899 Coulometer

Manual

8.899.8001EN / 2020-02-25

Metrohm

Metrohm AG
CH-9100 Herisau
Switzerland
Phone +41 71 353 85 85
Fax +41 71 353 89 01
info@metrohm.com
www.metrohm.com

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

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.

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1 Introduction

1.1 Instrument description

The 899 Coulometer is a titrator used for coulometric water content determination according to Karl Fischer. There are method templates available which are already configured except for a few parameters. The methods can be modified and stored under a new name. The methods can be exported to a connected USB flash drive. This function allows you to copy methods quickly and easily from one device to another.

The 899 Coulometer has an integrated magnetic stirrer that is visible on the top side of the housing. The titration cell (coulometry) can easily be attached to the magnetic stirrer. Thanks to its compact construction, you can use the instrument in a small space as a stand-alone titrator. Furthermore, the remote interface permits you to connect a sample changer with oven module and to perform automated determinations.

As an alternative to the power supply with the power supply unit provided, the 899 Coulometer can also be operated by means of an accumulator (a so-called Power Box). The Power Box is available as optional equipment. It is particularly appropriate for use in environments where the power supply is either unstable or absent.

1.1.1 Connectors

The instrument is equipped with the following connectors:

- **Electrical connection**
  For connecting to the power supply with the aid of the power supply unit provided or for connecting to the 6.2164.500 Power Box.
- **MSB connector (Metrohm Serial Bus)**
  For connecting an additional stirrer.
- **USB (OTG) connector**
  The 6.2151.100 adapter can be used to connect, for example, a printer, a USB flash drive or a USB keyboard.
- **Sensor connectors**
  Three connectors for the following sensor types:
  - Double Pt electrode
  - Generator electrode
  - Temperature sensor (Pt1000)
- **Grounding socket**
  For grounding the coulometer.
- **Remote connector**
  For connecting instruments with a remote interface (e.g. *885 Compact Oven SC*).

1.1.2 Intended use

The 899 Coulometer is designed for usage as a titrator in analytical laboratories. Its application field is coulometric water content determination according to Karl Fischer.

This instrument is suitable for processing chemicals and flammable samples. Therefore, the use of the 899 Coulometer requires the user to have basic knowledge and experience in handling toxic 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 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**)*	**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-threatening 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 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 Tubing and capillary connections

CAUTION

Leaks in tubing and capillary connections are a safety risk. Tighten all connections well by hand. Avoid applying excessive force to tubing connections. Damaged tubing ends lead to leakage. Appropriate tools can be used to loosen connections.

Check the connections regularly for leakage. If the instrument is used mainly in unattended operation, then weekly inspections are mandatory.

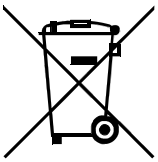
1.3.4 Flammable solvents and chemicals

WARNING

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

- 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.

2 Overview of the instrument

2.1 Front

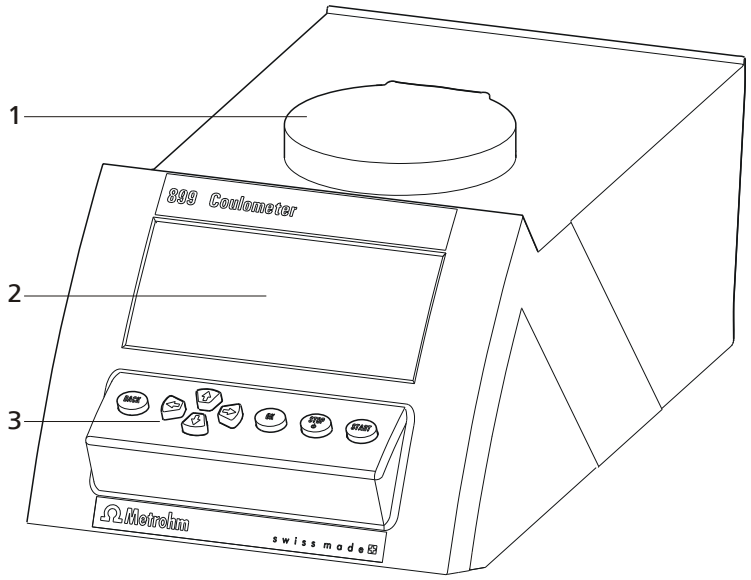


Figure 1 Front 899 Coulometer

1 Magnetic stirrer For attaching the titration vessel holder.	**2 Display**
3 Keypad	

2.2 Rear

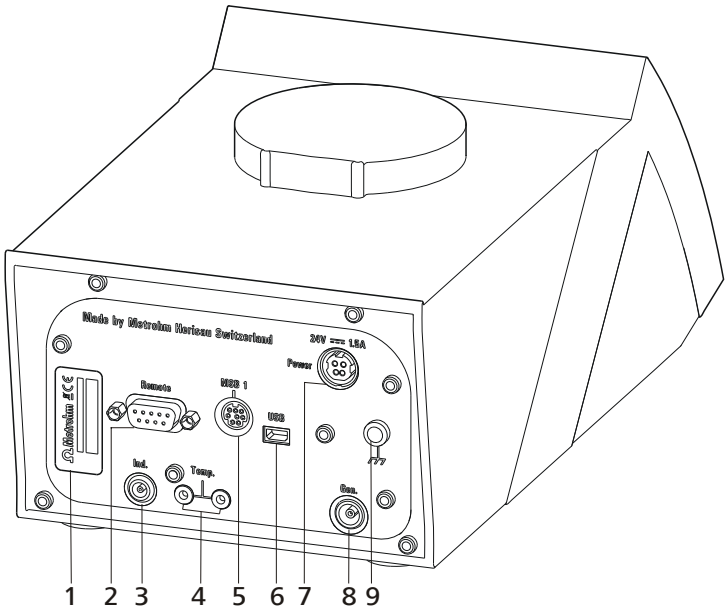


Figure 2 Rear 899 Coulometer

1 Type plate
Contains the serial number.

2 Remote connector
For connecting instruments with a remote interface. D-Sub, 9-pin.

3 Electrode connector (Ind.)
For connecting a metal electrode (double Pt wire electrode). Socket F.

4 Temperature sensor connector (Temp.)
For connecting a temperature sensor of the type Pt1000. Two B sockets, 2 mm.

5 MSB connector (MSB 1)
Metrohm Serial Bus. For connecting an external stirrer. Mini DIN, 8-pin.

6 USB (OTG) connector
For connecting printers, USB flash drives, USB hubs, etc.

7 Mains connection socket (Power)
For connecting the external power supply unit or a Power Box (6.2164.500).

8 Electrode connector (Gen.)
For connecting a generator electrode.

9 Grounding socket
For grounding the Coulometer. Socket B, 4 mm.

3 Installation

3.1 Setting up the instrument

3.1.1 Packaging

The instrument is supplied in protective 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 and free of vibrations and which provides protection against corrosive atmosphere and contamination by chemicals.

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

3.2 Setting up the titration cell for coulometry

3.2.1 Inserting the titration cell

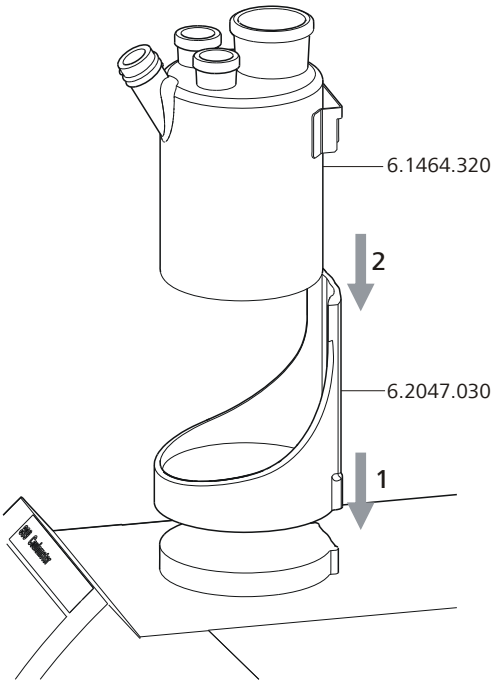


Figure 3 Inserting the titration cell (coulometry)

Set up the titration cell on the magnetic stirrer as follows:

1 Attach the 6.2047.030 titration vessel holder to the magnetic stirrer.

2 Insert the 6.1464.320 titration cell into the titration vessel holder.

3.2.2 Preparing the titration cell

Filling the adsorber tube

Before setting up the titration cell, the 6.1403.030 adsorber tube has to be filled with 6.2811.000 molecular sieve. Proceed as follows:

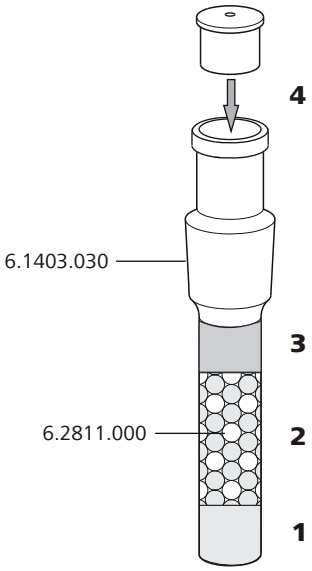


Figure 4 Filling the adsorber tube

1 Insert a small cotton plug into the bottom of the adsorber tube. Do not pack the cotton too tightly.

2 Fill the molecular sieve up to ¾ of the height.

3 Place a small cotton plug on the molecular sieve. Do not pack the cotton too tightly.

4 Seal the adsorber tube with the appropriate cover.

NOTICE

Note that the molecular sieve must be replaced at regular intervals. Each time you refill the adsorber tube with molecular sieve, you can, for example, write the date directly on the adsorber tube.

Equipping the titration cell

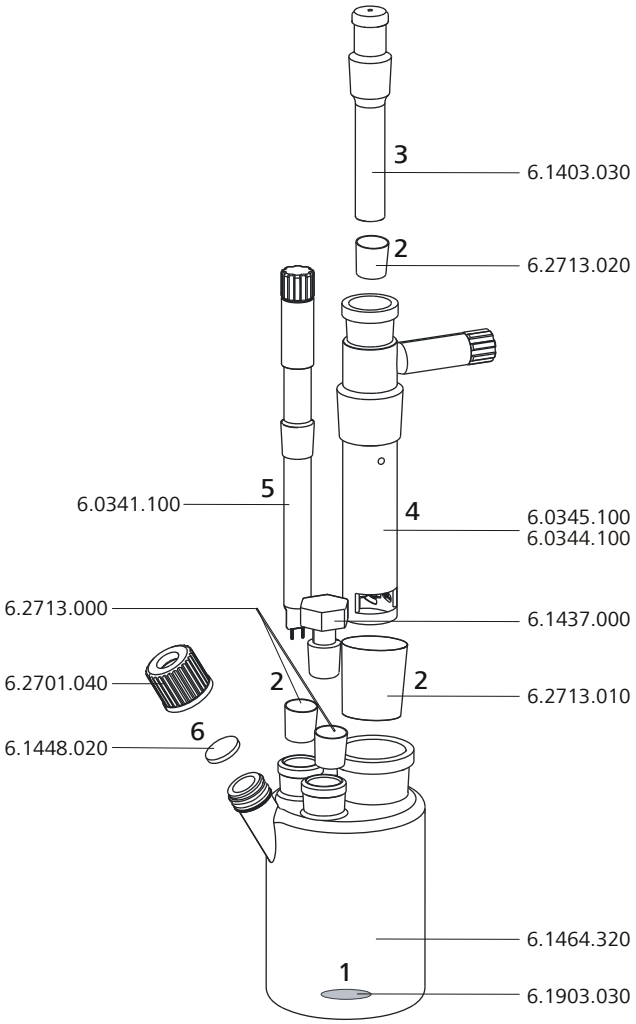


Figure 5 Equipping the titration cell (coulometry)

Equip the titration cell as follows:

1 Place the 6.1903.030 stirring bar in the titration cell.

2 Cut the 6.2713.0x0 ground-joint sleeves to the correct length and attach them to the ground joints of the inserts (electrodes, adsorber tube, etc.).

Make sure that the edges of the ground-joint sleeves are cut to size cleanly and that there are no fringes. The ground-joint sleeves must not protrude at the lower edge of the ground-joint opening.

3 Insert the 6.1403.030 adsorber tube into the generator electrode.

4 Insert the 6.0345.100 generator electrode without diaphragm or the 6.0344.100 generator electrode with diaphragm together with the adsorber tube into the large ground-joint opening at the rear.

5 Insert the 6.0341.100 indicator electrode into the left ground-joint opening.

6 Place the 6.1448.020 septum on the front opening of the titration cell and screw it shut with the 6.2701.040 screw cap.

Tighten the screw cap only enough so that it seals. The septum is not permitted to bend.

Filling the titration cell (generator electrode with diaphragm)

Proceed as follows when using a generator electrode with a diaphragm:

1 Fill approximately 5 mL of catholyte into the generator electrode.

2 Fill approximately 100 mL of anolyte into the titration cell with the aid of the 6.2738.000 funnel. The level of the anolyte should be roughly 1 - 2 mm above the level of the catholyte.

3 Close the remaining ground-joint opening on the right with the 6.1437.000 ground-joint stopper (with ground-joint sleeve attached).

Filling the titration cell (generator electrode without diaphragm)

Proceed as follows when using a generator electrode without a diaphragm:

1 Fill approximately 100 mL of reagent into the titration cell with the aid of the 6.2738.000 funnel.

2 Close the remaining ground-joint opening on the right with the 6.1437.000 ground-joint stopper (with ground-joint sleeve attached).

3.2.3 Mounting the addition and aspiration tube (utilization with Ti Stand)

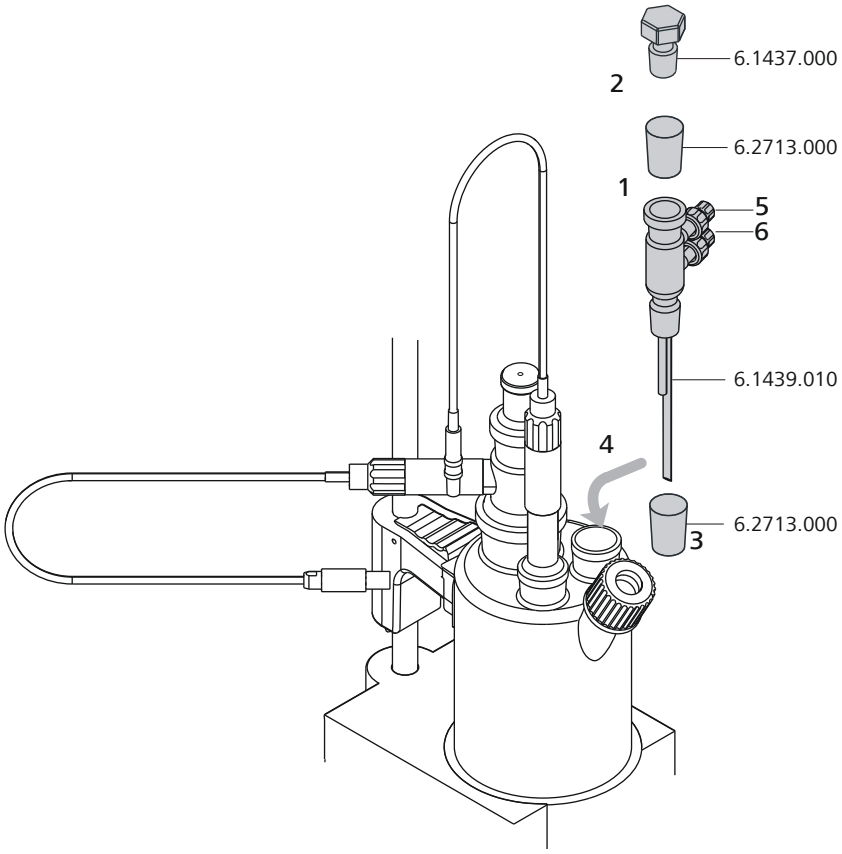


Figure 6 Mounting the addition and aspiration tube

Insert the addition and aspiration tube as follows into the titration cell:

1 Attach the 6.2713.000 ground-joint sleeve that has been cut to size to the ground joint of the 6.1437.000 stopper.

2 Insert the stopper into the 6.1439.010 addition and aspiration tube.

3 Attach the 6.2713.000 ground-joint sleeve that has been cut to size to the ground joint of the addition and aspiration tube.

4 Insert this assembly into the ground-joint opening.

5 Connect the tubing for the reagent addition at the upper connector of the addition and aspiration tube (5).

6 Connect the tubing for the aspiration of the titration cell at the lower connector of the addition and aspiration tube (6).

Details regarding how to connect the addition tubing and the aspiration tubing can be found in the manual for the *803 Ti Stand*.

3.2.4 Using the titration cell with a Karl Fischer oven

When samples release their water only slowly or only at higher temperatures, the oven method is used. The sample is heated in a KF oven (e.g. *860 KF Thermoprep*) and the water that is released is transferred to the titration cell with a carrier gas. You will find recommended parameter settings for determinations with a Karl Fischer oven in chapter *9.5, page 97*.

A detailed description of how to set up the titration cell with the KF oven can be found in the respective manual.

