852 Titrando

Manual

8.852.8003EN / 2020-08-10

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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 The Titrando system

The Titrando is the heart of the modular Titrando system. Operation is carried out either by a Touch Control with a touch-sensitive screen ("stand-alone titrator") or by a computer with a corresponding software.

A Titrando system can contain numerous, various kinds of instruments. The following figure provides an overview of the peripheral devices you can connect to the 852 Titrando.

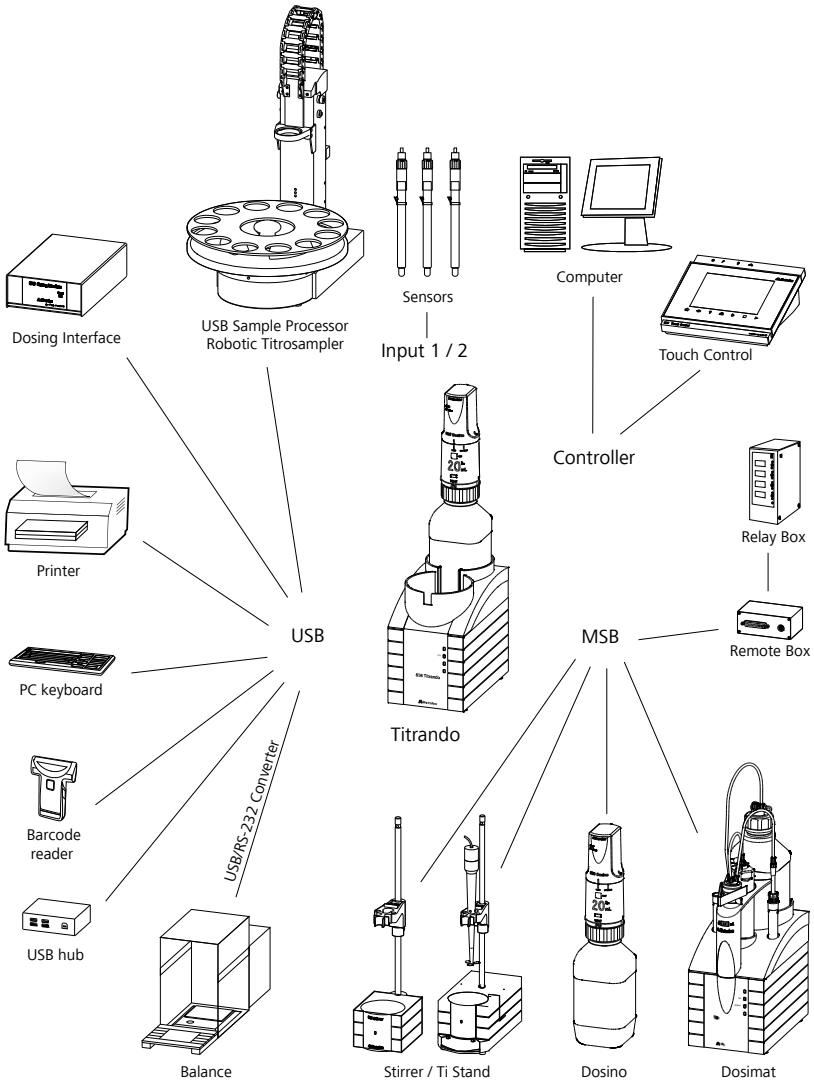


Figure 1 The Titrando system

Up to three control instruments (Titrando, Dosing Interface, USB Sample Processor, etc.) can be controlled via USB connection during operation with the 900 Touch Control.

You can request information on special applications in the "Application Bulletins" and "Application Notes", available free of charge via the Metrohm representative responsible. Various monographs on the subjects of titration techniques and electrodes are also available.

Updating the device software is described in the help of the corresponding PC software.

1.2 Instrument description

The 852 Titrando has the following characteristics:

- **Operation**
  Operation is carried out by means of a touch-sensitive Touch Control or with a high-performance PC software.
- **MSB connectors**
  Four MSB connectors (Metrohm Serial Bus) for connecting dosing devices (Dosimat with exchange unit or Dosino with dosing unit), stirrers, titration stands and Remote Boxes.
- **USB connectors**
  Two USB connectors, through which devices such as printers, PC keyboards, barcode readers or additional control devices (USB Sample Processor, Titrando, Dosing Interface, etc.) can be connected.
- **Measuring interface**
  Two measuring interfaces. Measuring interface 1 has one measuring input each for:
  - a generator electrode
  - a temperature sensor (Pt1000 or NTC)
  - a double Pt electrode

  Measuring interface 2 has one measuring input each for:
  - a temperature sensor (Pt1000 or NTC)
  - a polarizable electrode

1.3 Titration modes – Measuring modes – Dosing commands

The 852 Titrando supports the following titration modes, measuring modes and dosing commands:

- **MET**
  Monotonic equivalence point titration. The reagent is added in constant volume steps.
  Measuring modes:
  - **Ipol** (voltametric measurement with selectable polarization current)
  - **Upol** (amperometric measurement with selectable polarization voltage)
- **SET**
  Endpoint titration at one or two specified endpoints, for volumetric bromine index determination.
  - **Ipol** (voltametric measurement with selectable polarization current)
  - **Upol** (amperometric measurement with selectable polarization voltage)
- **KFT**
  Volumetric water content determination according to Karl Fischer.
  Measuring modes:
  - **Ipol** (voltametric measurement with selectable polarization current)
  - **Upol** (amperometric measurement with selectable polarization voltage)
- **KFC**
  Coulometric water content determination according to Karl Fischer.
  Measuring mode:
  - **Ipol** (voltametric measurement with selectable polarization current)
- **BRC**
  Coulometric bromine index determination. Determining the amount of double bonds in e.g. mineral oils.
  Measuring mode:
  - **Ipol** (voltametric measurement with selectable polarization current)

- **MEAS**
  The following measuring modes can be selected for measurements:
  - **Ipol** (voltametric measurement with selectable polarization current)
  - **Upol** (amperometric measurement with selectable polarization voltage)
  - **T** (temperature measurement)
- **Dosing commands**
  The following commands for dosing can be selected:
  - **PREP** (rinsing the cylinder and tubings of an exchange unit or dosing unit)
  - **EMPTY** (emptying the cylinder and tubings of a dosing unit)
  - **ADD** (dosing a specified volume)
  - **LQH** (carrying out complex dosing tasks with a Dosino)

Parallel determinations by coulometric (KFC or BRC) and volumetric titration (KFT or SET/MET) are not possible with the Touch Control.

