTCFE

Qty.	Order no.	Description
1	6.1808.050	**Connector tubing nozzle M8**

1 M8 outer thread and 1 tubing olive. E.g. for thermostat jacket of Exchange Units and stability measuring instruments.

Material:	PVDF
Length (mm):	31.5

Qty.	Order no.	Description
2	6.1821.040	**Filter tube** Filter tube for 6.1608.050 Drying bottle. For Rancimats and Karl Fischer Ovens. Length (mm): 112
1	6.1830.030	**Heatable outlet tubing** Heatable outlet tubing for 860 KF Thermoprep.
1	6.2048.030	**Guidance rod** Guidance rod for 860 KF Thermoprep.
1	6.2049.040	**Needle holder with Luer-lock for 860 KF Thermoprep** Length (mm): 58

Qty.	Order no.	Description	
1	6.2060.010	**Cable support** Used with 860 KF Thermoprep.	
1	6.2063.010	**Sample holder for 6.2419.007 sample vials (6 mL)**	
1	6.2419.007	**Sample vial 6 ml / 100 pieces** 6.1448.050 septum caps. Used for KF-determinations with oven. Volume (mL): 6	
1	6.2621.110	**Crimping tongs** Crimping tongs for sealing 6 mL sample vials (6.2419.000, 6.2419.007). Length (mm): 210	

Qty.	Order no.	Description	
1	6.2621.120	**Hexagon key 1.5 mm**	
1	6.2621.130	**Hexagon key 2 mm**	
1	6.2627.000	**Oven insert** Used with 860 KF Thermoprep.	
1	6.2724.010	**Dust filter** Dust filter for Rancimats and for Karl Fischer ovens Outer diameter (mm): 44 Length (mm): 53	

Qty.	Order no.	Description	
1	**6.2739.000**	**Wrench** For tightening connectors Length (mm): 68	
1	**6.2811.000**	**Molecular sieve** Molecular sieve. Bottle containing 250 g. Pore size: 0.3 nm. Without moisture indicator. For Rancimats and Karl Fischer instructions.	
1	**6.2816.070**	**Injection needle** Used with 832 KF Thermoprep. Material: Stainless steel (AISI 304)	
1	**6.2816.080**	**Outlet needle** Used with KF Thermoprep and Oven Sample Processor. Material: Stainless steel (AISI 304)	
1	**6.2122.0x0**	**Mains cable with C13 line socket IEC-60320-C13**	

Qty.	Order no.	Description
		Cable plug according to customer requirements.
		Switzerland: Type SEV 12 6.2122.020
		Germany, …: Type CEE(7), VII 6.2122.040
		USA, …: Type NEMA/ASA 6.2122.070
1	**8.860.8003EN**	**860 KF Thermoprep Manual**

11.2 Optional accessories

Order no.	Description
6.1448.050	**Aluminum septum caps / 1000 pieces**

Including Al sealing.

Material:	Aluminum
Material 2:	Silicone
Height (mm):	7.4
Outer diameter (mm):	20.7

6.2049.050 **Needle holder with Luer-lock for 860 KF Thermoprep**

Length (mm): 73

6.2419.000 **Sample vial 6 mL / 1000 pieces**

For 6.1448.050 septum caps.

Height (mm):	38
Outer diameter (mm):	22
Volume (mL):	6

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