3.2.5 Using the titration cell with a sample changer

If a large number of samples have to be processed, the determination of the water content can be automated with the aid of a sample changer with oven module (e.g. *885 Compact Oven SC*). You will find recommended parameter settings for determinations with a Karl Fischer oven in chapter *9.5, page 97*.

A detailed description of how to set up the titration cell with the sample changer can be found in the respective manual.

3.3 Connecting the coulometer to the power supply

You can supply the 899 Coulometer with electricity in two different ways:

- Connect the coulometer directly to the power supply with the aid of the power supply unit provided.
- Connect the coulometer to the 6.2164.500 Power Box if no stable power supply is available.

3.3.1 Connecting the power supply unit

The 899 Coulometer has an external power supply unit for a 24 V power supply (DC). This is connected to the power socket of the coulometer.

WARNING

An incorrect supply voltage can damage the instrument.

Operate the instrument only with the supply voltage specified for it. Use the supplied power supply unit exclusively.

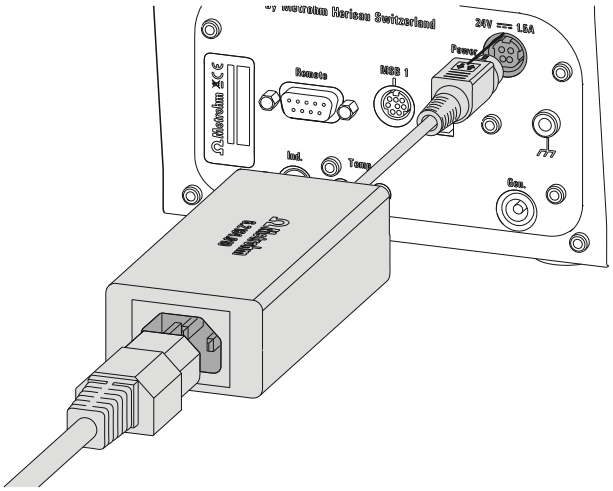


Figure 7 Connecting the power supply unit

Proceed as follows:

1 Connect the plug of the external power supply unit with the power socket *(2-**7**)* of the coulometer.

NOTICE

The plug of the power supply unit is protected against accidental disconnection of the cable by means of a pull-out protection feature. If you wish to pull out the plug, you first need to pull back the outer plug sleeve.

2 Connect the power supply cable with the external power supply unit of the coulometer and with the power supply.

CAUTION

Switch off the 899 Coulometer correctly by pressing the red **[STOP]** key before you interrupt the electricity supply. If this is not done, then there is a danger of data loss.

3.3.2 Connecting the Power Box

As an alternative to the power from the power supply, it is possible to provide the 899 Coulometer with electricity through the 6.2164.500 Power Box. This means that you can also use the instrument in environments in which no stable power supply is available. Details regarding the Power Box can be found in the respective manual.

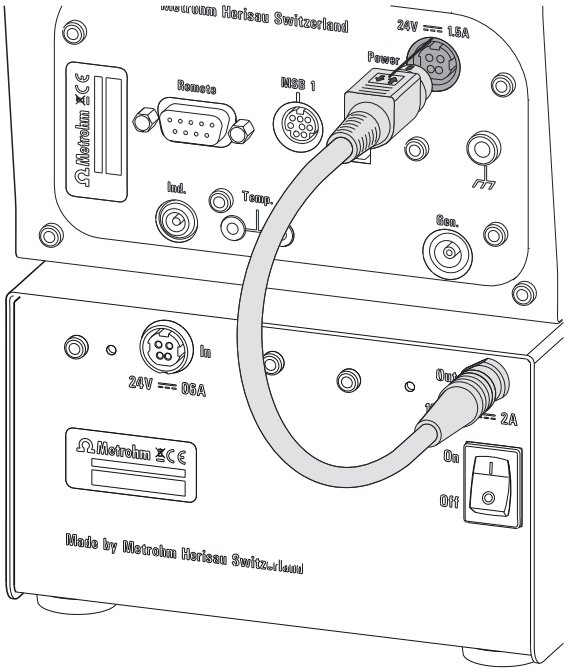


Figure 8 Connecting the Power Box

Proceed as follows:

1 Connect the plug of the Power Box with the power socket *(2-**7**)* of the coulometer.

NOTICE

The Power Box plug is protected against accidental disconnection of the cable by means of a pull-out protection feature. If you wish to pull out the plug, you first need to pull back the outer plug sleeve.

CAUTION

Switch off the 899 Coulometer correctly by pressing the red **[STOP]** key before you interrupt the connection with the Power Box. If this is not done, then there is a danger of data loss.

3.4 Connecting sensors

The measuring interface contains the following measuring inputs:

- **Gen.** for a generator electrode
- **Ind.** for a double Pt electrode
- **Temp.** for a temperature sensor of the Pt1000 type

CAUTION

Under all circumstances, avoid mixing up the electrode cable from the indicator electrode with the one from the generator electrode. Mark the screw heads of the cables accordingly.

3.4.1 Connecting a generator electrode

Screwing the electrode cable to the generator electrode

1 Unscrew the cover of the generator electrode.

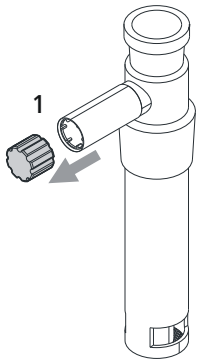


Figure 9 Unscrewing the cover from the generator electrode

2 Tighten the 6.2104.120 electrode cable to the generator electrode.

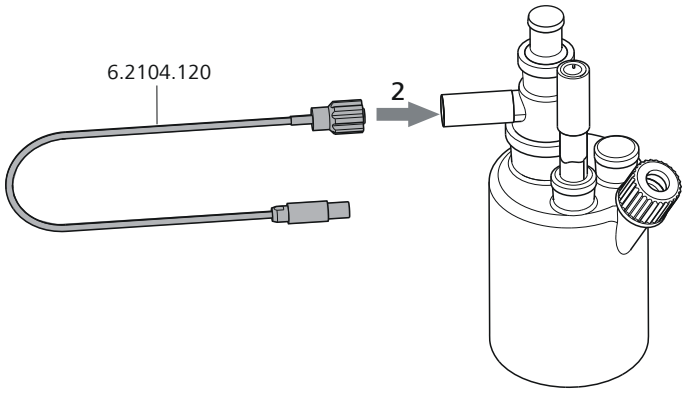


Figure 10 Screwing the electrode cable to the generator electrode

Connecting the electrode cable to the coulometer

1 Plug the electrode plug into the **Gen.** socket of the coulometer.

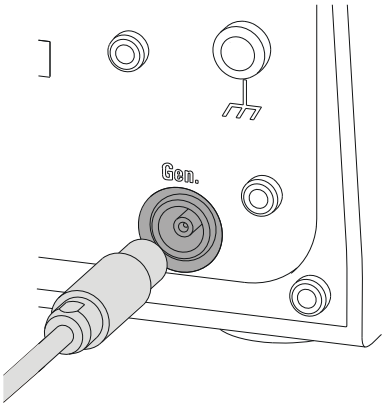


Figure 11 Connecting a generator electrode

NOTICE

The electrode cable is protected against accidental disconnection of the cable by means of a pull-out protection. If you wish to pull out the plug again, you first need to pull back the outer plug sleeve.

3.4.2 Connecting an indicator electrode

Screwing the electrode cable to the indicator electrode

1 Unscrew the cover of the indicator electrode.

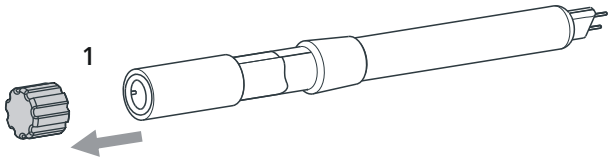


Figure 12 Unscrewing the cover from the indicator electrode

2 Tighten the 6.2104.020 electrode cable to the indicator electrode.

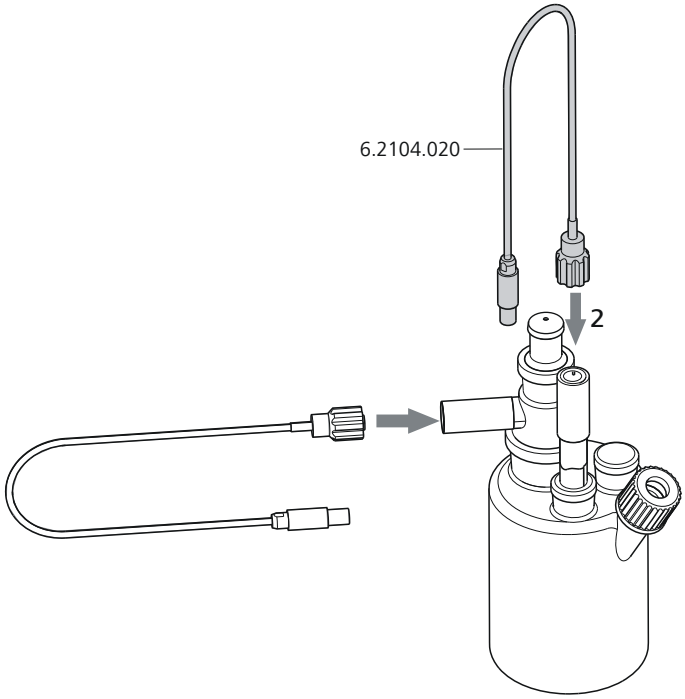


Figure 13 Screwing the electrode cable to the indicator electrode

Connecting the electrode cable to the coulometer

1 Plug the electrode plug into the **Ind.** socket of the coulometer.

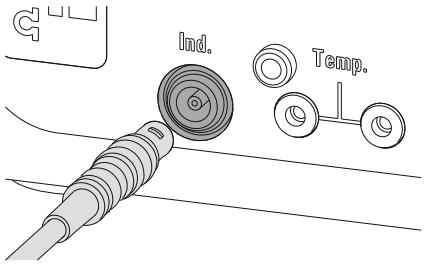


Figure 14 Connecting an indicator electrode

NOTICE

The electrode cable is protected against accidental disconnection of the cable by means of a pull-out protection. If you wish to pull out the plug again, you first need to pull back the outer plug sleeve.

3.4.3 Connecting a temperature sensor

A temperature sensor of the Pt1000 type can be connected to the **Temp.** connector.

Connect the temperature sensor as follows:

1 Insert the plugs of the temperature sensor into the **Temp.** sockets of the coulometer.

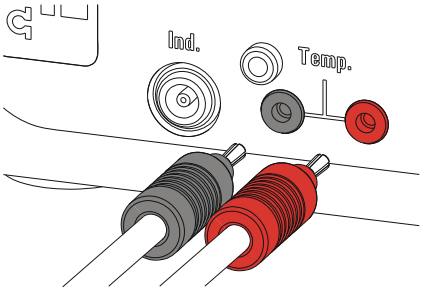


Figure 15 Connecting a temperature sensor

NOTICE

Always insert the red plug into the red socket. This is the only way that shielding against electrical interference can be ensured.

3.5 Connecting an additional stirrer

Instead of the built-in magnetic stirrer, you can use the magnetic stirrers *801 Stirrer* or *803 Ti Stand*.

Proceed as follows:

1 Switch off the coulometer.

2 Connect the connection cable of the magnetic stirrer to MSB 1 on the rear of the coulometer.

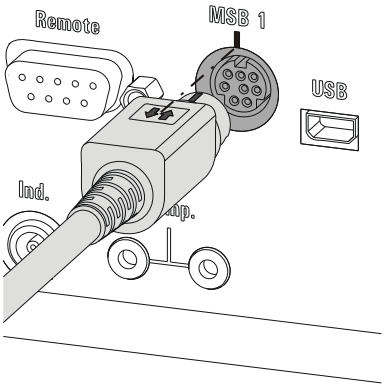


Figure 16 Connecting a stirrer

NOTICE

Make sure that the flat side of the plug matches the marking on the socket.

3 Switch on the coulometer.

3.6 Connecting a balance

As a rule, balances are equipped with a serial RS-232 interface. To connect a balance, you require a 6.2148.030 RS-232/USB Box.

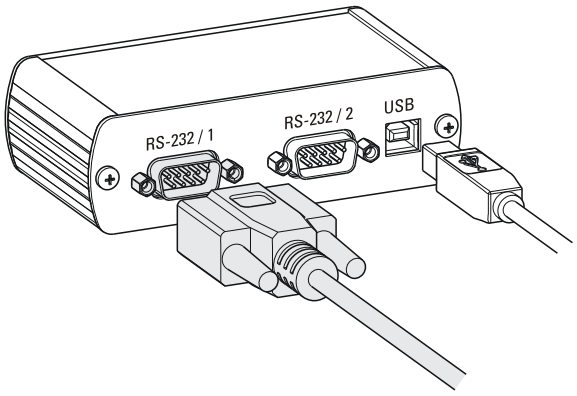


Figure 17 Connecting the balance via RS-232/USB Box

Proceed as follows:

1. Connect the RS-232/USB Box with a 6.2151.020 connecting cable (USB A - USB B) on the USB (OTG) connector of the coulometer.

   You can connect the RS-232/USB Box to the coulometer either via a USB hub or using a 6.2151.100 USB MINI (OTG) - USB A adapter *(see chapter 3.7, page 24)*.

2. Connect the 9-pin plug of the respective balance connecting cable to the **RS-232/1** connector. Consult the following list or contact the balance manufacturer in order to find the correct balance connecting cable.

NOTICE

The parameters for the RS-232 interfaces on the 899 Coulometer must match those on the balance *(see "Editing the COM1 settings", page 69)*. Also check the manual for the balance.

The following table offers an overview of the balances that you can use together with the 899 Coulometer and of which cable you will need for connection to the RS-232 interface:

Balance	Cable
AND ER, FR, FX with RS-232 interface (OP-03)	6.2125.020 + 6.2125.010
Mettler AB, AG, PR (LC-RS9)	In the scope of delivery for the balance
Mettler AM, PM, PE with interface option 016 or Mettler AJ, PJ with interface option 018	6.2146.020 + 6.2125.010 Also from Mettler: ME 47473 adapter and either ME 42500 hand switch or ME 46278 foot switch
Mettler AT	6.2146.020 + 6.2125.010 Also from Mettler: ME 42500 hand switch or ME 46278 foot switch
Mettler AX, MX, UMX, PG, AB-S, PB-S, XP, XS	6.2134.120
Mettler AE with interface option 011 or 012	6.2125.020 + 6.2125.010 Also from Mettler: ME 42500 hand switch or ME 46278 foot switch
Ohaus Voyager, Explorer, Analytical Plus	Cable AS017-09 from Ohaus
Precisa balances with RS-232-C interface	6.2125.080 + 6.2125.010
Sartorius MP8, MC, LA, Genius, Cubis	6.2134.060
Shimadzu BX, BW	6.2125.080 + 6.2125.010

3.7 Connecting a keyboard, printer and other USB devices

The 899 Coulometer has a USB (OTG) connector. Use the 6.2151.100 USB MINI (OTG) - USB A adapter supplied for connecting USB devices.

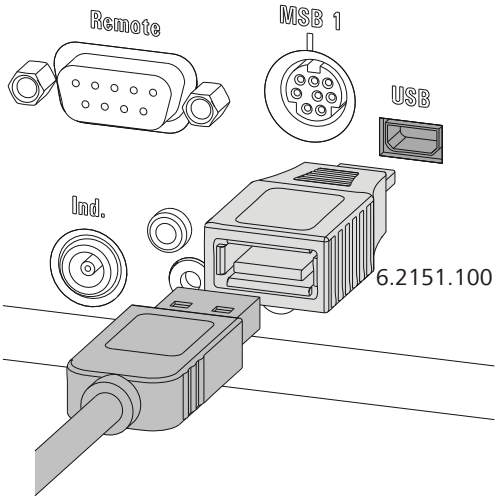


Figure 18 Connecting USB devices

CAUTION

Switch the instrument off before connecting or disconnecting a USB device or a USB flash drive.

The 899 Coulometer can recognize the USB device only immediately after switching on.

NOTICE

Many USB devices need a so-called USB hub in order to work correctly.

A USB hub is a distributor to which several USB devices can be connected. USB hubs are available in specialty stores in a number of different models.

The 6.2147.000 numerical USB keypad can, in addition to its function as keyboard, also be used as a USB hub. It has two USB connectors. You cannot however use these two USB connectors to connect any PC keyboards, barcode readers or additional keypads with numerical keypads. These devices are recognized as input devices (like the numerical USB keypad) and they cannot be switched in series.

The following list provides you with an overview of the various USB devices and how you can connect them to the Coulometer.