1.4 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.

1.4.1 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.

1.5 Safety instructions

1.5.1 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.

1.5.2 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.

1.5.3 Working with liquids

CAUTION

Periodically check all system connections for leaks. Observe the relevant regulations in respect to working with flammable and/or toxic fluids and their disposal.

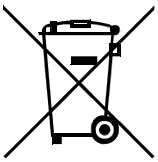
1.5.4 Flammable solvents and chemicals

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.

1.5.5 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

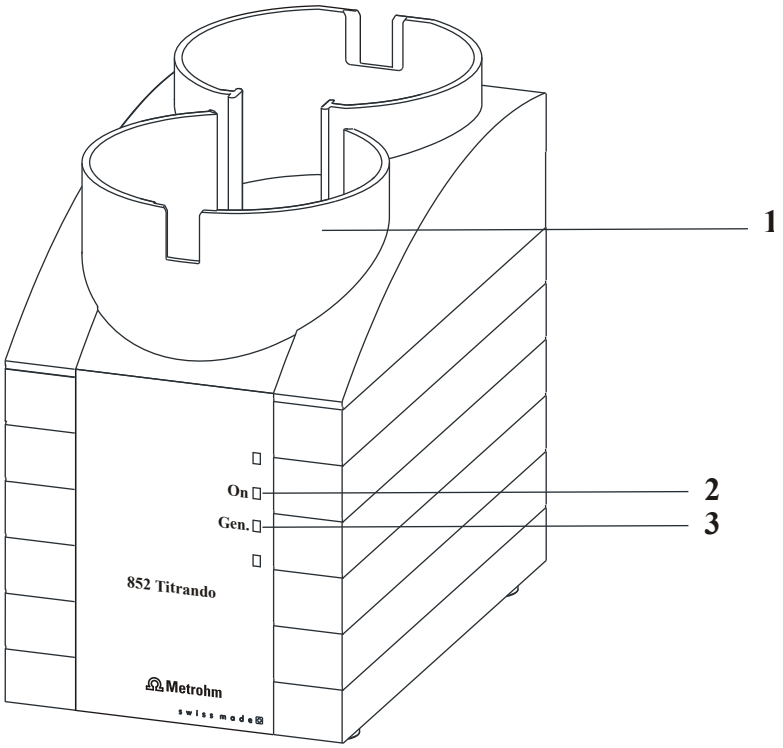


Figure 2 Front 852 Titrando

1 Bottle holder
With holding clamps, for two reagent bottles.

2 "On" LED
Lights up when the Titrando is ready for operation.

3 "Gen." LED
Lights up when the Titrando is ready for operation and the generator electrode is connected.

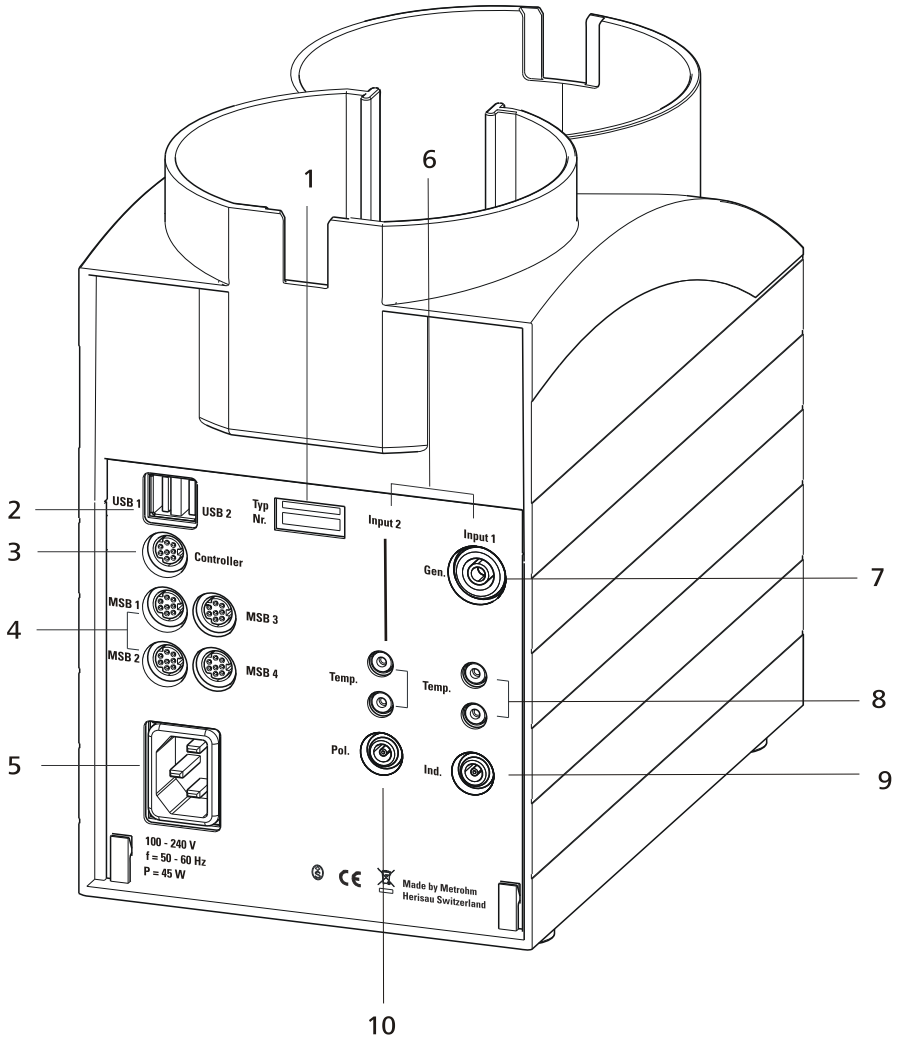


Figure 3 Rear 852 Titrando

1 Type plate
Contains specifications concerning supply voltage, instrument type and serial number.

2 USB connector (USB 1 and USB 2)
USB ports (type A) for connecting printer, keyboard, barcode reader, additional Titrandos, USB Sample Processor, etc.