USB device	Connection options
USB flash drives (for the backup or storing of methods)	▪ With 6.2151.100 adapter USB MINI (OTG) - USB A ▪ With USB hub ▪ With 6.2147.000 numerical USB keypad
6.2147.000 numerical USB keypad (for comfortable numerical input and for navigating in the dialog)	▪ With 6.2151.100 adapter USB MINI (OTG) - USB A ▪ With USB hub
6.2148.030 RS-232/USB Box (for connecting a balance, a PC or to the RS-232 remote control)	▪ With 6.2151.100 adapter USB MINI (OTG) - USB A ▪ With USB hub ▪ With 6.2147.000 numerical USB keypad
USB hub (with or without an own power supply)	▪ With 6.2151.100 adapter USB MINI (OTG) - USB A
"Custom Neo's" printer with 6.2151.120 cable	▪ Directly on the USB (OTG) connector of the 899 Coulometer
"Custom Neo's" printer with 6.2151.020 cable	▪ With 6.2151.100 adapter USB MINI (OTG) - USB A ▪ With USB hub ▪ With 6.2147.000 numerical USB keypad
USB printer with 6.2151.020 connecting cable	Depending on the model of the printer: ▪ With 6.2151.100 adapter USB MINI (OTG) - USB A or ▪ With USB hub ▪ With 6.2147.000 numerical USB keypad
PC mouse with USB cable (for navigating in the dialog)	▪ With USB hub ▪ With 6.2147.000 numerical USB keypad

USB device	Connection options
PC keyboard with USB cable (for the comfortable input of letters and numbers)	▪ With USB hub
Barcode reader with USB cable	▪ With USB hub
Keypad with numerical keypad with USB cable	▪ With USB hub

If you wish to connect **several different instruments that do not have their own power supply**, then it is possible you will need to use a USB hub that does have its own power supply (*self powered*). The USB (OTG) connector of the 899 Coulometer is not designed for supplying power to several devices with elevated electricity requirements.

Also observe the instructions in *chapter 9.3, page 93*.

Examples:

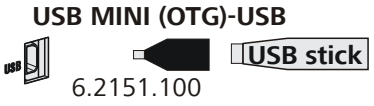


Figure 19 Connecting the USB flash drive

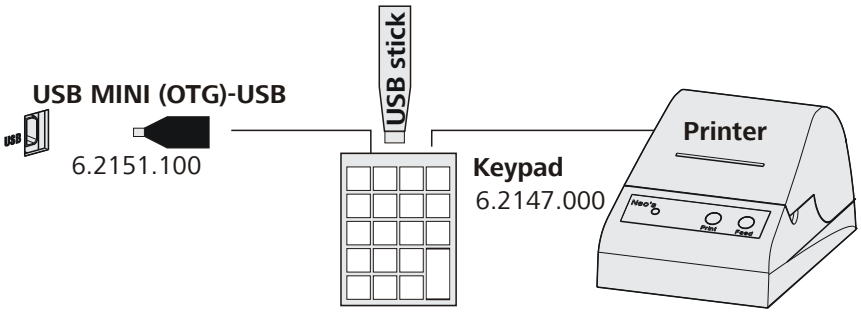


Figure 20 Connecting the 6.2147.000 USB keyboard with USB flash drive and printer

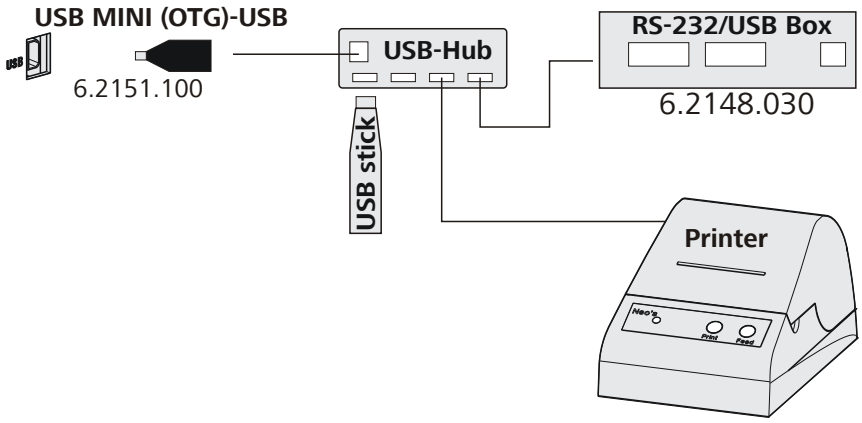


Figure 21 Connecting the USB hub with USB flash drive, printer and 6.2148.030 RS-232/USB Box

3.8 Connecting a sample changer to the remote connector

The 899 Coulometer can be connected to a sample changer with oven module with the aid of the 6.2141.390 remote cable. This makes it possible to integrate the Coulometer in an automation system.

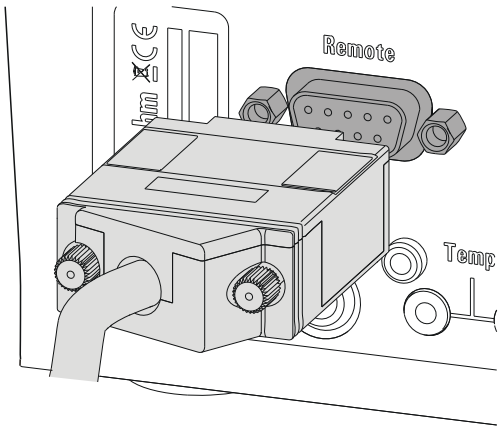


Figure 22 Connecting a remote cable

Details regarding the use of the sample changer (e.g. *885 Compact Oven SC*) can be found in the respective manual.

4 Coulometric titration

4.1 Principle of coulometry according to Karl Fischer

The **coulometric Karl Fischer titration** is a variation of the classic water content determination method according to Karl Fischer. The conventional method works with a methanolic solution of iodine, sulfur dioxide and a base as buffer substance. If an aqueous sample is titrated, then several reactions take place that can be summarized in the following sum equation:

$$H_2O + I_2 + [RNH]SO_3CH_3 + 2\ RN \rightleftarrows [RNH]SO_4CH_3 + 2\ [RNH]I$$

According to the equation above the I_2 reacts quantitatively with H_2O. This chemical equation serves as a basis for the water content determination.

With the **coulometric Karl Fischer titration**, the necessary iodine is directly and electrochemically generated in the electrolyte containing iodine ("electronic buret"). Between the amount of electric charge and the amount of generated iodine, there is a strictly quantitative relationship, which is used for high-precision dosing of the iodine. Because the coulometric Karl Fischer method is an **absolute determination**, no titer needs to be determined. It must only be ensured that the reaction generating the iodine runs with a 100% current efficiency. All of the reagents available today ensure this.

The endpoint indication is effected voltametrically by modulating an alternating current of constant strength to a double Pt electrode. This results in a voltage differential between the Pt wires. This is drastically reduced as soon as even the slightest amounts of free iodine are present. This circumstance is used for detecting the endpoint of the titration.

4.2 Working with water standards

4.2.1 Certified water standards

Commercially available, certified water standards with water content of 1.00 ± 0.003 mg/g and/or 0.10 ± 0.005 mg/g should be used for validating the instrument as a whole, integrated system.

NOTICE

The 1.0 mg/g water standard is easier to handle and is therefore preferred.

Table 1 Recommended weighing ranges

1.0 mg/g water standard	0.2 - 2.0 g
0.1 mg/g water standard	0.5 - 5.0 g

4.2.2 Practical recommendations

For validation, it is essential to work very accurately. In order to minimize any measurement inaccuracies that could occur, the sample preparation and the sample processing should proceed in accordance with a defined scheme:

1. Put on gloves (always for Karl Fischer titration).
2. Use a clean syringe.

NOTICE

If you are working with the 0.1 mg/g water standard, then you must use a glass syringe. If you are working with the 1.0 mg/g water standard, then you may use either a plastic syringe or a glass syringe.

3. Take a new ampoule of water standard and shake it briefly.
4. With a folded paper towel held between thumb and index finger, break open the ampoule at the marking.
5. Draw approx. 1 mL of the water standard into the syringe.

6 Pull the plunger of the syringe up to the end and shake the syringe back and forth somewhat.

The inside of the syringe is rinsed by water standard and freed of water contamination.

7 Dispose of the used water standard in a waste bottle.

8 Draw the rest of the water standard into the syringe, aspirating as little air as possible.

9 Push out any air bubbles that may be present in the syringe.

10 Wipe off the needle with a lint-free paper towel and cover it with the appropriate cap.

11 Place the syringe on the balance and press **[TARA]**.

12 As soon as the drift on the 899 Coulometer is stable, take the syringe in your hand, press **[START]** and inject approx. 1 mL of the water standard through the septum.

There are two possibilities:

- Version 1:
  Inject the water standard without immersing the needle in the reagent liquid. If a little drop remains on the end of the needle, it must be aspirated back before pulling the needle out of the septum.
  The water standard should not be sprayed from the syringe onto the electrode nor onto the wall of the titration cell.
- Version 2:
  Inject the water standard directly under the surface of the reagent liquid.
  Take care to ensure that you do not aspirate any liquid when you withdraw the syringe from the reagent liquid.

13 Close the syringe with the same cap and place it back on the balance.

14 Read off the value displayed by the balance and enter it on the Coulometer as the sample size.

If you have connected a balance to the Coulometer, you may transmit the sample size directly from the balance.

15 The next determination can be started as soon as the determination has been finished and the titration cell has been conditioned (drift stable) again.

4.3 Sample addition

This chapter contains a few notes concerning sample addition. An exhaustive discussion of this topic is not possible here. Further notes can be found in the literature from the reagent manufacturers and in the following **Metrohm Application Bulletins**:

Bulletin No.	Title
No. 142	Karl Fischer water content determination in non-explosive gases
No. 145	Determination of low water contents in plastics using the KF oven method
No. 209	Coulometric water content determinations according to the the Karl Fischer method in insulating oils, hydrocarbons and their products

4.3.1 Size of the sample size

The sample weight should be small in order to be able to titrate as many samples as possible in the same electrolyte solution and to keep the titration time short. However, ensure that the sample contains at least 50 µg of H_2O. The following table helps you determine the appropriate sample size.

Table 2 Recommended sample sizes

Water content of the sample	Sample size	Resulting water content
10,000 ppm = 1%	10 mg - 100 mg	100 µg - 1,000 µg
1,000 ppm = 0.1%	100 mg - 1 g	100 µg - 1,000 µg
100 ppm = 0.01%	1 g	100 µg
10 ppm = 0.001%	5 g	50 µg

4.3.2 Working with liquid samples

Liquid samples are added with a syringe. The samples can be injected two different ways:

- One uses a syringe with a long needle, which one immerses in the reagent during the injection.
- One uses a syringe with a short needle and aspirates the last drops back into the needle.

The best way for you to determine the injected sample amount is to reweigh the sample.

Glass syringes should be used for the **determination of traces and validations**. We recommend obtaining these from a specialized syringe manufacturer.

Highly volatile samples and samples of low viscosity should be cooled before sampling. Doing so avoids losses while working. The syringe must, however, not be cooled directly, as condensation could be formed. For the same reason, no air may be aspirated into a syringe into which a cooled sample has been aspirated beforehand.

Samples of high viscosity can be thinned by heating. The syringe must be heated as well. The same target can be reached by diluting with suitable solvents. In this case the water content of the solvent has to be determined and subtracted as a blank value.

Pastes and fats can be added to the titration cell with a syringe without needle. You can use the ground-joint opening for this. If you also wish to aspirate, you can use the opening with the septum stopper. The best way for you to determine the sample amount is to reweigh the sample.

If samples contain only **traces of water**, then the syringe has to be predried well. If possible, the syringe should be rinsed with the sample solution by filling in and discarding solution several times.

4.3.3 Working with solid samples

If possible, solid samples are to be extracted or dissolved in a suitable solvent. The resulting solution is injected, during which a blank value correction for the solvent must be carried out.

If no suitable solvent can be found for a solid sample, or if the sample reacts with the Karl Fischer reagent, then a Karl Fischer oven should be used.

If solid samples are added directly into the titration cell, then the generator electrode without diaphragm should be used. The samples can be added through the ground-joint opening or through the side opening. While doing so, take care to ensure that

- the sample releases its moisture completely.
- no side reaction with the Karl Fischer reagent takes place.
- the surfaces of the electrodes are not covered by the sample substance (incomplete KF reaction).
- the Pt grid of the generator electrode does not become damaged.
- the Pt wires of the indicator electrode do not become damaged.

4.4 Optimum working conditions

4.4.1 General

When a titration cell that has been well dried-out beforehand is put into operation with a generator electrode without diaphragm, the basic drift will be reached within approx. 30 minutes. It is recommended that the titration cell be repeatedly and carefully shaken during this time.

For generator electrodes with diaphragms, you should expect a preparation time of approx. 2 hours.

To obtain precise determinations of amounts of water less than 100 µg, it may also be of advantage to condition the titration cell overnight before using it.

If the Coulometer is switched off for extended periods with the titration cell filled, it will take some time for it to become conditioned again after it is switched back on.

With continuous use, the Coulometer should not be switched off overnight.

4.4.2 Drift

A constant drift in the range of ≤ 4 µg/min is all right. Lower values are, however, quite possible. Higher but stable values will still produce good results because it is possible to compensate for the drift.

A constantly high drift can be caused by water-containing deposits in inaccessible parts of the titration cell. In these cases, shaking the cell can reduce the value. Ensure that there are no drops above the liquid level in the titration cell.

If you are working with a generator electrode with diaphragm, do not shake the cell so hard that the catholyte and anolyte mix with one another. If the drift remains too high for a prolonged time, even after shaking the cell, then the electrolyte solutions should be replaced. The catholyte should be replaced once per week.

A wet catholyte can be another reason for the excessively high drift. The wet catholyte can be dried with a KF one-component reagent.

When you work with a Karl Fischer oven, a drift ≤ of 10 µg/min is all right. The drift depends on the gas flow (the smaller the gas flow, the lower the drift) and on the humidity of the surroundings.

4.4.3 Reagent replacement

The electrolyte solutions must be replaced in the following cases:

- The titration cell is too full.
- The KF reagent has reached its capacity limit.
- The drift is too high, and cannot be reduced by shaking the titration cell.
- A two-phase-mixture is being formed in the titration cell; in this case it is also only possible to aspirate the sample phase.

Exhausted electrolyte solution is best disposed of by aspiration. To do this, you can use, for example, an *803 Ti Stand* with built-in membrane pump. An advantage is that the titration cell does not have to be disassembled.

In the event of severe contamination, the titration cell can be rinsed with a suitable solvent which is also aspirated.

In the case of a generator electrode with diaphragm, the catholyte should be replaced once per week. Longer use can cause blackening and yellow precipitates in the cathode chamber. An unpleasant smell is also a sign of having used the catholyte for too long.

4.4.4 Indicator electrode

A new indicator electrode can take a certain warm-up time to form the surface. During this time, unexpectedly long titration times and excessively high measurement results can occur. These phenomena will, however, disappear after a short time of use. In order to accelerate the setting of a new indicator electrode, the 899 Coulometer can be conditioned (e.g. over night).

A contaminated indicator electrode can be carefully cleaned with an abrasive agent (6.2802.000 polishing set or toothpaste). After the cleaning, rinse with ethanol.

The two Pt wires of the indicator electrode should run as parallel as possible to one another. Check the Pt wires before inserting the electrode.

5 Operation

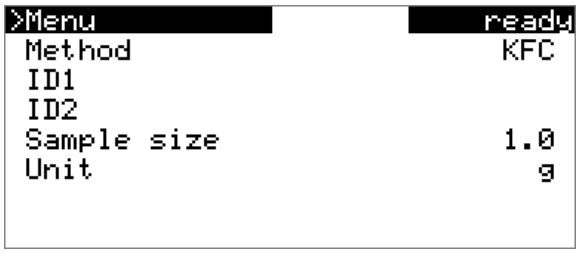
5.1 Switching the instrument on and off

Switching on the instrument

Proceed as follows:

1 ▪ Press the red **[STOP]** key.
The instrument is initialized and a system test performed. This process takes some time.

The main dialog is displayed:



Switching off the instrument

The instrument is switched off with the **[STOP]** key. The fact that the key needs to be pressed down for an extended time prevents accidental switch off.

Proceed as follows:

1 ▪ Keep the red **[STOP]** key pressed down for at least 3 s.

A progress bar is displayed. If the key is released during this time, then the instrument will not be switched off.

5.2 Fundamentals of operation

5.2.1 The keypad

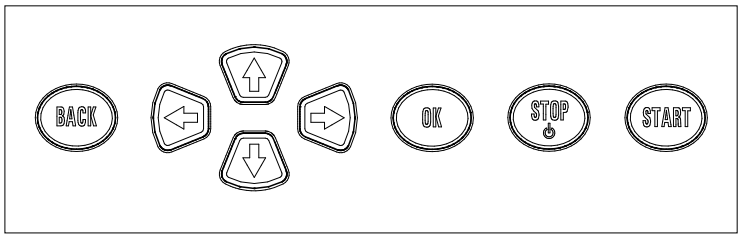
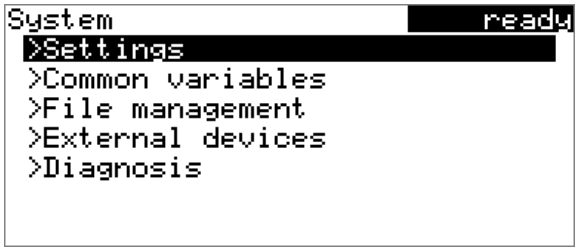


Figure 23 Keypad 899 Coulometer

BACK	Apply the input and exit the dialog.
⇧ ⇩	Move the selection bar either up or down by one line at a time. Select the character to be entered in the text editor.
⇦ ⇨	Select the character to be entered in the text and number editor. Select the individual functions in the function bar.
OK	Confirm the selection.
STOP	Stop an ongoing method run or a manual function. Switch the instrument on or off.
START	Start a method run or a manual function.