3 Connector (Controller)
For connecting a Touch Control or a PC with installed PC software. Mini DIN, 9-pin.

4 MSB connector (MSB 1 to MSB 4)
Metrohm Serial Bus. For connecting external dosing devices, stirrers or Remote Boxes. Mini DIN, 9-pin.

5 Power socket

6 Measuring interface 1 (Input 1) and measuring interface 2 (Input 2)
Input 1 for coulometry.

Input 2 for volumetry.

7 **Electrode connector (Gen.)**
For connecting a generator electrode.

8 **Temperature sensor connector (Temp.)**
For connecting temperature sensors (Pt1000 or NTC). Two B sockets, 2 mm.

9 **Electrode connector (Ind.)**
For connecting a double Pt electrode with coulometric measurements. Socket F.

10 **Electrode connector (Pol.)**
For connecting a polarizable electrode, e.g. a double Pt electrode with volumetric measurements. Socket F.

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 Connecting a controller

3.2.1 Operation

Two different versions are available for operating the 852 Titrando:

- A Touch Control with touch-sensitive screen. It forms a "stand-alone instrument" together with the 852 Titrando.
- A computer enables operation of the 852 Titrando with the help of a PC software, e.g. *tiamo*.

CAUTION

Take care to ensure that the power cord is pulled out of the power socket before either setting up or disconnecting connections between the instruments.

3.2.1.1 Connecting a Touch Control

NOTICE

The 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 marked with arrows.

Connect the Touch Control as follows:

1 ▪ Insert the plug of the Touch Control connection cable into the **Controller** socket.

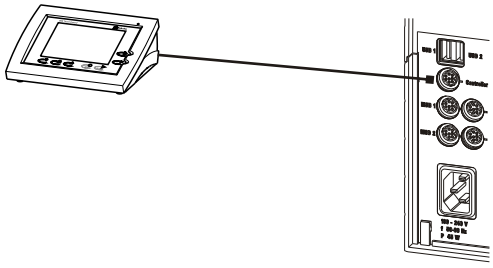


Figure 4 Connecting the Touch Control

2 ▪ Connect the MSB devices *(see chapter 3.3, page 16)*.
▪ Connect the USB devices *(see chapter 3.4, page 20)*.

3 ▪ Connect the Titrando to the power grid *(see chapter 3.2.1.2, page 13)*.

4 ▪ Switch on the Touch Control.

The Touch Control power supply is supplied through the Titrando. Automatic system tests are performed on both instruments at the time of activation. The **On** LED on the front of the Titrando lights up when the system test has been completed and the instrument is ready for operation.

CAUTION

The Touch Control must be shut down properly by deactivation with the power switch on the rear of the instrument before the power supply is interrupted. If this is not done, then there is a danger of data loss. Because of the fact that the power supply for the Touch Control is provided through the Titrando, you must never disconnect the Titrando from the power grid (e.g. by deactivating with a connector strip) before you have deactivated the Touch Control.

If you would prefer not to position the Touch Control directly next to the Titrando, then you can lengthen the connection with the 6.2151.010 cable. The maximum connection length permitted is 5 m.

3.2.1.2 Connecting the instrument to the power grid

WARNING

Electric shock from electrical potential

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

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

Connecting the power cord

Accessories

Power cord with the following specifications:

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

NOTICE

Do not use a not permitted power cord!

1 Plugging in the power cord

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

3.2.1.3 Connecting a computer

The 852 Titrando requires a USB connection to a computer in order to be able to be controlled by a computer software. Using a 6.2151.000 controller cable, the instrument can be connected directly, either to a USB socket on a computer, to a connected USB hub or to a different Metrohm control instrument.

You need administrator rights for the installation of driver software and control software on your computer.

Cable connection and driver installation

A driver installation is required in order to ensure that the 852 Titrando is recognized by the computer software. To accomplish this, you must comply with the procedures specified. The following steps are necessary:

1 Install the software

- Insert the computer software installation CD and carry out the installation program directions.
- Exit the program if you have started it after the installation.

2 Establishing the cable connections

- Connect all peripheral devices to the instrument, *see Chapter 3.3, page 16* and *see Chapter 3.4, page 20*.
- Connect the instrument to the power grid if you have not already done this *(see chapter 3.2.1.2, page 13)*.
  The "On" LED on the 852 Titrando is not yet illuminated!
- Connect the instrument to a USB connector (type A) of your computer (see manual of your computer). The 6.2151.000 cable is used for this purpose.

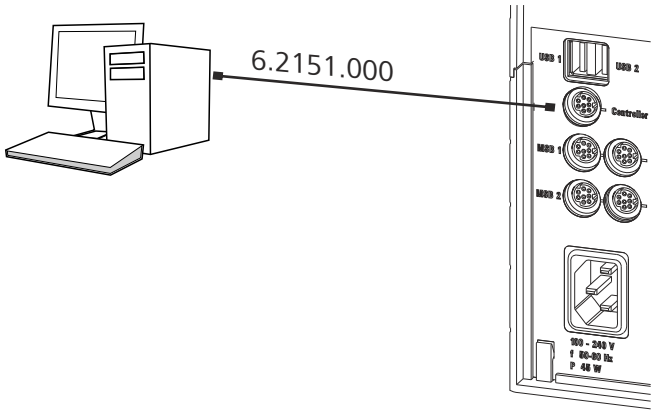


Figure 5 Connecting the computer

The instrument is recognized. Depending on the version of the Windows operating system used, the driver installation proceeds differently afterwards. Either the necessary driver software is installed automatically or an installation wizard is started.

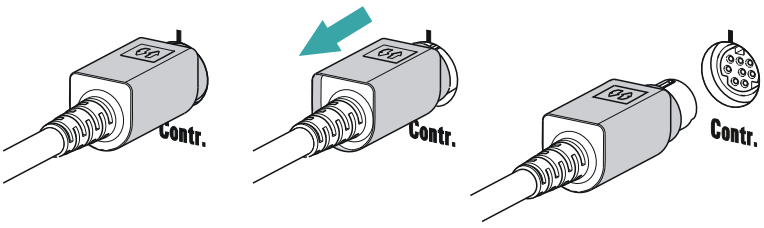
3 Follow the instructions of the installation wizard.

The "On" LED on the 852 Titrando lights up when the driver installation has been completed and the instrument is ready for operation.

If problems should occur during installation, contact your company's IT support team.

NOTICE

The plug on the instrument end of the 6.2151.000 controller cable is protected against accidental disconnection 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 marked with arrows.



Registering and configuring the instrument in the computer software

The instrument must be registered in the configuration of your computer software. Once that has been done, you can then configure it according to your requirements. Proceed as follows:

1 Setting up the instrument

- Start the computer software.
  The instrument is automatically recognized. The configuration dialog for the instrument is displayed.
- Make configuration settings for the instrument and its connectors.

More detailed information concerning the configuration of the instrument can be found in the documentation for the respective computer software.

3.3 Connecting MSB devices

In order to connect MSB devices, e.g. stirrers or dosing devices, Metrohm instruments are equipped with up to a maximum of four connectors on what is referred to as the *Metrohm Serial Bus* (MSB). Various kinds of peripheral devices can be connected in sequence (in series, as a "Daisy Chain") at a single MSB connector (8-pin Mini DIN socket) and controlled simultaneously by the respective control instrument. In addition to the connection cable, stirrers and the Remote Box are each equipped with their own MSB socket for this purpose.

The following figure provides an overview of the instruments that can be connected to an MSB socket, along with a number of different cabling variations.

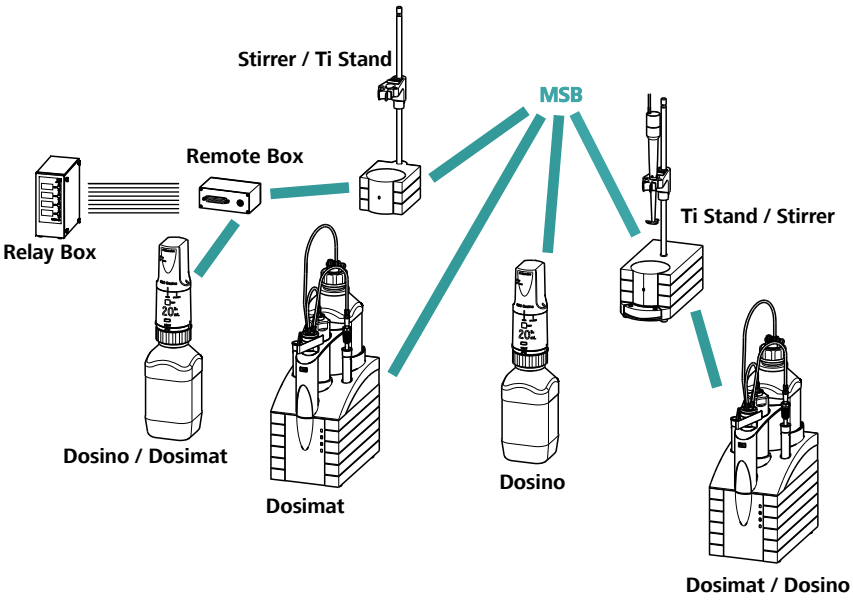


Figure 6 MSB connections

The control instrument determines which peripheral devices are supported.

NOTICE

When connecting MSB devices together, the following must be observed:

- Only one device of the same type can be used at a single MSB connector at one time.
- Dosing devices of the 700 Dosino and 685 Dosimat plus type cannot be connected together with other MSB devices on a shared connector. These dosing devices must be connected separately.

CAUTION

Exit the control software before you plug in MSB devices. When it is switched on, the control instrument automatically recognizes which device is connected to which MSB connector. The operating unit or the control software enters the connected MSB devices into the system configuration (device manager).

MSB connections can be extended with the 6.2151.010 cable. The maximum connection length permitted is 15 m.

3.3.1 Connecting a dosing device

Four dosing devices can be connected to the instrument (**MSB 1 to MSB 4**).

The types of dosing devices that are supported are:

- 800 Dosino
- 700 Dosino
- 805 Dosimat
- 685 Dosimat plus

Proceed as follows:

1 Connecting a dosing device

- Exit the control software.
- Connect the connection cable of the dosing device to one of the sockets marked with **MSB** on the rear of the control device.
- Start the control software.

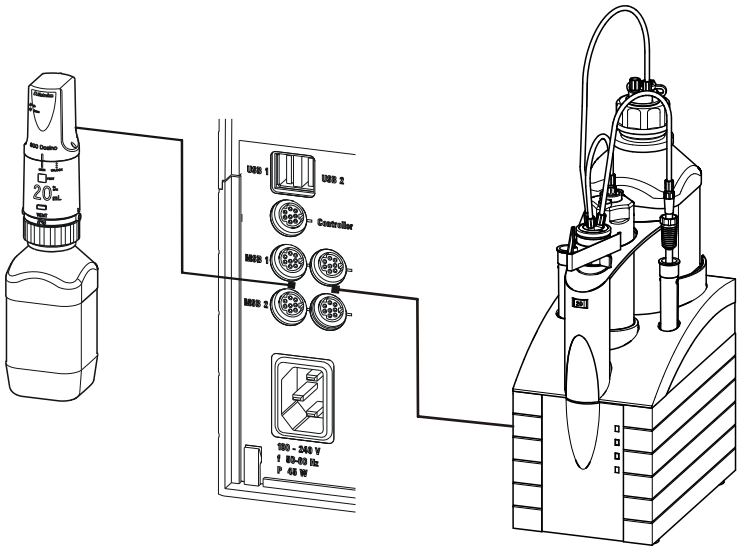


Figure 7 Connecting a dosing device

3.3.2 Connecting a stirrer or titration stand

You can use the following instruments:

These devices have a built-in magnetic stirrer (stirring "from below"):

- 801 Stirrer
- 803 Ti Stand

This device has no built-in magnetic stirrer (stirring "from above"):

- 804 Ti Stand with rod stirrer 802 Stirrer

Connect a stirrer or a titration stand as follows:

1 Connecting the stirrer or titration stand

- Exit the control software.
- Connect the connection cable of the magnetic stirrer or of the titration stand to one of the sockets marked with **MSB** on the rear of the control instrument.
- 804 Ti Stand only: Connect the rod stirrer to the stirrer connector (socket with stirrer symbol) of the titration stand.
- Start the control software.