5.2.2 Structure of the dialog windows



The current dialog title is displayed on the left-hand side of the title bar. The current status of the system is displayed in the upper right-hand corner:

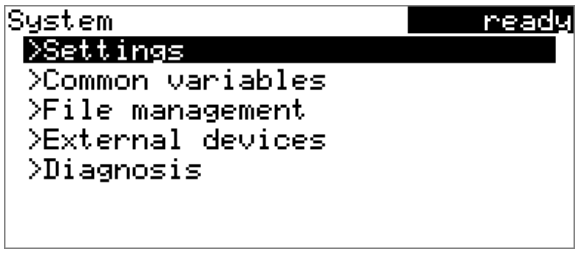
ready	The instrument is in normal status.
cond.busy	The working medium is being conditioned.
cond.ok	The working medium is conditioned.
busy	A method has been started.
hold	A method has been paused.

Some dialogs have a so-called function bar on the bottom line. The functions contained therein can be selected with the arrow keys **[⇦]** or **[⇨]** and executed with **[OK]**.

5.2.3 Navigating in the dialog

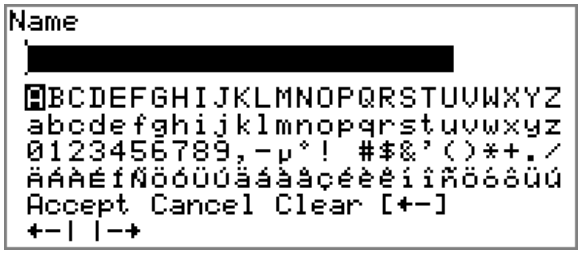
The selection bar is displayed in inverted style. Use the arrow keys **[⇧]** and **[⇩]** to move the selection bar upward or downward one line at a time. If a dialog text is marked with " **>** ", then additional settings are available in a subordinate dialog. Use **[OK]** to access this dialog.

Example: System settings



Use the **[BACK]** key to return to the next higher level.

5.2.4 Entering text and numbers



In the editing dialog for text input or numerical input, select the individual characters with the arrow keys. Use **[OK]** to apply the character in the input field. The following functions are available:

Editing function	Description
Accept	The modification is applied and the editing dialog is exited.
Cancel	The editing dialog is exited without applying the modification.

Editing function	Description
Clear	The content of the input field is deleted completely.
[←]	The character left of the cursor is deleted (backspace).
←|	Text editor only The cursor within the input field is shifted to the left by one character each time that **[OK]** is pressed.
|→	Text editor only The cursor within the input field is shifted to the right by one character each time that **[OK]** is pressed.
[BACK]	The modification is applied and the editing dialog is exited.

The **[BACK]** key has the same function as **Accept**.

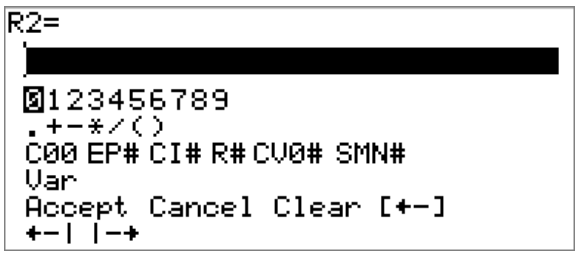
A commercially available USB keyboard can be connected to make it easier to enter text and numbers. The assignment of the keys on the PC keyboard is described in *chapter 9.3.2, page 94*.

5.2.5 Selecting from a selection list

In a selection list, select the individual entries with the arrow keys **[⇧]** and **[⇩]**. Accept the selection with **[OK]** or **[BACK]**.

5.3 Formula editor

The formulas for the calculations are entered with the formula editor. The formula editor is equipped with an automatic syntax check. This is triggered as soon as a formula is applied. The generally valid rules of priority apply for the calculation operations.



Variable	Description
C00	Sample size
EP#	Water quantity at the endpoint EP# (# = 1…9)
CI#	Sample identification (# = 1…2)
R#	Result (# = 1…5)
CV0#	Common variable (# = 1…5)
SMN#	Mean value of result R# (# = 1…5)
Var	List of additional variables *(see "Variables", page 39)*

"#" stands for a sequential number that you must enter manually. Example: if you apply the variable **EP#** in the formula, only **EP** is entered. You will still need to enter the number yourself.

The meanings of the editing functions are explained in *chapter 5.2.4, page 37*.

Variables

Pressing **Var** displays a list with additional variables. You can enter these variables either directly into the formula or also by selecting them from the list and applying them with **[OK]**.

Variable	Description
MCQ	End quantity, i.e. total amount of removed water at the end of the titration (in µg)
MCD	Duration of the entire titration
MDC	Drift for drift correction

Variable	Description
DDC	Time for drift correction
MIM	Initial measured value, i.e. measured value prior to the processing of the start conditions
MIT	Initial temperature, i.e. temperature prior to the processing of the start conditions
MCM	End measured value
MCT	End temperature
DD	Duration of the entire determination

5.4 Methods

5.4.1 Method templates

The 899 Coulometer contains method templates which are already configured except for a few parameters.

The following method templates can be selected:

KFC — Coulometric Karl Fischer titration.

The blank value is not taken into account for the calculation.

KFC-Blank — Coulometric Karl Fischer titration minus the blank value.

Blank — Coulometric blank value determination.

The method templates differ only in their calculation formulas.

5.4.2 Loading a method template

Proceed as follows to load a method template:

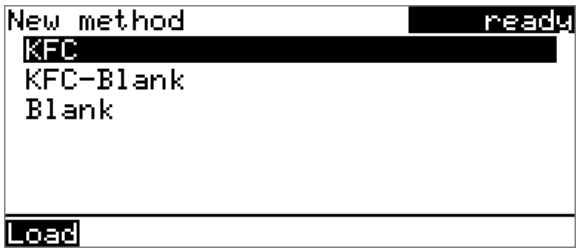
1 Opening method templates

- In the main dialog, select **Method** and press **[OK]**.

The method table with the stored methods opens:

2 In the function bar, select **New** and press **[OK]**.

The list with method templates opens:



3 Loading the method template

- Select the desired method template and press **[OK]**.

The method is now loaded and is displayed in the main dialog under **Method**.

5.4.3 Saving a method

If you modify method parameters, then you can save these as your own method. A maximum of 100 methods can be saved.

To save a method, proceed as follows:

1 Opening the method table

- In the main dialog, select **Method** and press **[OK]**.

The method table opens:

2 Modifying/applying the method name

- In the function bar, select **Store** and press **[OK]**.
  The name of the method template is suggested as the method name (e.g. **KFC-Blank**). If the method has already been saved once, then the method name already used will be displayed:

Applying the name:

- Press **[BACK]**.

The method will be saved and the method table is displayed.

Entering a new name:

- Press **[OK]**.
  The text editor opens.
- Enter a method name (max. 12 characters) and apply with **Accept** or **[BACK]**.
- Press **[BACK]**.

The method will be saved and the method table is displayed.

5.4.4 Exporting a method

The methods can be exported to a connected USB flash drive.

NOTICE

This function is possible only if a USB flash drive is connected as an external storage medium.

To export a method, proceed as follows:

1 Opening the method table

- In the main dialog, select **Method** and press **[OK]**.

The method table with the stored methods opens:

2 Selecting the method

- Select the desired method.

3 Exporting the method

- In the function bar, select **Export** and press **[OK]**.

The method is being exported. The directory structure on the USB flash drive is listed in *chapter 6.3, page 66*.

5.5 Control

Menu ▸ Control

In the dialog **Control**, the settings for the execution of a single determination or of one sample series are defined.

Sample table

If this parameter is activated, the sample data for a sample series can be entered in a table *(see chapter 5.7, page 45)*.

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

Autostart

If this parameter is activated, a new determination is started automatically at the end of a determination. This continues until the number specified has been reached (see **Number of autostarts**).

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

NOTICE

If you are working with a sample changer with oven module (e.g. *885 Compact Oven SC*), then the **Autostart** parameter must be set to **off**, because the sample changer starts the titration.

Number of autostarts

This parameter is visible only when **Autostart** = **on**.

Number of automatic starts.

Input range	**1 - 50**
Selection	**table**
Default value	**table**

table
The number of automatic starts corresponds to the number of samples in the sample table.

5.6 Sample data

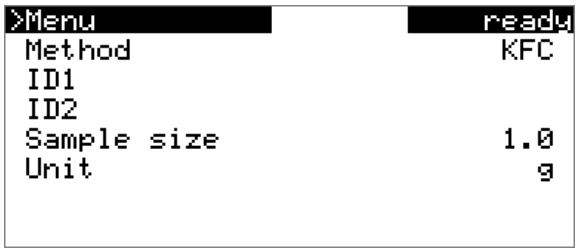
You can enter the sample data (identification, sample size, etc.) in a variety of ways:

- Directly in the main dialog.
- Using the sample table. This is particularly useful with sample series. The sample table is a table in which the sample data for up to 99 samples can be entered *(see chapter 5.7, page 45)*.
- Automatic request immediately after the start of the determination *(see chapter 5.6.2, page 45)*.

You can also send the sample size and the unit from a connected balance in any case. With some balances, the sample identification and method can be also sent *(see chapter 9.2, page 92)*.

5.6.1 Entering sample data in the main dialog

For a sample, you can enter the sample data directly in the main dialog, even while the determination is running *(see chapter 5.9, page 52)*.



ID1

Sample identification. The sample identification can be used in calculations as the variable **CI1**.

Entry	**max. 10 characters**
Default value	**empty**

ID2

Sample identification. The sample identification can be used in calculations as the variable **CI2**.

Entry	**max. 10 characters**
Default value	**empty**

Sample size

Sample size. The value of the sample size can be used in calculations as the variable **C00**.

Input range	**–999999999 - 9999999999**
Default value	**1.0**

Unit

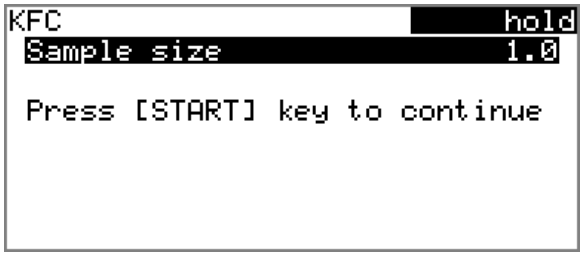
Unit of the sample size.

Selection	**g** | **mg** | **µg** | **mL** | **µL** | **pieces** | **User-defined**
Default value	**g**

User-defined
A user-defined unit can be created. This will be added to the selection list. The previous entry will be overwritten as soon as the new unit has been defined.

5.6.2 Requesting sample data at the start of the determination

The sample data can be requested immediately after the start of the determination in order to ensure that the sample data entry is not forgotten. This automatic request is indispensable when you reweigh your samples.



The corresponding parameters must be activated under **Start conditions** for this purpose. If the parameter **Hold at request** is activated, then the run will be paused and must be continued with **[START]** after the input of the sample data. If **Hold at request** is deactivated, then the titration will be started in the background. This dialog will be displayed until the entering of the sample data is confirmed with **[START]**, even if the the titration is already completed. This ensures that the sample data is available for calculations.

5.7 Sample table

5.7.1 General

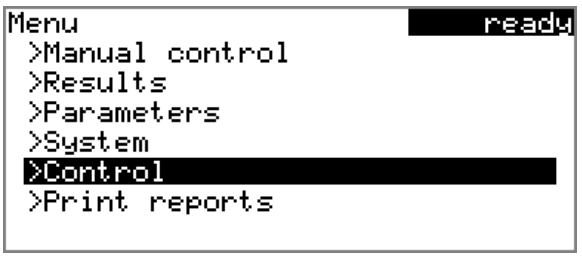
The sample table is a table in which the sample data for up to 99 samples can be entered. The sample data can also be entered while a determination is running *(see chapter 5.9.2, page 53)*.

Activating the sample table

Proceed as follows to activate the sample table (parameter **Sample table** = **on**).

1 Opening the main menu

- In the main dialog, select **Menu** and press **[OK]**.



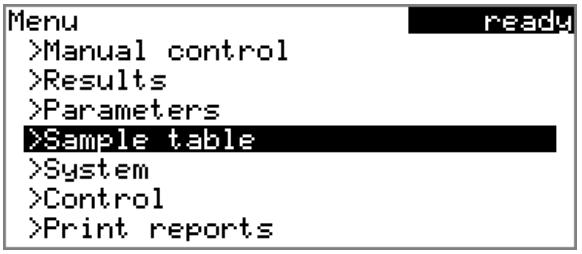
2 Opening the control dialog

- Select the menu item **Control** and press **[OK]**.

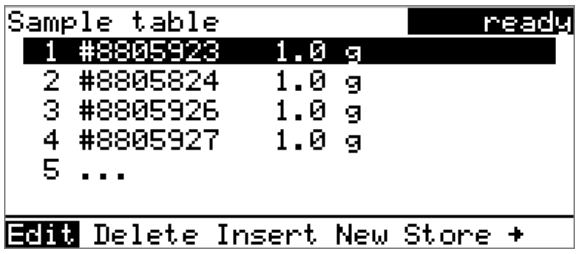
3 Activating the sample table

- Select **Sample table** and press **[OK]**.
- Select the entry **on** in the selection list and apply with **[OK]**.
- Press **[BACK]**.

The menu item **Sample table** is displayed in the main menu:



The sample table contains numbered lines. The identification (**ID1**) and the sample size of each sample are displayed.



Edit

Edit the data of the selected line, see following chapter.

Delete

Delete the selected line from the sample table.

Insert

Insert a new line above the line selected.

New

Delete the sample table completely. This function is visible only if the instrument is in **ready** status.

Store

Save the sample table. The sample table is saved in the internal device memory.

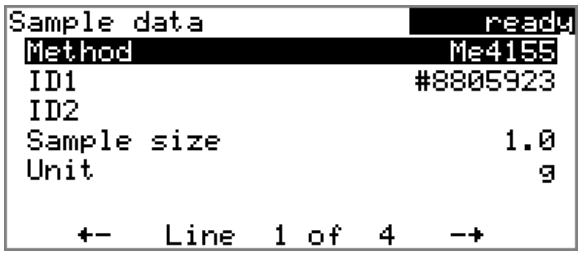
NOTICE

You can save only one sample table. When you save a sample table, any previously saved sample table will be overwritten automatically.

Load

Load the sample table from the internal device memory.

5.7.2 Editing the sample data



You will see at the very bottom the line number of the selected line and the line number of the last line containing data. In this example, the first line is opened and the sample table contains four lines.

One can scroll between the individual data sets with the keys **[⇦]** and **[⇨]**.

Inserting a new line

If you find yourself on the last line (i.e. **Line 4 of 4** in the above example), you can add a new line to the sample table by pressing **[⇨]** again. The sample data of the previous sample will be applied thereby.

Method

Method used for processing the sample.

Selection	**Selection of stored methods** | **empty**
Default value	**empty**

empty
The currently loaded method is used.

ID1

Sample identification. The sample identification can be used in calculations as the variable **CI1**.

Entry	**max. 10 characters**
Default value	**empty**

ID2

Sample identification. The sample identification can be used in calculations as the variable **CI2**.

Entry	**max. 10 characters**
Default value	**empty**

Sample size

Sample size. The value of the sample size can be used in calculations as the variable **C00**.

Input range	**–999999999 - 9999999999**
Default value	**1.0**

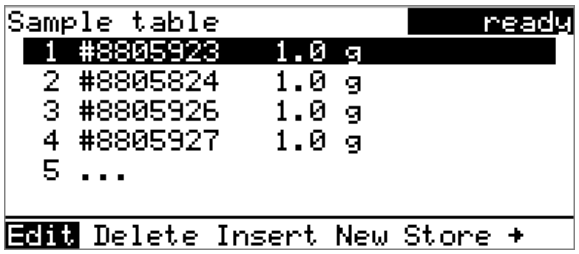
Unit

Unit of the sample size.

Selection	**g** | **mg** | **µg** | **mL** | **µL** | **pieces** | **User-defined**
Default value	**g**

User-defined
A user-defined unit can be created. This will be added to the selection list. The previous entry will be overwritten as soon as the new unit has been defined.

5.7.3 Sending the sample size from a balance



If the sample size is sent directly from the balance, then it will always be entered in a new line at the end of the sample table. It does not matter which line is selected or whether the sample table is even opened. In the above example, the sample size is entered in line 5.

NOTICE

If you would like to enter the sample size in a particular line, then you must open the corresponding editing dialog (i.e. the **Sample data** dialog is displayed).

If the editing dialog for the sample size is opened, then the sent value will be ignored.

5.8 Carrying out a determination

The sample size can be entered in the following ways when a determination is carried out:

- Enter manually on the instrument.
- Send automatically from a connected balance. For this purpose, check the manual for the balance.