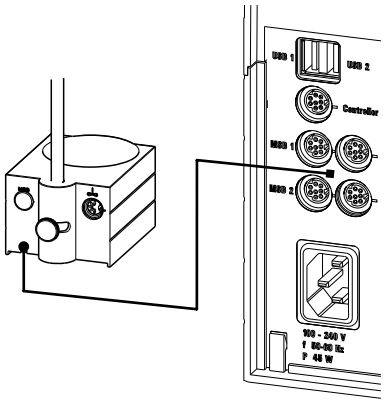


Figure 8 Connecting an MSB stirrer

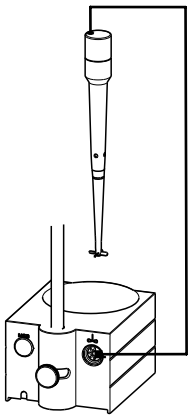


Figure 9 Connecting the rod stirrer to the titration stand

3.3.3 Connecting a Remote Box

Instruments that are controlled via remote lines and/or that send control signals via remote lines can be connected via the 6.2148.010 Remote Box. In addition to Metrohm, other instrument manufacturers also use similar connectors that make it possible to connect different instruments together. These interfaces are also frequently given the designations "TTL Logic", "I/O Control" or "Relay Control" and they generally have a signal level of 5 volts.

Control signals are understood to be electrical line statuses or electrical pulses (> 200 ms) which display the operating status of an instrument or which trigger or report an event. Sequences on a variety of instruments can thus be coordinated in a single complex automation system. However, no exchange of data is possible.

Proceed as follows:

1 Connecting the Remote Box

- Exit the control software.

- Connect the Remote Box connection cable to one of the sockets marked with **MSB** on the rear of the control instrument.
- Start the control software.

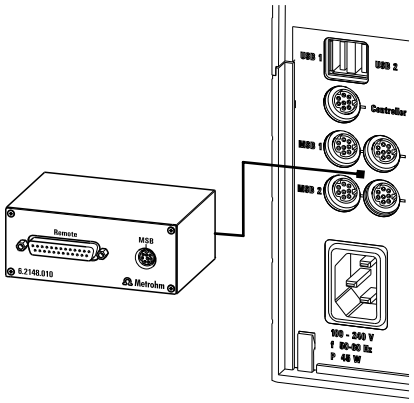


Figure 10 Connecting the Remote Box

You can connect the following instruments to the remote connector, among others:

- 849 Level Control (fill level monitoring in a canister)
- 731 Relay Box (switch box for 230/110 volt alternating current sockets and low-voltage direct current outlets)
- 843 Pump Station (for complex sample preparations or for rinsing external titration vessels)

The Remote Box also has an MSB socket at which a further MSB device, e.g. a dosing device or a stirrer, can be connected.

You will find precise information concerning the pin assignment of the interface on the Remote Box in the *appendix*.

3.4 Connecting USB devices

3.4.1 General

The 852 Titrando has two USB connectors (type A sockets) for peripheral devices with USB interfaces. The Titrando functions as a USB hub (distributor) no matter how it is operated. If you wish to connect more than two devices to the USB, you can also use an additional, commercially available USB hub.

CAUTION

If you operate the 852 Titrando with the aid of the Touch Control, take care to ensure that the Touch Control is switched off when you set up or disconnect connections between the various instruments. If you use a PC software to control the 852 Titrando, you should exit the program before you set up or disconnect the USB connections.

3.4.2 Connecting a USB hub

If you wish to connect more than two devices to the USB connector of the 852 Titrando, you can also use an additional commercially available USB hub (distributor). If you operate the 852 Titrando with the help of the Touch Control, then you should use a USB hub with its own power supply.

Connect the USB hub as follows:

1. Switch off the Touch Control and/or exit the PC software.
2. With the aid of the 6.2151.020 cable, connect the USB connector of the 852 Titrando (type A) with the USB connector of the hub (type B, see manual for the hub).
3. Switch on the Touch Control.

   The USB hub is recognized automatically.

3.4.3 Connecting a printer

Printers that are connected to the 852 Titrando with Touch Control must meet the following requirements:

- Printer languages: HP-PCL (PCL 3 to 5, PCL 3GUI), Canon BJL Commands or Epson ESC P/2
- Printer resolution: 300 dots/inch or 360 dots/inch (Epson)
- Paper size: A4 or Letter, single-sheet feed.

Connect the printer as follows:

1. Switch off the Touch Control.
2. With the aid of the 6.2151.020 cable, connect the USB connector of the 852 Titrando (type A) with the USB connector of the printer (type B, see manual for the printer).
3. Switch on the printer first, then the Touch Control.

4 Configure the printer in the device manager of the Touch Control (see Touch Control manual).

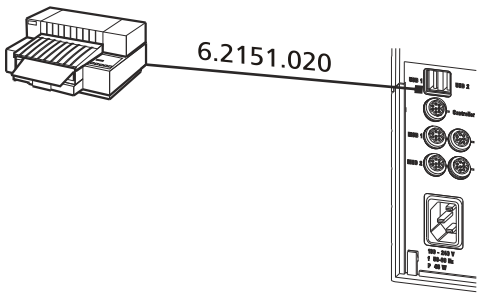


Figure 11 Connecting a printer

3.4.4 Connecting a balance

- Operation with a PC software:
  - Connect the balance directly to the serial connector (COM) of the computer. This is usually 9-pin and marked with the symbol **IOIOI**.
- Operation with Touch Control:
  - You will need the 6.2148.050 USB/RS-232 adapter to connect a balance.

The following table offers an overview of the balances that you can use together with the 852 Titrando 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

Balance	Cable
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

Operation with Touch Control

Connect the balance as follows:

1. Plug in the USB plug of the USB/RS-232 adapter at the USB connector of the 852 Titrando.
2. Connect the RS-232 interface of the USB/RS-232 adapter with the RS-232 interface of the balance (see table for cable).
3. Switch on the Touch Control.
4. Switch on the balance.
5. Activate the RS-232 interface of the balance if necessary.
6. Configure the RS-232 interface of the USB/RS-232 adapter in the device manager of the Touch Control (see Touch Control manual).

3.4.5 Connecting a PC keyboard (only for operation with Touch Control)

The PC keyboard is used as an aid for text and numerical input.

Connect the PC keyboard as follows:

1. Insert the USB plug of the keyboard into one of the USB sockets of the 852 Titrando.

2 Switch on the Touch Control.

The keyboard is recognized automatically and entered in the device manager.

3 Configure the keyboard in the device manager of the Touch Control (see Touch Control manual).

3.4.6 Connecting a barcode reader

The barcode reader is used as an aid for text and numerical input. You can connect a barcode reader with USB interface.

Operation with Touch Control

Connect the barcode reader as follows:

1 Insert the USB plug of the barcode reader into one of the USB sockets of the 852 Titrando.

2 Switch on the Touch Control.

The barcode reader is recognized automatically and entered in the device manager.

3 Configure the barcode reader in the device manager of the Touch Control (see Touch Control manual).

Settings on the barcode reader:

Program the barcode reader as follows (see also the manual for the barcode reader):

1 Switch the barcode reader to programming mode.

2 Specify the desired layout for the keyboard (USA, Germany, France, Spain, German-speaking Switzerland).

This setting must match the setting in the device manager (see the Touch Control manual).

3 Make sure that the barcode reader is set in such a way that Ctrl characters (ASCII 00 to 31) can be sent.

4 Program the barcode reader in such a way that the ASCII character 02 (STX or Ctrl B) is sent as the first character. This first character is normally referred to as the "Preamble" or "Prefix Code".

5 Program the barcode reader in such a way that the ASCII character 04 (EOT or Ctrl D) is sent as the last character. This last character is normally referred to as the "Postamble", "Record Suffix" or "Postfix Code".

6 Exit the programming mode.

3.5 Setting up the titration vessel

3.5.1 General

During the titration, it is important that the solution be well-mixed. The stirring rate should be high enough for a small "vortex" to appear. If the stirring rate is too high, then air bubbles will be aspirated. This results in incorrect measured values. If the stirring rate is too low, then the solution at the electrode will not be correctly mixed. In order to ensure that measurement is carried out in a well-mixed solution following addition of the titrant, the buret tip should be placed in a position where the turbulence is high. In addition, the distance between the addition of the titrant and the electrode should be as large as possible. Also take into account the stirring direction (counterclockwise or clockwise) when positioning electrode and buret tip (see figure below).

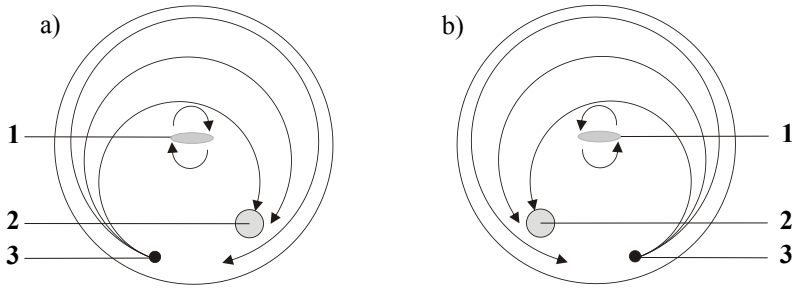


Figure 12 Schematic configuration of magnetic stirrer, indicator electrode and buret tip (Volumetry) /Generator electrode (Coulometry) during a titration. a) stirring direction clockwise, b) stirring direction counterclockwise.

1 Magnetic stirrer	**2 Indicator electrode**
3 Buret tip (Volumetry) / Generator electrode (Coulometry)	

3.5.2 Titration vessel for coulometric KF titration

3.5.2.1 Mounting the coulometer cell

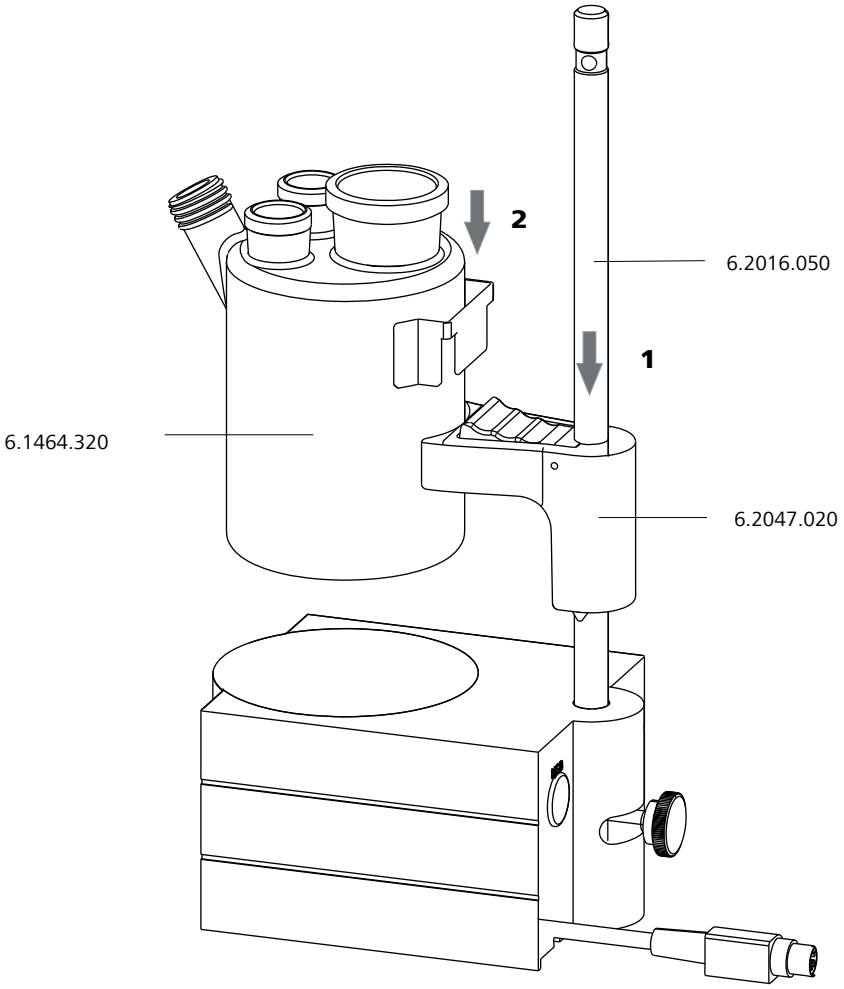


Figure 13 Mounting the coulometer cell

Mount the coulometer cell as follows on a titration stand:

1 Fix the 6.2047.020 titration vessel holder to the 6.2016.050 support rod.

2 Insert the 6.1464.320 titration vessel from above into the titration vessel holder.

3.5.2.2 Coulometer cell – Standard setup

Filling the adsorber tube

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

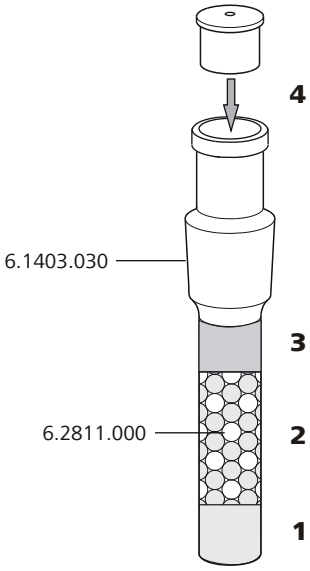


Figure 14 Filling the adsorber tube

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 coulometer cell

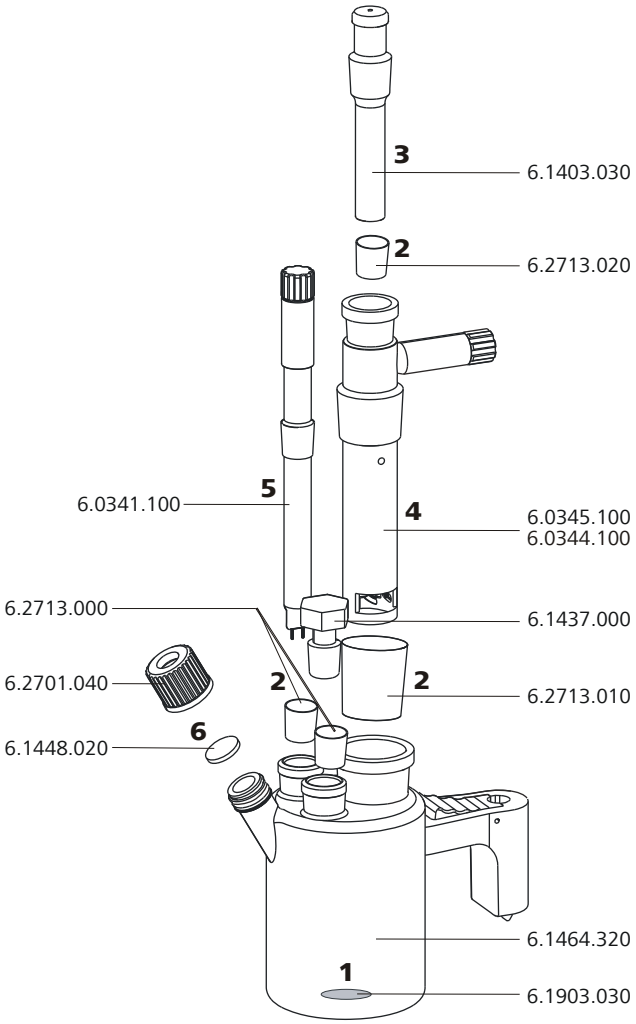


Figure 15 Equipping the coulometer cell

Equip the coulometer cell as follows:

1 Place the 6.1903.030 stirring bar in the coulometer 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.).

Take care to ensure that the edges of the ground-joint sleeves are cut to size cleanly and that there are no fringes. The ground-joint sleeves are not permitted to 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 coulometer 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 coulometer 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 coulometer 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 coulometer 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 coulometer 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).

Screwing on the electrode cable

Screw on the electrode cable as follows:

1 Unscrew the cover of the indicator electrode.

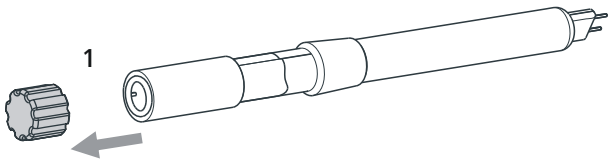


Figure 16 Unscrewing the cover from the indicator electrode

2 Unscrew the cover of the generator electrode.

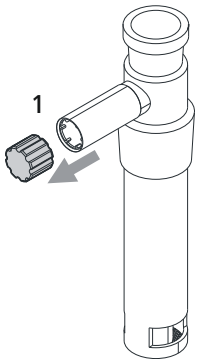


Figure 17 Unscrewing the cover from the generator electrode

3 Tighten the 6.2104.020 electrode cable to the indicator electrode.

4 Tighten the 6.2104.120 electrode cable to the generator electrode.

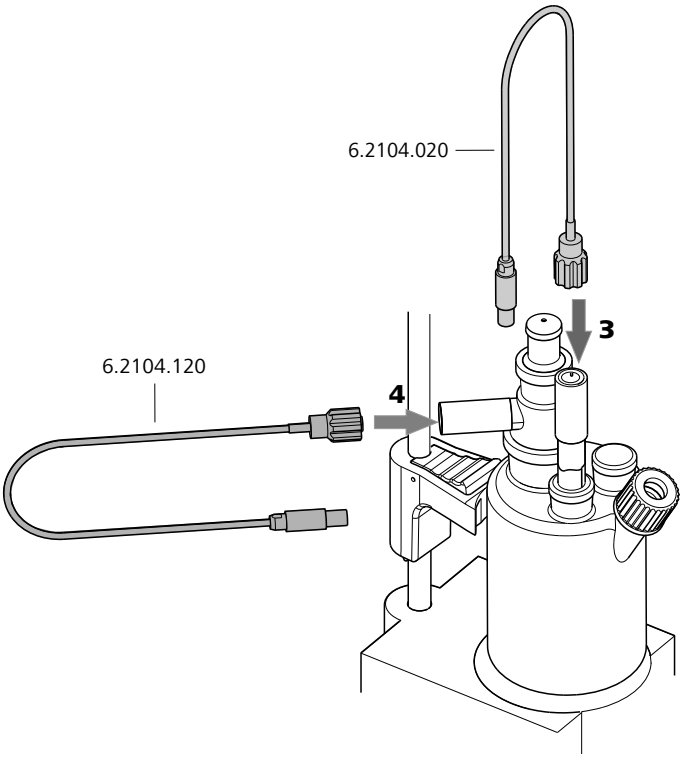


Figure 18 Screwing the electrode cable to the electrodes

NOTICE

Mark the screw head of the electrode cable. This prevents you from mixing up the indicator and generator electrode.