Proceed as follows to carry out a determination:

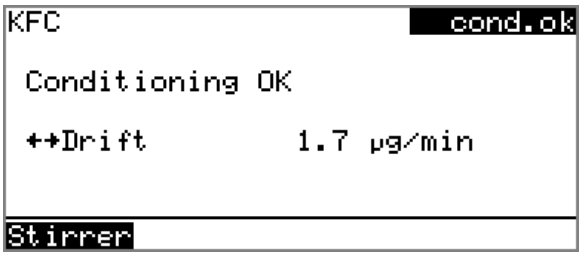
1 Loading the method

- *See Chapter "Methods", Page 40.*

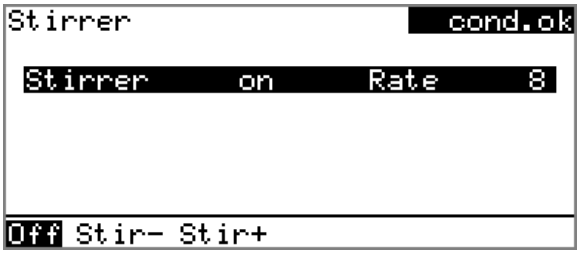
2 Starting conditioning

- Press **[START]**.

  Conditioning starts. **Conditioning not OK** is displayed until the endpoint is reached. The working medium is titrated to the endpoint. This is indicated by **Conditioning OK**. The status is kept stable.



The stirring rate can be modified with the **Stirrer** function. The following dialog is opened by pressing **[OK]**:



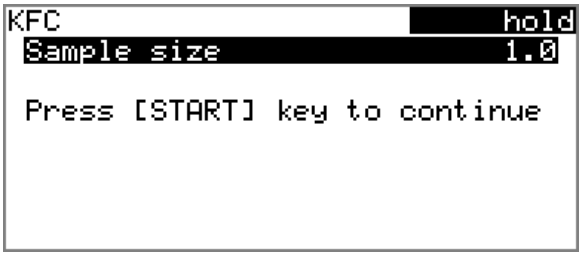
The stirring rate can be reduced with **Stir-** and increased with **Stir+**. **Off** switches the stirrer off. **On** is now displayed instead. This can be used to switch the stirrer back on. This dialog is exited with **[BACK]**.

3 Adding sample

- If **Conditioning OK** is displayed, press **[START]**.
  Conditioning is stopped. The request for adding the sample will be displayed for 8 s.
  The sample must be added during this time.

- Add the sample.

Afterward, the request for the sample size appears:



4 Entering the sample size

- Press **[OK]**.
  The editing dialog opens.
- Enter the sample size and apply with **Accept** or **[BACK]**.

5 Starting the titration

- Press **[START]**.

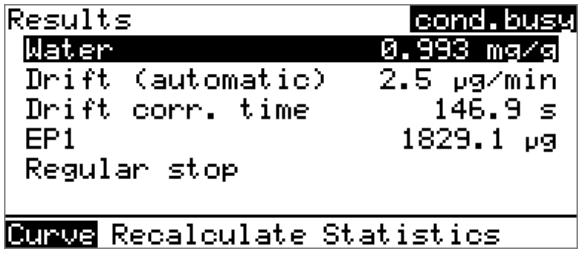
The titration starts and the curve is displayed:

The axes are scaled automatically.

The stirring rate can be modified during titration with the **Stirrer** function. The following dialog is opened by pressing **[OK]**:

The stirring rate can be reduced with **Stir-** and increased with **Stir+**. **Off** switches the stirrer off. **On** is now displayed instead. This can be used to switch the stirrer back on. This dialog is exited with **[BACK]**.

After the completion of the titration, the results dialog is displayed:



Conditioning is restarted automatically in the background. You can see the current status of the conditioning in the status display at the upper right in the dialog window (**cond.busy** or **cond.ok**).

6 Returning to the conditioning dialog

- Press **[BACK]**.
  The main dialog with the sample data of the previously ended titration is displayed.
- Select **Menu** and press **[OK]**.
- Select the menu item **Live dialog** and press **[OK]**.

The current status of the conditioning is displayed (see instruction step *2*).

If you wish to start the next titration, repeat the actions starting with instruction step *3*.

Canceling a determination manually

A determination can be canceled at any time with the **[STOP]** key.

5.9 Live modifications

5.9.1 Editing the sample data of the running determination

The sample data can be entered or modified in the main dialog while a determination is running. In calculations always the sample data entered at the end of the titration in the main dialog is used.

Proceed as follows to edit the sample data:

1 Displaying the main dialog

- Press **[BACK]**.

The main dialog is displayed. The determination continues to run in the background.

2 Editing the sample data

- Edit the sample data and apply with **Accept** or **[BACK]**.

3 Displaying the live dialog

- Press **[BACK]**.

or

- Select **Menu** and press **[OK]**.

- Select the menu item **Live dialog** and press **[OK]**.

The live dialog is displayed once again.

NOTICE

If the determination is finished while an editing dialog is opened (e.g. of the sample size), then this will be closed automatically and the results dialog will be displayed. The value entered must be entered once more and the determination must be recalculated.

Make sure that the editing dialogs are closed before the determination is finished.

5.9.2 Editing the sample table while a determination is running

You can insert new lines or delete existing ones or edit sample data while a determination is running.

NOTICE

We recommend that the editing dialogs always be closed in order to ensure that no problems occur during the run and that the current data is always available for calculation purposes.

Editing the sample table

Proceed as follows to edit the sample table:

1 Displaying the main dialog

- Press **[BACK]**.

The main dialog is displayed. The determination continues to run in the background.

2 Opening the main menu

- Select **Menu** and press **[OK]**.

3 Selecting the sample data

- Select the menu item **Sample table** and press **[OK]**.
- Select the desired line.
- In the function bar, select **Edit** and press **[OK]**.

4 Editing the sample data

- Edit the sample data and apply with **Accept** or **[BACK]**.

NOTICE

In addition to the sample data, the method can also be modified, except in cases where the determination is running.

5 Displaying the live dialog

- Select the menu item **Live dialog** in the main menu and press **[OK]**.

or

- Press **[BACK]** in the main dialog.

The live dialog is displayed once again.

Editing the sample data of the running determination

When you use the sample table, the editing of the sample data of the running determination proceeds as described in *chapter 5.9.1, page 52*. In addition, you have the option of editing these in the sample table. The first line always contains the sample data of the running determination. Simply select for this purpose the **Sample table***(see "Editing the sample table", page 53)* menu item in the main menu.

5.9.3 Editing the live parameters

Certain method parameters can be edited while a determination is being carried out. The only parameters that can be modified are those that can be selected. Nevertheless, all of the parameters are visible. The modified parameters are taken into account at once. If, for example, you increase the parameter **Extraction time** while the extraction time is still running, then the new value will be taken into account at once. If however you change this parameter after the extraction time has already expired, then the changed value will not be taken into account until the next determination.

Proceed as follows to edit the parameters:

1 Displaying the main dialog

- Press **[BACK]**.

The main dialog is displayed. The determination continues to run in the background.

2 Opening the main menu

- Select **Menu** and press **[OK]**.

3 Editing the method parameters

- Select the menu item **Parameters** and press **[OK]**.
- Change the desired parameters accordingly.

4 Displaying the live dialog

- Select the menu item **Live dialog** in the main menu and press **[OK]**.

or

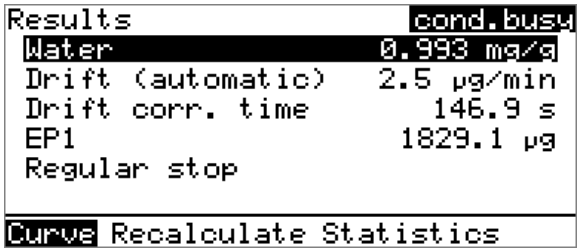
- Press **[BACK]** in the main dialog.

The live dialog is displayed once again.

5.10 Results

Menu ▸ Results

After the completion of the titration, the results dialog is displayed:



The calculated result and details concerning the drift and the endpoint are shown in the overview.

Curve

Display the curve of the current determination.

Recalculate

Recalculate the current determination. The procedure will be executed immediately.

Statistics

Display the statistical overview of a determination series *(see chapter 5.11, page 57)*.

Displaying the curve

The curve of the current determination can be displayed with the **Curve** function.

The arrow keys **[⇦]** and **[⇨]** can be used to move to the individual measuring points. A cross hair is used to show the current position on the curve. The data (water quantity, measured value, time, etc.) for the respective measuring point is indicated on the right-hand side.

Recalculating

NOTICE

Recalculation cannot be undone.

All of the results are recalculated with the **Recalculate** function. This is necessary if, for example, the calculation or the sample size has been modified.

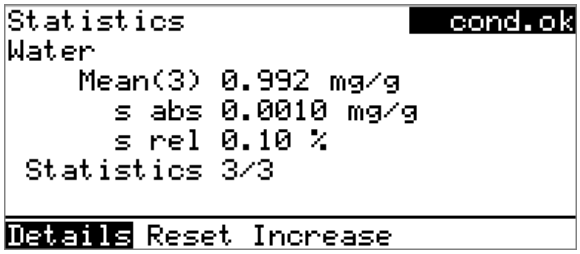
5.11 Statistics

Menu ▸ Results ▸ Statistics

The statistical overview of a determination series can be displayed in the **Results** dialog with the **Statistics** function.

NOTICE

This function is visible only if statistics has been activated.



The mean value (**Mean**), the absolute and the relative standard deviation (**s abs** and **s rel**) are displayed in the overview. For the mean value, the number of individual results from which it has been calculated is displayed in parentheses. In this example, it is 3. The **Statistics** line shows how many determinations have already been carried out and how many determinations are to be carried out in total. All three determinations were carried out in this example.

Details

Display additional data.

Reset

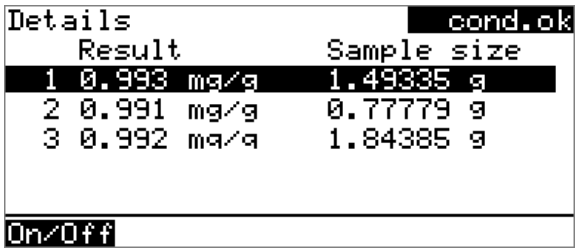
Delete all statistics data.

Increase

Add a further determination to the determination series.

Displaying statistical details

Additional data from the determination series can be displayed with the **Details** function.



The result and the sample size of each determination are shown.

On/Off

Remove the selected determination from the statistics. The line will then be marked with an asterisk (*), the statistics will be recalculated automatically. If several calculations are defined in the method, then all the results will be removed from the statistics.

Deleting statistical data

All statistical data is deleted with the **Reset** function. The statistics data is deleted automatically in the following cases:

- When all of the determinations of the determination series have been carried out and a new determination has been started afterwards.
- When a new method is loaded.

Adding a determination to a determination series

You can use the function **Increase** to add an additional sample to a determination series, e.g. because a determination was faulty and had to be removed from the statistics. The second number in the **Statistics** line will be increased automatically by one.

5.12 Printing a report manually

Menu ▸ Print reports

Proceed as follows to print a report manually:

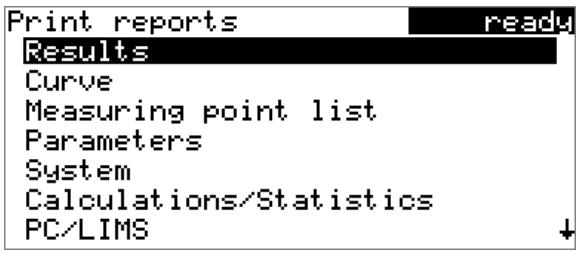
1 Opening the main menu

- In the main dialog, select **Menu** and press **[OK]**.

2 Opening the print dialog

- Select the menu item **Print reports** and press **[OK]**.

The dialog window with the available reports opens:



3 Selecting the report

- Select the desired report and press **[OK]**.

The report is printed out.

The following reports can be printed out manually:

Results	Result report with determination properties, sample data, calculated results, etc.
Curve	Curve report. The width of the curve is defined in the system settings *(see "Graphics width", page 68)*.
Measuring point list	Measuring point list report.
Parameters	Report with all method parameters of the loaded method.

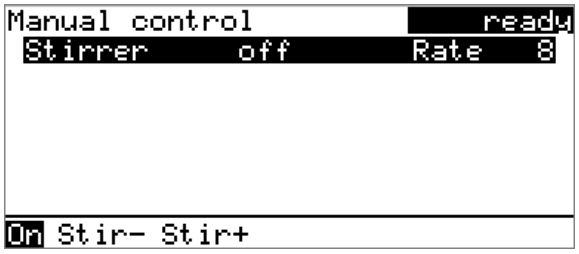
System	System report with system settings, external devices, etc.
Calculations/Statistics	Calculation report. The statistics are also printed out in the case of multiple determinations. The individual determinations with the respective sample size, the mean value, the absolute and the relative standard deviation are printed out for each result.
PC/LIMS	Machine-readable report with all of the data for a determination. This report can be saved as a TXT file to a connected USB flash drive or sent to a terminal program or a LIMS via an RS-232 interface. The definition is made in the system settings *(see "PC/LIMS report", page 68)*.
Report as in method	The reports that are defined in the method will be printed out.

5.13 Manual control

Menu ▸ Manual control

The following function is available in the manual control:

- Stirring



The available subfunctions are listed in the function bar.

5.13.1 Stirring

Stirrers that are connected or installed in the Coulometer can be controlled manually.

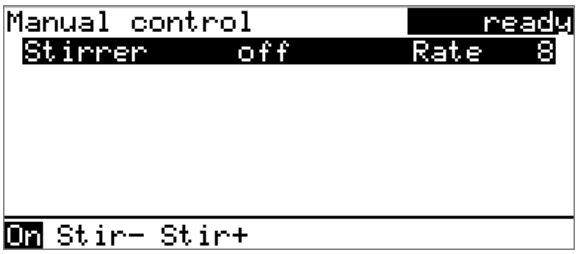
Proceed as follows:

1 Opening the manual control

- In the main dialog, select **Menu** and press **[OK]**.
  The main menu opens.
- Select the menu item **Manual control** and press **[OK]**.

Manual control opens.

2 Setting the stirring rate



- In the function bar, select **Stir-** or **Stir+**.
  The stirring rate will be increased or decreased by one step each time the **[OK]** key is pressed.
  The algebraic sign changes the direction in which the stirring is done. When viewing the stirrer from above, this means:
  - "+": counterclockwise rotation
  - "–": clockwise rotation

3 Switching on the stirrer

- In the function bar, select **On** and press **[OK]**.

The stirrer is started and stirs at the rate which has been set. **Off** is now displayed in the function bar.

4 Switching off the stirrer

- In the function bar, select **Off** and confirm with **[OK]**.

The stirrer is stopped.

6 System settings

6.1 Basic settings

Menu ▸ System ▸ Settings

This chapter contains a description of general instrument settings.

User name

A user name can be entered here for the report. This parameter will only be printed if a user has been defined.

Entry	**max. 12 characters**
Default value	**empty**

Instrument name

A instrument name can be entered here for the report. This parameter will only be printed if a designation has been defined.

Entry	**max. 10 characters**
Default value	**empty**

Serial number

Serial number of the instrument. This is printed as a part of the instrument identification in the report header.

Program version

Version number of the instrument software. This is printed as a part of the instrument identification in the report header.

Time

Current time. Only valid numbers can be entered.

Format: hh:mm:ss

Date

Current date. Only valid numbers can be entered.

Format: YYYY:MM:DD

Language

Setting the dialog language. An additional language can be selected besides English.

NOTICE

In order to ensure that a second language can be selected, it must first be installed. This installation must be carried out by specialist personnel. In chapter *Language files, page 71*, you will find details regarding the installation of a second language.

Dialog type

The user dialog can be limited for routine operations. One can operate normally with methods in the limited dialog. However, no settings can be made or methods deleted.

The resetting of the dialog will take effect as soon as you exit the main menu.

The limitation of the dialog results in the following:

- The menu items **System**, **Parameters** and **Control** are not shown in the main menu.
- Methods can only be loaded, but not deleted, exported or created.

NOTICE

If the limited dialog for routine operation is activated, then the expert dialog cannot be activated during ongoing operation. To change the dialog type, the 899 Coulometer must be switched off and then back on again. The expert dialog can be forced as soon as the instrument is started up again. Then it is possible to enter whatever settings one wishes, e.g. the changing of the dialog type. If the instrument is switched off again without changing the dialog type, then the routine dialog will remain activated.

Forcing the expert dialog:

- Switch on the instrument.
- Wait for the display of the instrument logo with the lettering **easy, safe, precise**.
- Press the **[STOP]** key once again and hold it down while also briefly pressing the **[BACK]** key.
- Release both keys once again.

Selection	**Expert** | **Routine**
Default value	**Expert**

Expert
Complete dialog.

Routine
Limited dialog for routine operations.

Contrast

The contrast of the display can be adjusted with the arrow keys **[⇦]** and **[⇨]**.

- **[⇦]**: the contrast will be decreased by one step each time the key is pressed.
- **[⇨]**: the contrast will be increased by one step each time the key is pressed.

Input range	**150 - 240**
Default value	**212**

NOTICE

Alternatively, the contrast can also be modified in the following manner:

Keep the red **[STOP]** key pressed down. As soon as the progress bar appears, also press the arrow key **[⇩]** or **[⇧]** repeatedly.

This method will, however, cause the contrast to be modified by several steps.

Beep

If this parameter is activated, then a short beep will sound in the following cases:

- When a key is pressed.
- At the end of the determination.
- When the system remains conditioned without interruption for 10 seconds.

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

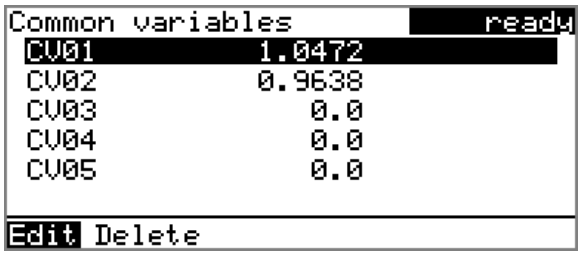
6.2 Managing common variables

6.2.1 General

Menu ▸ System ▸ Common variables

The instrument offers the possibility of saving five **method-independent variables**, so-called common variables. These variables remain saved in the instrument and can be used in future calculations. A common variable is useful, e.g. for the following applications:

- Determination of a blank value which will be taken into account during the content determination of the sample.



The common variables have the non-changeable designation **CV01… CV05**. The value is displayed for every variable. No unit can be assigned to the common variables.

Edit

See the following chapter for editing the data of the selected common variable.

Delete

Set the selected common variable to **invalid**.

6.2.2 Editing common variables

The common variables can be modified as follows:

- Manually in this dialog.
- Automatic assignment from the determination run. A calculation result must be configured accordingly for this purpose (see below).

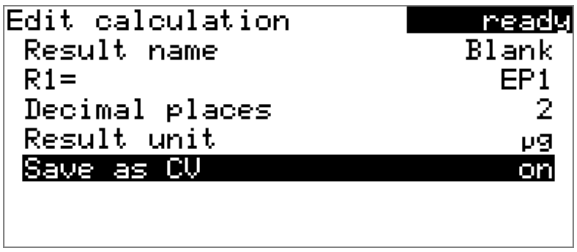
Assigning a result automatically to a common variable

Proceed as follows:

1 Opening the editing dialog of the result

- Select the menu item **Parameters ▸ Calculation** and press **[OK]**.

- Select the result whose value is to be assigned to a common variable.
- In the function bar, select **Edit** and press **[OK]**.



2 Adjusting the result properties

- Select the parameter **Save as CV** and press **[OK]**.
- Select the entry **on** in the selection list and apply with **[OK]**.

The assignment of the result to a common variable occurs automatically according to the following scheme:

- Result **R1** ⇨ Common Variable **CV01**
- Result **R2** ⇨ Common Variable **CV02**
- etc.

NOTICE

If you have set the parameter **Statistics** to **on**, then the mean value of the results will be assigned to the respective common variable.

6.3 File management

Menu ▸ System ▸ File management

NOTICE

This menu item is visible only if a USB flash drive has been connected as an external storage medium.

Methods can be imported and deleted from a USB flash drive in this dialog. Only methods located in the **Files** directory are displayed in the list *(see "Directory structure on the USB flash drive", page 67)*.

A backup of the system can be created (all data and settings). Similarly, an existing backup can be reloaded.

Import

Import the selected method.

Delete

Delete the selected method.

Backup

Create a backup of all data and settings on the USB flash drive.

NOTICE

Only **one** backup can be created on the same USB flash drive.

If a backup is already stored on the flash drive, then this will be overwritten as soon as the function is performed once again.

Restore

Load the backup from a connected USB flash drive.

Directory structure on the USB flash drive

A directory with the instrument number will be created on the USB flash drive. The structure within this directory appears as follows:

Figure 24 Directory structure on the USB flash drive

Backup — All of the files of the backup are stored in this directory. The directory is created as soon as a backup is created for the first time.

Files — Exported methods are stored in this directory. The directory is created as soon as a backup is exported for the first time.

Only methods located in this directory can be imported.

pc_lims_report — PC/LIMS reports are stored in this directory as TXT files. The directory is created as soon as a PC/LIMS report is printed for the first time.

6.4 Configuring external devices

Menu ▸ System ▸ External devices

PC/LIMS report

Specification of the storage location for the PC/LIMS report. The PC/LIMS report is a machine-readable report with all of the important data for a determination. It can be saved as follows:

- as a TXT file on a USB flash drive.
- to a LIMS via an RS-232 interface. The 6.2148.030 RS-232/USB Box is required for this purpose.

Selection	**COM2** | **USB Stick**
Default value	**USB Stick**

COM2
The report is sent via the serial COM2 interface. The interface parameters set in the dialog **COM2 settings** are used *(see "Editing the COM2 settings", page 70)*.

USB Stick
The report will be saved as a TXT file on the USB flash drive in the folder **pc_lims_report**.

Printer

If a printer is connected, then the printer type needs to be defined here in order for the reports to be printed out correctly.

The printers that have the designation **ESC-POS** are so-called POS printers (point-of-sale printers), i.e. they print on continuous paper.

Selection	**Citizen (ESC-POS)** | **Custom (ESC-POS)** | **Epson** | **Epson (ESC-POS)** | **HP DeskJet** | **HP LaserJet** | **Seiko (ESC-POS)**
Default value	**Custom (ESC-POS)**

Graphics width

Adjust the width of the curve to be printed out to the paper width of the printer to be used. The default value depends on the selected printer. The height of the curve is 2/3 of the width.

Input range	**100 - 3000 Pixels**

Keyboard layout

A commercially available USB keyboard can be connected to make it easier to enter text and numbers. Specify the country-specific keyboard layout.

Selection	**English US** | **French FR** | **German CH** | **German DE** | **Spanish ES**
Default value	**English US**

Balance

If you have connected a balance, then you must define the balance type here.

Selection	**AND** | **Mettler** | **Mettler AT** | **Mettler AX** | **Ohaus** | **Precisa** | **Sartorius** | **Shimadzu**
Default value	**Sartorius**

The following table indicates the balance type that needs to be selected for the balance model:

Balance	Balance type
AND	**AND**
Mettler AB, AE, AG, AM, AJ, PE, PM, PJ, PR, XP, XS	**Mettler**
Mettler AT	**Mettler AT**
Mettler AX, MX, UMX, PG, AB-S, PB-S	**Mettler AX**
Ohaus Voyager, Explorer, Analytical Plus	**Ohaus**
Precisa	**Precisa**
Sartorius	**Sartorius**
Shimadzu BX, BW	**Shimadzu**

Editing the COM1 settings

Menu ▸ System ▸ External devices ▸ COM1 settings

The interface parameters for the connected balance are set under **COM1 settings**.

Baud rate

Transfer rate in characters per second.

Selection	**1200** | **2400** | **4800** | **9600** | **19200** | **38400** | **57600** | **115200**
Default value	**9600**

Data bits

Number of data bits.

Selection	**7** | **8**
Default value	**8**

Stop bits

Number of stop bits.

Selection	**1** | **2**
Default value	**1**

Parity

Type of parity testing.

Selection	**even** | **none** | **odd**
Default value	**none**

Handshake

Type of the data transfer protocol.

Selection	**hardware** | **software** | **none**
Default value	**hardware**

NOTICE

If communications problems occur, set the parameter **Handshake** to **software**, and make another attempt.

Editing the COM2 settings

Menu ▸ System ▸ External devices ▸ COM2 settings

The interface parameters for instruments connected to the **RS-232/2** connector of the RS-232/USB Box (e.g. PC) are set under **COM2 settings**. The parameters and input ranges are identical to those for the COM1 interface.

6.5 Instrument diagnosis

6.5.1 Loading program versions and language files

Menu ▸ System ▸ Diagnosis

New program versions or language files can be loaded from a USB flash drive. The corresponding file must be saved on the USB flash drive in a directory with the instrument number (e.g. 899).

You can distinguish between language files and program files by noting how the file name is constructed.

Program files

They are instrument-specific. The file name has the following structure:

5XXXyyyy.bin where

XXX = Instrument type (e.g. 899 for the 899 Coulometer)

yyyy = Program version

Language files

They can be recognized by means of the two-digit language code in the file name. A language file contains the dialog texts for various instrument types. It is not instrument-specific. The file name has the following structure:

5848xxxxYY.bin where

xxxx = Version number

YY = Language, e.g. DE (German), FR (French), ES (Spanish)

Loading a file

Proceed as follows:

1 Connecting the USB flash drive

- Plug in the USB flash drive with the 6.2151.100 adapter (USB MINI (OTG) - USB A) at the instrument's USB port.
- Switch on the instrument.

2 Opening the update dialog

- Under **Menu ▸ System ▸ Diagnosis**, select the menu item **Software update**.
- Press **[OK]**.

3 Opening the file selection

- Press **[OK]**.

The selection list with the program and language files present on the USB flash drive opens.

4 Selecting the file

- Use the arrow keys to select the required file.
- Press **[OK]**.

5 Starting the update

- Press **[START]**.

The update process is started, it runs automatically. At the end of the process, the instrument will be switched off automatically and switched back on again. No user intervention is required.

6.5.2 Diagnosis functions

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 representative regarding the precise terms and conditions involved in concluding a corresponding maintenance agreement.

7 Parameters

7.1 Coulometric Karl Fischer titrations (KFC)

7.1.1 Conditioning

Menu ▸ Parameters ▸ Conditioning

Under **Conditioning**, the conditions required for conditioning are defined.

Conditioning

If this parameter is activated, then the first time the titration is started the working medium will be titrated to the endpoint with the specified control parameters. The status is kept stable. The actual method run does not begin until **[START]** has been pressed once more. Conditioning will be carried out again automatically after the titration.

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

Start drift

If the measured drift is smaller than this value for a certain time (so-called stabilizing time), then **Conditioning OK** will be displayed and the titration can be started. The stabilizing time is defined under **Conditioning ▸ Stabilizing time**.

Input range	**1 - 999 µg/min**
Default value	**20 µg/min**

Drift correction

The end point amount can be drift-corrected. To accomplish this, the drift is multiplied by the drift correction time and this value is subsequently subtracted from the amount of the end point. The drift correction time is the time interval between the end of conditioning and the end of the determination.

Selection	**auto** | **manual** | **off**
Default value	**auto**

auto
The value of the current drift is automatically applied at the start of the titration.

manual
If the drift is known throughout a longer period of time, this can be entered manually.

off
No drift correction takes place.

Drift value

This parameter can only be edited with **Drift correction** = **manual**.

Drift for manual drift correction.

Input range	**0.0 - 99.9 µg/min**
Default value	**0.0 µg/min**

Automatic start

If this parameter is activated, then the determination is automatically started when the measured voltage suddenly changes. This setting will be ignored as long as the working medium has not been conditioned.

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

NOTICE

If you use the automatic start function, then we recommend that the start drift be reduced and that a stabilizing time be defined.

Recommendation: Start drift ≤ 10 µg/min / Stabilizing time ≥ 30 s

Threshold value

This parameter can only be edited with **Automatic start** = **on**.

If the change in voltage is higher than this value, then the determination will automatically be started.

Input range	**0 - 999 mV**
Default value	**50 mV**

Stabilizing time

Waiting time, during which the measured drift has to be smaller than the start drift defined until **Conditioning OK** is displayed. The start drift is defined under **Conditioning ▸ Start drift**.

Input range	**0 - 999999 s**
Default value	**0 s**

Cond. stop time

Maximum permissible time over which conditioning may take place. Conditioning is stopped as soon as the specified time has elapsed.

Input range	**1 - 999999 s**

Selection	**off**
Default value	**off**

Measured value display

If this parameter is activated, then the currently measured value is displayed during the conditioning.

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

7.1.2 Start conditions

Menu ▸ Parameters ▸ Start conditions

The parameters that are carried out before the start of titration are defined under **Start conditions**.

Pause

Waiting time, for example for dissolving the sample. During this time no iodine is generated.

Input range	**0 - 999999 s**
Default value	**0 s**

Request sample ID

Selection of the sample identification that is queried in the method run.

Selection	**ID1** | **ID2** | **ID1&ID2** | **off**
Default value	**off**

Request sample size

If this parameter is activated, then the value for the sample size will be requested.

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

Request sample unit

If this parameter is activated, then the unit for the sample size will be requested.

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

Hold at request

If this parameter is activated, then the run will be paused during the request. If the parameter is deactivated, then the titration will be started in the background.

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

7.1.3 Control parameters

Menu ▸ Parameters ▸ Control parameters

The control parameters for the endpoint are defined under **Control parameters**.

Endpoint at

Measured value for the endpoint.

Input range	**–1250.0 - 1250.0 mV**
Default value	**50.0 mV**
Selection	**off**

NOTICE

Note that the parameter **Endpoint at** is linked with the parameters **Dynamics** and **I(pol)**. Explanations in this connection can be found in chapter *9.4 Control parameters and polarization current, page 95*.

Titration rate

Three predefined sets of parameters can be selected for the titration rate.

Selection	**slow** | **optimal** | **fast** | **user**
Default value	**optimal**

slow
For samples with a low water content or samples which release their moisture only slowly.

optimal
For all standard titrations. The parameters have been optimized for the most frequent applications.

fast
For uncritical samples with high water content.

user
The individual titration parameters can be modified.

The settings of the individual titration rates are listed in *table 3, page 77*.

Dynamics

This parameter can only be edited with **Titration rate** = **user**.

This parameter defines the control range before the specified endpoint. Within the control range, the iodine is generated step by step, the genera-

tion is finely controlled. The closer the endpoint, the slower the iodine is generated until the rate defined under **Min. rate** is reached. The larger the control range, the slower the titration. Outside the control range, iodine is being continuously generated, the rate is defined under **Max. rate**.

Input range	**0.1 - 1250.0 mV**
Default value	**70.0 mV**
Selection	**off**

off
The generation of iodine is finely controlled during the entire titration.

NOTICE

Note that the parameter **Dynamics** is linked with the parameters **End-point at** and **I(pol)**. Explanations in this connection can be found in chapter *9.4 Control parameters and polarization current, page 95*.

Max. rate

This parameter can only be edited with **Titration rate** = **user**.

Rate at which iodine is generated outside the control range.

Input range	**1.5 - 2241.0 µg/min**
Selection	**max.**
Default value	**max.**

Min. rate

This parameter can only be edited with **Titration rate** = **user**.

Rate at which iodine is generated at the very beginning of the titration and in the control range at the end of the titration. This parameter has a decisive influence on the titration rate and thus also on the accuracy. The smaller the selected minimum rate, the slower the titration.

Input range	**0.3 - 999.9 µg/min**
Default value	**15.0 µg/min**

Table 3 Default values of the predefined titration rates for KFC

	Titration rate		
	slow	**optimal**	**fast**
Dynamics	120.0 mV	70.0 mV	30.0 mV
Max. rate	1000.0 µg/min	maximum	maximum
Min. rate	0.3 µg/min	15.0 µg/min	30.0 µg/min

Stop criterion

The titration is canceled when the endpoint has been reached and this stop criterion has been fulfilled. If no stop criterion has been selected then the titration will not be canceled. The stop conditions *(see chapter 7.1.5, page 81)* always lead to a stop, even if the stop criterion has not been reached.

Selection	**drift** | **time** | **rel. drift** | **off**
Default value	**rel. drift**

drift
The titration is canceled when the stop drift has been reached.

time
The titration is canceled if the endpoint has been exceeded during a certain time period (**Delay time**).

rel. drift
The titration is canceled when the sum of the drift at the start of the titration and the relative stop drift has been reached.

off
The titration will not be canceled until the stop conditions have been fulfilled.

Stop drift

This parameter can only be edited with **Stop criterion** = **drift**.

The titration is canceled when the endpoint and the stop drift have been reached.

Input range	**1 - 999 µg/min**
Default value	**5 µg/min**

Delay time

This parameter can only be edited with **Stop criterion** = **time**.

The titration will be canceled if the following criteria are both met simultaneously.

- The voltage at the indicator electrode lies in the range "endpoint +5 mV and endpoint –2 mV".
- The time defined here has expired.

If, for example, the parameter **Endpoint at** is set to 50 mV and the parameter **Delay time** defined here is 10 s, then the voltage at the indicator electrode must be between 55 mV and 48 mV for 10 seconds so that the titration would be canceled.

Input range	**0 - 999 s**
Default value	**10 s**

Relative stop drift

This parameter can only be edited with **Stop criterion** = **rel. drift**.

The titration is canceled when the endpoint and the sum of the drift at the start of the titration and the relative stop drift have been reached.

Input range	**1 - 999 µg/min**
Default value	**5 µg/min**

7.1.4 Titration parameters

Menu ▸ Parameters ▸ Titration parameters

Under **Titration parameters**, the parameters influencing the sequence of the entire titration are defined.

Extraction time

Minimum duration of the titration. The titration will not be canceled during the extraction time, even if the endpoint has already been reached. The titration is however canceled if a stop condition is fulfilled during this time *(see chapter 7.1.5, page 81)*. The entering of an extraction time is e.g. recommended with samples that release water slowly or if a Karl Fischer oven is used.

Input range	**0 - 999999 s**
Default value	**0 s**

Generator electr.

Type of generator electrode.

Selection	**with diaphr.** | **w/o diaphr.**
Default value	**w/o diaphr.**

with diaphr.
Generator electrode with diaphragm.

w/o diaphr.
Generator electrode without diaphragm.

Generator current

Polarization current at the generator electrode.

Selection	**100 mA** | **200 mA** | **400 mA** | **auto**
Default value	**400 mA**

400 mA
default value, when **Generator electr.** = **w/o diaphr.**

auto
The current is adapted to the conductivity of the reagent and automatically reduced near the endpoint. Default value, when **Generator electr.** = **with diaphr.**

Stirrer

The stirrer is switched on at the start of the determination when this parameter is activated.

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

Stirring rate

Setting the stirring rate. It can be set in steps of –15 to +15. The default setting **8** corresponds to 1000 rpm. The formula for calculating the rotational speed is specified in *chapter 9.1, page 92*. The optimum stirring rate can be tested in the manual control.

The algebraic sign changes the direction in which the stirring is done. When viewing the stirrer from above, this means:

- "+": counterclockwise rotation
- "–": clockwise rotation

Input range	**–15 - 15**
Default value	**8**

I(pol)

The polarization current is the current that is applied to a polarizable electrode during the voltametric measurement.

Selection	**5 µA** | **10 µA** | **20 µA** | **30 µA**
Default value	**10 µA**

NOTICE

Note that the parameter **I(pol)** is linked with the parameters **Dynamics** and **Endpoint at**. Explanations in this connection can be found in chapter *9.4 Control parameters and polarization current, page 95*.

Electrode test

In the case of polarizable electrodes, an electrode test can be carried out. A check is made that the electrode is properly connected and that no short-circuit is present. The electrode test is carried out as soon as the determination is started.

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

Time interval MP

Time interval for entering a measuring point in the measuring point list. The measuring point list is limited to 1000 measuring points.

Input range	**0.1 - 999999.0 s**
Default value	**2.0 s**

Temperature

Temperature entered manually.

Input range	**–20.0 - 150.0 °C**
Default value	**25.0 °C**

7.1.5 Stop conditions

Menu ▸ Parameters ▸ Stop conditions

The conditions for stopping a titration are defined under **Stop conditions**, if this does not occur automatically. This could be the case when the endpoint set is not reached or if the stop criterion *(see "Stop criterion", page 78)* is not fulfilled.

Stop time

The titration is canceled when the specified time has elapsed since the start of the titration.

Input range	**1 - 999999 s**
Selection	**off**
Default value	**off**

7.1.6 Calculation

7.1.6.1 General

Menu ▸ Parameters ▸ Calculation

A maximum of five calculations can be defined in one method. A series of variables (raw data from the determination, previously calculated results) is available for the calculations. Definition can be made for each calculation as to whether the result is to be saved as a common variable.

The result name is specified in the list for each calculation.

The method templates **KFC**, **KFC-Blank** and **Blank** each contains one calculation. Each of these calculations already bears an appropriate result name. You can modify these as needed.

Edit

See the following chapter for editing the data of the selected calculation.

Delete

Delete the selected calculation.

7.1.6.2 Editing calculations

Method "KFC"

Result name

The result name is the text which will be shown in the results display and in the report.

Entry	**12 characters**
Default value	**R1: Water / R2-R5: empty**

Calculation formula "R1"

EP1/C00

R2…R5

Display of the calculation formula. A special editor is opened for the definition *(see chapter 5.3, page 39)*.

Entry	**30 characters**
Default value	**empty**

Decimal places

Number of decimal places used to display the result.

Input range	**0 - 5**
Default value	**R1: 1 / R2-R5: 2**

Result unit

The result unit is displayed and saved together with the result.

R1	
Selection	**%** | **mg** | **mg/piece** | **mg/g** | **mg/mL** | **ppm** | **µg** | **User-defined**
Default value	**ppm**

R2-R5	
Selection	**%** | **mg** | **mg/piece** | **mg/g** | **mg/mL** | **ppm** | **µg** | **User-defined**
Default value	**%**

User-defined
A user-defined unit can be created. This will be added to the selection list. The previous entry will be overwritten as soon as the new unit has been defined. A blank entry can be generated this way as well.

Save as CV

The calculated result can be saved as a method-independent variable, a so-called common variable. The result is then also available in other methods for calculations. If Statistics has been activated, then the current mean value of the determination series will be saved.

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

Method "KFC-Blank"

Result name

The result name is the text which will be shown in the results display and in the report.

Entry	**12 characters**
Default value	**R1: Water / R2-R5: empty**

Calculation formula "R1"

(EP1–CV01)/C00

CV01 is the Common Variable 01 and corresponds to the result from the calculation of the *Method "Blank", page 84*.

R2…R5

Display of the calculation formula. A special editor is opened for the definition *(see chapter 5.3, page 39)*.

Entry	**30 characters**
Default value	**empty**

Decimal places

Number of decimal places used to display the result.

Input range	**0 - 5**
Default value	**R1: 1 / R2-R5: 2**

Result unit

The result unit is displayed and saved together with the result.

R1

Selection	**%** | **mg** | **mg/piece** | **mg/g** | **mg/mL** | **ppm** | **µg** | **User-defined**
Default value	**ppm**

R2-R5

Selection	**%** | **mg** | **mg/piece** | **mg/g** | **mg/mL** | **ppm** | **µg** | **User-defined**
Default value	**%**

User-defined
A user-defined unit can be created. This will be added to the selection list. The previous entry will be overwritten as soon as the new unit has been defined. A blank entry can be generated this way as well.

Save as CV

The calculated result can be saved as a method-independent variable, a so-called common variable. The result is then also available in other methods for calculations. If Statistics has been activated, then the current mean value of the determination series will be saved.

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

Method "Blank"

Result name

The result name is the text which will be shown in the results display and in the report.

Entry	**12 characters**
Default value	**R1: Blank / R2-R5: empty**

Calculation formula "R1"

EP1

R2...R5

Display of the calculation formula. A special editor is opened for the definition *(see chapter 5.3, page 39)*.

Entry	**30 characters**
Default value	**empty**

Decimal places

Number of decimal places used to display the result.

Input range	**0 - 5**
Default value	**2**

Result unit

The result unit is displayed and saved together with the result.

R1	
Selection	**%** | **mg** | **mg/piece** | **mg/g** | **mg/mL** | **ppm** | **µg** | **User-defined**
Default value	**µg**

R2-R5	
Selection	**%** | **mg** | **mg/piece** | **mg/g** | **mg/mL** | **ppm** | **µg** | **User-defined**
Default value	**%**

User-defined
A user-defined unit can be created. This will be added to the selection list. The previous entry will be overwritten as soon as the new unit has been defined. A blank entry can be generated this way as well.

Save as CV

The calculated result can be saved as a method-independent variable, a so-called common variable. The result is then also available in other methods for calculations. If Statistics has been activated, then the current mean value of the determination series will be saved.

R1	
Selection	**on** | **off**
Default value	**on**

R2-R5	
Selection	**on** | **off**
Default value	**off**

7.1.7 Statistics

Menu ▸ Parameters ▸ Statistics

The statistics calculation of a multiple determination is activated under **Statistics** and definition is made as to how many determinations the series contains.

Statistics

If this function is activated, then statistics calculations will be carried out for all of the defined results.

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

Number of determinations

The number of determinations that are carried out for the statistics calculations.

If an additional determination has to be added to the determination series, because one determination has been incorrect, for example, then this can be accomplished in the statistical overview *(see chapter 5.11, page 57)*.

Input range	**2 - 20**
Default value	**3**

7.1.8 Reports

Menu ▸ Parameters ▸ Reports

The reports that will be printed out automatically in connection with a determination are defined under **Reports**.

Results

The result report contains the calculated results, endpoints, sample data, etc.

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

Curve

Curve report. The width of the curve is defined in the system settings *(see "Graphics width", page 68)*.

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

Calculations/Statistics

Output of the calculation formulas for the individual results. Results are specified with full accuracy. This makes recalculation with an external program possible. If Statistics has been activated, then the following data will be printed out as well:

- Result and sample size of the individual determinations
- Mean value as well as absolute and relative standard deviation

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

Measuring point list

Output of the measuring point list.

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

Parameters

All of the parameters of the current method are printed out in the parameter report.

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

PC/LIMS

The PC/LIMS report is a machine-readable report with all of the data important for a determination. The PC/LIMS report can be saved as a TXT file on a USB storage medium or sent via an RS-232 interface to a LIMS. The output location is defined in the system settings *(see "PC/LIMS report", page 68)*.

The file name of the TXT file is constructed as follows: *PC_LIMS_Report-ID1-YYYYMMDD-hhmmss.txt*.

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

8 Troubleshooting

8.1 Karl Fischer titration

Problem	Cause	Remedy
The drift is very high during conditioning.	*The titration cell is leaking.*	▪ Check the septum and replace it if necessary. ▪ Replace the molecular sieve. ▪ Check the ground-joint sleeves for frayed edges. If necessary, trim the edges neatly or replace the ground-joint sleeves.
	Deposits containing water can be found in the titration cell.	▪ Shake the titration cell.
	The reagent is exhausted or contaminated.	▪ Replace the reagent.
	The catholyte is old or moist.	▪ The catholyte must be replaced at least once per week. ▪ Dry the catholyte with KF one-component reagent.
	The diaphragm of the generator electrode is contaminated.	▪ Clean the diaphragm (in accordance with directions in the leaflet for the generator electrode).
	A side reaction is taking place.	▪ Consider using a KF oven. ▪ See technical literature.
	The molecular sieve on the KF oven has reached its capacity limit.	▪ Replace the molecular sieve.
	The gas flow from the KF oven into the titration cell is too high.	▪ Reduce the gas flow (if you are working with the oven method with sample vials, set the gas flow between 40 and 60 mL/min).
The drift becomes greater after each titration.	*The sample releases water very slowly.*	▪ Consider using a KF oven. ▪ See technical literature.

Problem	Cause	Remedy
The drift is fluctuating.	*The sample solution is poorly stirred.*	▪ Set the stirring rate in such a way that the sample solution will become very well mixed.
	*The **Control parameters** are set incorrectly.*	▪ Reset the **Control parameters** to default values.
The titration time is too long.	*The drift during conditioning is not stable.*	▪ Wait before starting the titration until the drift has become stable.
	*The parameter **Start drift** is set too high.*	▪ Set the **Start drift** lower. ▪ Adjust the **Stop drift** or use the **Relative stop drift**.
	*The parameter **Stop drift** is set too low.*	▪ Set the **Stop drift** higher or use the **Relative stop drift**.
	The water quantity is too high.	▪ Observe the sample size and water contents in accordance with *Table "Recommended sample sizes", page 31*.
	*The **Control parameters** are set incorrectly.*	▪ Set the parameter **Dynamics** lower and the parameter **Max. rate** higher.
The result is too high.	*The titration cell is not yet correctly conditioned.*	▪ Shake the titration cell and wait until the drift is stable.
	The sample contains oxidizable substances.	▪ Consider using a KF oven. ▪ See technical literature.
	*The parameter **Stop drift** is set too low.*	▪ Set the **Stop drift** higher or use the **Relative stop drift**.
	The drift correction is too small (e.g. for the use of a KF oven or for manual drift correction).	▪ Start the titration as long as the gas flow still exists between the conditioning vial and the titration cell. ▪ Set the parameter **Drift correction** to **auto** (= automatic drift correction).
The result is too low.	*The sample releases iodine.*	▪ Consider using a KF oven. ▪ See technical literature.
	*The parameter **Stop drift** is set too high.*	▪ Set the **Stop drift** lower.

Problem	Cause	Remedy
	The drift correction is too large (e.g. with excessively high start drift, with fluctuating drift or with manual drift correction).	▪ Ensure stable and lower drift. ▪ Set the parameter **Drift correction** to **auto** (= automatic drift correction).
	*The parameter **Min. rate** is set too low.*	▪ Set the parameter **Min. rate** higher. ▪ Reset the **Control parameters** to default values.
The results are spread widely.	*The sample is nonhomogenous.*	▪ Homogenize the sample if possible. ▪ Possibly use a larger sample size.
	The sample addition is difficult to reproduce.	▪ Observe the instructions in *Chapter 4.3, page 31*.
	The drift fluctuates.	▪ Ensure stable drift.

8.2 Miscellaneous

Problem	Cause	Remedy
No report is printed.	*The printer is not recognized by the instrument.*	▪ Switch the 899 Coulometer off and then back on again. ▪ Use a USB hub and connect the printer to the USB hub.
	The printer model is not compatible.	Use a printer which fulfills the required specifications *(see chapter 9.3.4, page 95)*.
The USB keyboard or the PC mouse does not function.	*The keyboard or mouse is not recognized by the instrument.*	▪ Switch the 899 Coulometer off and then back on again. ▪ Use a USB hub and connect the keyboard or mouse to the USB hub.
	The keyboard or the mouse is not compatible.	Use a model which fulfills the required specifications *(see chapter 9.3, page 93)*.
The display is no longer readable.	*The contrast is set incorrectly.*	Adjust the contrast correctly *(see chapter 6.1, page 62)*.
Mettler XP balances send "R" or "O ----" as ID1.	*The automatic calibration of the balance is activated.*	Deactivate the automatic calibration.

Problem	Cause	Remedy
Message 020-511 "Action not possible" is displayed.	*The USB flash drive is no longer connected.*	1. Connect the USB flash drive. 2. Switch the instrument off and then back on again.
	The USB flash drive is full.	▪ Use a different USB flash drive. ▪ Delete files with the aid of a PC.

9 Appendix

9.1 Stirring rate

The stirring rate can be adjusted in steps from –15 to +15.

The approximate rotational speed for the internal magnetic stirrer (depends on the product version) can be calculated with the following formula:

Rotational speed/min (r/min) = 125 · Stirring rate

Example:

Configured stirring rate: 8

Rotational speed in revolutions per minutes = 125 · 8 = 1,000

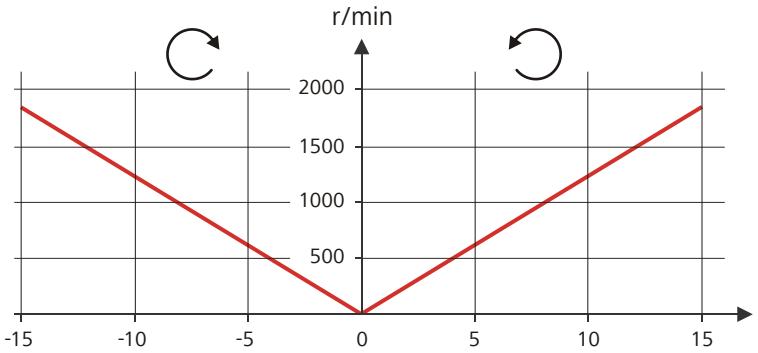


Figure 25 Rotational speed depending on the stirring rate

The information on the separately connectable 802 propeller stirrer can be found in the "802 Stirrer" manual.

9.2 Balance

The sample size and the associated unit can be sent from a connected balance. The sample size is transmitted as a number with up to ten characters (including algebraic sign and decimal point).

Sample size and unit are sent as a single character string. They are separated by a space character. The string is terminated with the ASCII characters **CR** and **LF**.

If the balance sends a negative sample size (e.g. when you are reweighing a sample), then the algebraic sign is adopted. The algebraic sign is, however, ignored for the calculations.

NOTICE

With some balances, the sample identification and the method can be sent in addition to the sample size.

Make sure that the balance does not send the sample size until the end.

Mettler AX

For the Mettler AX balance, the fields that contain the sample identification or the method must be designated as follows:

- Designation for the field with the method name: **METHOD**
- Designation for the field with sample identification 1: **ID1**
- Designation for the field with sample identification 2: **ID2**

9.3 USB devices

NOTICE

USB peripheral devices that are to be connected must support either the *USB 1.0/1.1 (Full Speed)* or the *USB 2.0 (High Speed)* standard. The maximum data transfer rate is however in any case 12 MBit/s.

Keyboards, PC mice and barcode readers are so-called HID devices (**H**uman **I**nterface **D**evice) and can be connected via a USB hub only.

Printers should also be connected via a USB hub. Depending on the manufacturer or printer type a direct connection is however possible.

9.3.1 6.2147.000 numerical USB keypad

The **[Num Lock]** key must be pressed for navigating in the dialog. The arrow keys are effective in conjunction with it.

The respective editing dialog must be opened for the numerical input.

Table 4 Key assignment

Key of the 899 Coulometer or function in the editing dialog	Key on the numerical USB keypad
[BACK]	**[Home]**
[⇧] [⇩]	**[↑] [↓]**
[⇦] [⇨]	**[←] [→]**
[OK]	**[Enter]**

Key of the 899 Coulometer or function in the editing dialog	Key on the numerical USB keypad
[←–]	**[BS]** (backspace)
Clear	**[Del]**
Accept	**[Home]**

9.3.2 Key assignment of a USB keyboard

A commercially available USB keyboard can be connected to make it easier to enter text and numbers.

The respective editing dialog must be opened for the text input and numerical input.

Table 5 Key assignment

Key of the 899 Coulometer or function in the editing dialog	Key on the USB keyboard
[BACK]	**[Esc]**
[⇧] [⇩]	**[↑] [↓]**
[⇦] [⇨]	**[←] [→]**
[OK]	**[↵]** (enter key) *or* **[Enter]** on the numerical keypad
[STOP]	**[Ctrl]** + **[S]**
[START]	**[Ctrl]** + **[G]**
[←–]	**[←]** (backspace)
Clear	**[Delete]**
Cancel	**[Ctrl]** + **[Q]**
Accept	**[Esc]**

NOTICE

The lettering of the USB keyboard may differ from above lettering, depending on the country-specific keyboard used.

9.3.3 PC mouse

In order to make navigating in the dialog of the 899 Coulometer easier, a PC mouse can be connected.

Table 6 Mouse functions

Key of the 899 Coulometer	Mouse function
[OK]	Left mouse button
[BACK]	Right mouse button
[⇧] [⇩] [⇦] [⇨]	Mouse movement vertical/horizontal
[⇧] [⇩]	Scroll wheel vertical

9.3.4 Printer

The range of USB printers available is extremely varied and constantly changing. The following points must be taken into account when selecting a printer:

- USB interface necessary
- Printer language: HP-PCL, Canon BJL Commands, Epson ESC P/2 or ESC/POS

NOTICE

Inexpensive printers are often designed solely for use with a PC and may not be equipped with one of the printer languages listed above. Such models are not suitable for this reason.

9.4 Control parameters and polarization current

The standard control parameters are optimally set for most determinations and should not be altered. Should you nevertheless need to change the control parameters for special reagents and/or samples, please note that the following parameters are dependent on one another:

- The polarization current of the indicator electrode *(see parameter "I(pol)", page 80)*
- The end point *(see parameter "Endpoint at", page 76)*
- The control range *(see parameter "Dynamics", page 76)*

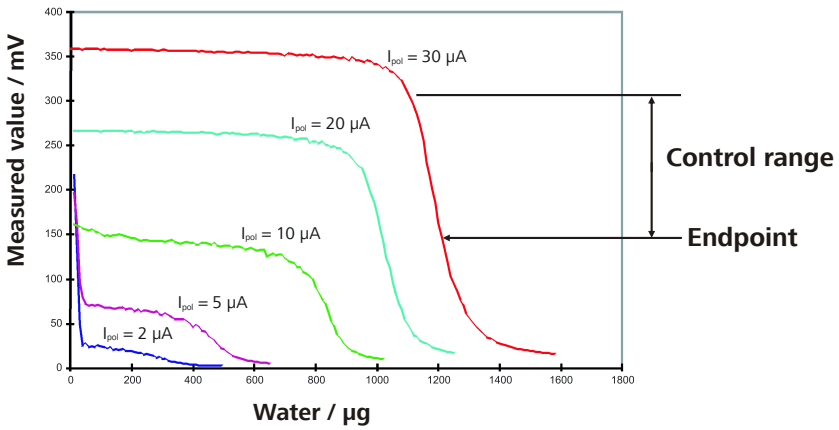


Figure 26 Control parameters and polarization current

The diagram shows Karl Fischer titration curves at different polarization currents (Reagent Coulomat AD). It is clear to see that the position of the endpoint varies with the polarization current. The curves have different slopes, i.e. the dynamics must also be adapted. Polarization currents < 10 µA are not suitable for this application. The following table gives you an idea of the optimal control parameters for various polarization currents.

Table 7 Optimum control parameters at different polarization currents

I(pol) (polarization current)	**10 µA**	**20 µA**	**30 µA**
Endpoint at	50 mV	100 mV	150 mV
Dynamics	70 mV	100 mV	120 mV
Max. rate	Default value	Default value	Default value
Min. rate	Default value	Default value	Default value
Stop drift	Default value	Default value	Default value

The indicator electrode is activated after a certain period of use in the same reagent, i.e. the titration curve becomes steeper. If the titration curve is too steep, then slowly fluctuating drift values could occur during conditioning. You can solve this problem by setting the endpoint lower. Endpoints that have been set too low could however prolong the titration. This could have unfavorable effects on the measuring error.

9.5 Method examples for working with the Karl Fischer oven

You will find the recommended parameter settings for working with a Karl Fischer oven in the following table. The methods can be used either with a sample changer with oven module (e.g. *885 Compact Oven SC*) or with a Karl Fischer oven (e.g. *860 KF Thermoprep*). The table contains only parameter settings that differ from the default values.

Parameters	Blank value determination (method template Blank)	Water content determination (method template KFC-Blank)
>Conditioning		
Start drift	10 µg/min *)	10 µg/min *)
Stabilizing time	30 s	30 s
>Start conditions		
Request sample size	off	off
Hold at request	off	off
>Titration parameters		
Extraction time	180 s	180 s
Stirring rate	12	12
>Statistics		
Statistics	on	–
Number of determinations	3	–
>Reports		
Results	on	on
Calculations/Statistics	on	–

*) If you work in an environment with high humidity, then we recommend that the parameter **Start drift** be set to 20 µg/min.

9.6 System initialization

In very rare instances, a faulty file system (e.g. because of a program crash) may lead to an impairment of program functioning. The internal file system must be initialized in such cases.

CAUTION

All user data (methods, etc.) are deleted if a system initialization is carried out. Afterwards, the instrument will have the factory settings again.

We recommend creating a backup of the system at regular intervals in order to avoid data losses.

After a system initialization the program versions and language files do not have to be reloaded. Only the selection of the dialog language may have to be reset in the system settings.

Proceed as follows for the system initialization:

1 Switching off the instrument

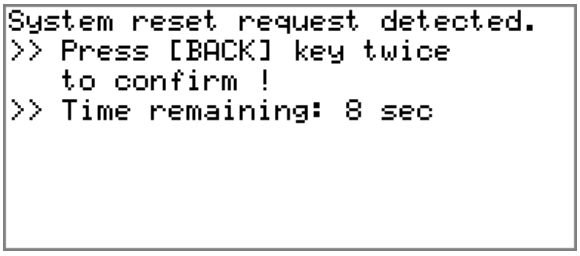
- Keep the red **[STOP]** key pressed down for at least 3 s.

A progress bar is displayed. If the key is released during this time, then the instrument will not be switched off.

2 Switching on the instrument

- Keep the red **[STOP]** key pressed down for approx. 10 s.

The dialog for confirmation of the initialization is displayed for 8 s. The initialization must be confirmed during this time.



3 Confirming the initialization

NOTICE

If the request is not confirmed within 8 s, then the procedure will be canceled.

- Press **[BACK]** twice.

Initialization is started. The process takes approximately 80 s. The instrument will be automatically restarted after successful initialization.

9.7 Remote interface

9.7.1 Pin assignment of the remote interface

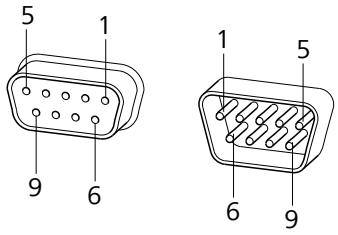


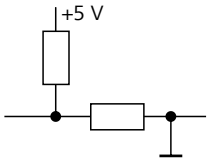
Figure 27 Pin assignment of remote socket and remote plug

The above figure of the pin assignment applies for all Metrohm instruments with 9-pin D-Sub remote connector.

Table 8 Inputs and outputs of the remote interface

Pin No.	Assignment	Function
1	Output 0	Ready/EOD
2	Output 1	-
3	Output 2	Titration
4	Output 3	Cond OK
5	Output 4	Error
6	0 volt (GND)	
7	+5 volts	
8	Input 0	Start
9	Input 1	Stop

Inputs

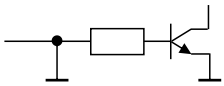


approx. 5 kΩ Pull-up

t_p > 100 ms

active = low, inactive = high

Outputs



Open Collector

t_p > 200 ms

active = low, inactive = high

I_C = 20 mA, V_{CEO} = 40 V

+5 V: maximum load = 20 mA

9.7.2 Status diagram of the remote interface

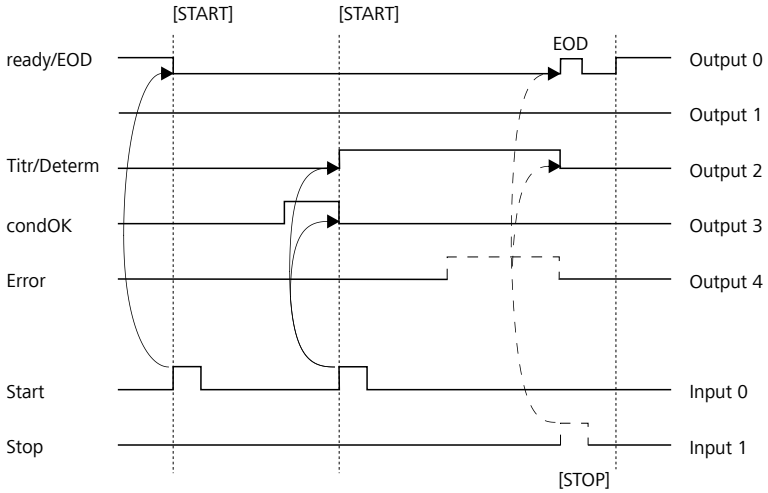


Figure 28 Remote status diagram

EOD = End of Determination

9.8 Remote control via an RS-232 connection

The 899 Coulometer can be remote controlled via an RS-232 connection. For this, a **6.2148.030 RS-232/USB Box** is necessary. Connect the RS-232/USB Box to the USB interface of the instrument.

The RS-232/USB Box has two connectors for RS-232 instruments. The RS-232 remote control functions only through the connector **RS-232/2**. The connector RS-232/1 is designed for connection with a balance. Connect the **RS-232/2** connector of the RS-232/USB Box to a serial interface (COM interface marked with the symbol **IOIOI**) of a PC. Use a **6.2134.040 connection cable**.

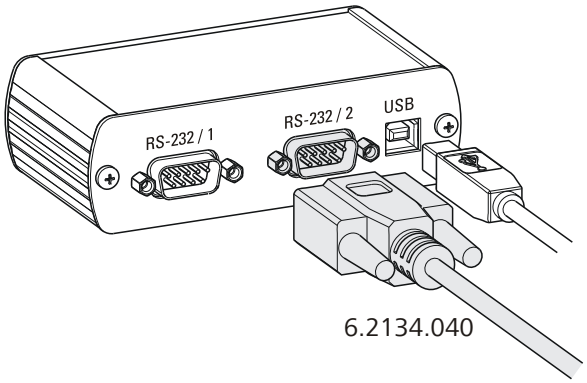


Figure 29 Connecting the RS-232/USB Box to the PC

Interface parameters

We recommend the following parameters for the RS-232 interface **COM2**:

- Baud rate: **19200**
- Data bits: **8**
- Stop bits: **1**
- Parity: **none**
- Handshake: **software**

On the 899 Coulometer, adjust the interface parameters under **Menu ▸ System ▸ External devices ▸ COM2 settings**.

Transfer protocol

The data communication is synchronous. On each command there is an acknowledgement by the instrument.

A command must be sent to the instrument with the control characters **CR LF** as terminator. The acknowledgements of the instrument will also be transferred with **CR LF** as terminator.

The instrument does not send spontaneous messages.

9.8.1 Commands and variables

Command	Function	Comment
$G	Start/Continue	Corresponds to the key **[START]** or **[Continue]**
$S	Stop	Corresponds to the key **[STOP]**
$H	Hold	Hold the method run
$D	Scan instrument status	Acknowledgements: *Ready;0*, *Busy;0*, *Hold;0* or *Cond;0* (0 = no message) If a message on the instrument requires the interaction of the user, the acknowledgement of the status scan displays the corresponding message number. Example: Busy;012-111 = **012-111 Conditioning not OK** The message can be confirmed with **[Yes]** or **[No]**, see below.
$A	Confirm message	Confirm the message on the instrument with **[OK]** A mandatory status scan providing the message number must take place immediately before confirming the message, see above.
$A(OK), **$A(CANCEL)**	Confirm message	Confirm the message with **[OK]** or **[Cancel]**
$A(YES), **$A(NO)**	Confirm message	Confirm the message with **[Yes]** or **[No]**
$L(*method name*)	Load method	The method name has to be known and unique.
$Q(*variable*)	Request variable value	Examples for variables: *EP1*, *R1*, *C00*. Complete list of the variables, *see page 39*.

The values of the variables are only available after the end of a determination (in the status 'ready').

Acknowledgement of the instrument	Comment
OK	Command executed
E1	Method not found
E2	Invalid variable
E3	Invalid command

9.9 Arithmetic algorithms in the 899 Coulometer

Numerical format

The software of the 899 Coulometer calculates in accordance with the widespread standard IEEE 754 (IEEE Standard for Binary Floating-Point Arithmetic for Microprocessor Systems). This means that the numbers are used in calculations in "double precision" (64 bit). Decimal numbers are converted into binary numbers in the computer and used in this form for calculations. The output on the display and in reports once again contains decimal numbers; the binary numbers are thus converted back into decimal numbers. In order to be able to check the internal calculations performed by the computer yourself in accordance with IEEE 754, the numbers are reproduced in the calculation report in complete accuracy. A minimal difference may arise between an originally entered decimal number and the internal computer representation in complete accuracy in the range of the rear decimal places. This difference results from the fact that an exact binary equivalent does not exist for every decimal number. If, for example, you enter the sample size 50.3 mg, this will be depicted in the calculation report in "double precision" with 5.02999999999999E+01.

Rounding-off process

Measured values and results are rounded to the defined number of decimal places (commercial rounding, in accordance with the US Pharmacopeia USP). If the digit at the first dropped decimal place is **1, 2, 3 or 4**, then it will be rounded off; if this digit is **5, 6, 7, 8 or 9**, then it will be rounded up. Negative digits will be rounded in accordance with their amount, i.e. away from zero.

Examples:

2.33 yields **2.3**

2.35 yields **2.4**

2.47 yields **2.5**

–2.38 yields **–2.4**

–2.45 yields **–2.5**

Statistics

The arithmetic mean value and the absolute and relative standard deviations of results are calculated:

You can statistically evaluate a maximum of five results ($1 \leq k \leq 5$) calculated in a determination. A statistical series can contain a maximum of 20 determinations ($1 \leq n \leq 20$).

The following convention applies to the subsequent formulas:

$1 \leq n \leq 20$ and $1 \leq k \leq 5$.

Mean value:

$$\bar{x}_k = \frac{1}{n} \cdot \sum_{i=1}^{n} R_{k,i}$$

Absolute standard deviation:

$$S\,abs_k = +\sqrt{\frac{\sum_{i=1}^{n} \left(R_{k,i} - \bar{x}_k\right)^2}{n-1}}$$

Relative standard deviation (in %):

$$S\,rel_k = 100 \cdot \frac{S\,abs_k}{\bar{x}_k}$$

Explanations

The individual values are incorporated in the statistics with full accuracy.

15 significant places are yielded when the 64 bit numerical format is applied for the floating-point number in decimal presentation.

The accuracy can be controlled by the selection of the prefix of the unit (milli, micro) and the number of decimal places.

Example:

The result displayed, **1234.56789158763 mg/L**, has 15 significant places. It should be rounded off to three decimal places according to the above rounding-off process:

- **1234.568 mg/L**.

If the same result is expressed in "**g/L**" (**1.23456789158763 g/L**), and is also rounded off to three decimal place, this yields

- **1.235 g/L**.

I.e. you obtain the lowest losses in accuracy with rounding when you select the application and the numerical format in such a way that the numbers displayed have as many places before the decimal point as possible.

A complete recalculation of the statistics using a pocket calculator or PC calculation programs may exhibit deviations. This can be explained by the different binary numerical formats used by these computers.

NOTICE

The above losses of accuracy by rounding off in the range of significant places are only relevant theoretically. They are generally several orders of magnitude less than measurement technique uncertainties (balance errors, dosing errors, measuring errors).

10 Technical specifications

10.1 Measuring inputs

The measuring cycle is 100 ms for the measuring mode Ipol AC.

10.1.1 Indicator electrode

One measuring input (**Ind.**) for an indicator electrode.

Measuring mode Ipol	Determination with adjustable polarization current. Voltametric endpoint indication with alternating current.
Polarization current	typical: 5, 10, 20 and 30 µA

10.1.2 Temperature

One measuring input (**Temp.**) for one Pt1000-model temperature sensor.

Measuring range	–150…+250 °C
Resolution	0.1 °C
Measuring accuracy	±0.2 °C (Applies for measuring range –20…+150 °C; ±1 digit; without sensor error, under reference conditions.)

10.2 Generator connector

One electrode connector **(Gen.)** for a generator electrode.

10.2.1 Generator electrode

Iodine generation with current pulses or continuous current.

Current at the electrode	**auto**: variable direct current 100, 200, 400 mA: pulsating direct current
I_{max}	400 mA

10.3 Internal stirrer

Rotational speed	
Maximum rotational speed	±(1700…1900) rpm
Setting of the rotational speed	±15 steps
Change in rotational speed per step	±(115…125) rpm
Fuse	Electronic overload protection

10.4 Interfaces and connectors

MSB connector MSB1	8-pin Mini DIN socket for connecting an additional stirrer.
USB (OTG) connector	For connecting USB peripheral devices.
Remote connector	For connecting instruments with a remote interface.

10.5 Power supply

External power supply unit	24 V, 65 W
Input voltage range	20…28 V DC
Maximum power consumption	36 W

10.6 Ambient temperature

Nominal function range	+5…+45 °C (at a maximum of 85% humidity)
Storage	–20…+60 °C
Transport	–40…+60 °C

10.7 Reference conditions

Ambient temperature	+25 °C (± 3 °C)
Relative humidity	≤ 60%
Operating temperature status	Instrument in operation at least 30 min
Validity of the data	after calibration

10.8 Dimensions

Width	143.5 mm
Height (without titration vessel holder)	112 mm
Height (with titration vessel holder)	195 mm
Depth	281.5 mm
Weight (including power supply unit)	2800 g
Material	
Housing	PBT
Material	
Base	Stainless steel 1.4301

11 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. **899**) 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.

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