3.5.2.3 Coulometer cell with addition and aspiration tube (utilization with Ti Stand)

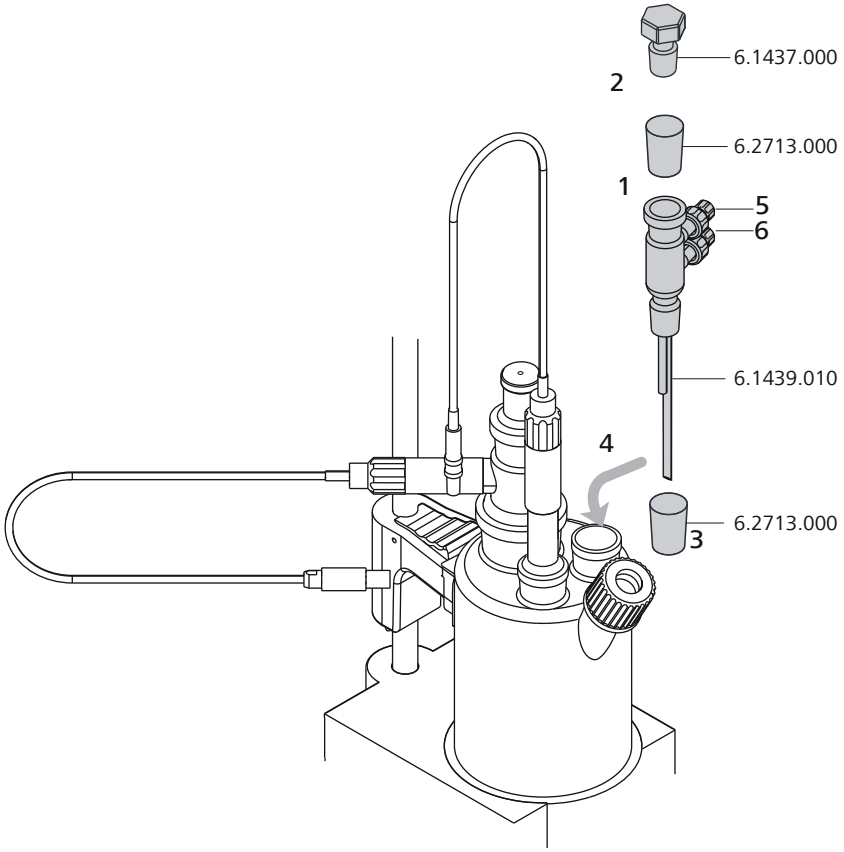


Figure 19 Mounting the addition and aspiration tube

Insert the addition and aspiration tube as follows into the coulometer 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).

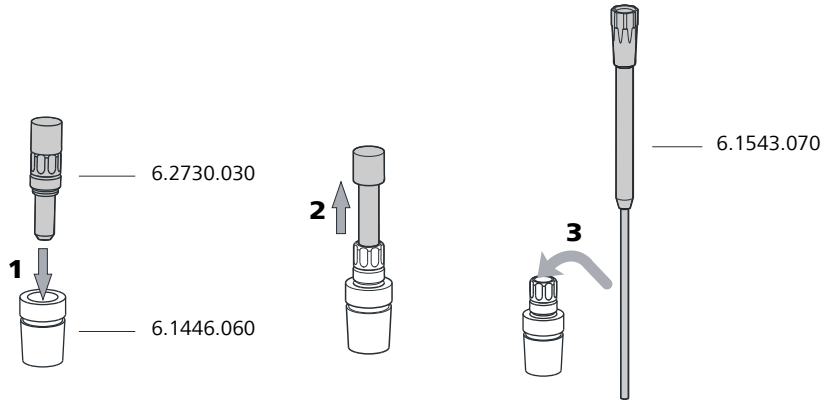
6 Connect the tubing for the aspiration of the coulometer cell at the lower connector of the addition and aspiration tube (6).

3.5.2.4 Coulometer cell with aspiration equipment (utilization with Dosino)

A Dosino allows the automatic replacement of reagents.

For aspiration, the 6.5617.000 aspiration equipment is used, including a complete dosing unit and a 50 mL glass cylinder. For aspirating greasy samples when only the sample and not the whole reagent is aspirated, we recommend using a dosing unit with a 20 mL cylinder. For highly viscous samples a dosing unit with a 10 mL cylinder is suitable.

Mounting the aspiration tip

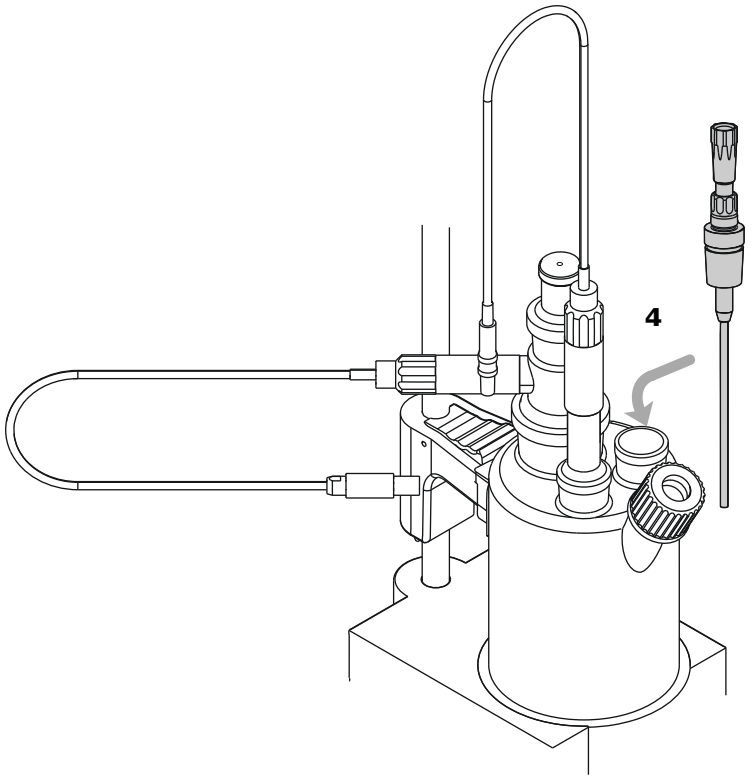


Insert the aspiration tip into the coulometer cell as follows:

1 Screw the 6.2730.030 nipple, with stopper and O-ring, onto the 6.1446.060 stopper.

2 Pull out the stopper.

3 Slide the 6.1543.070 aspiration tip through the stopper.



4 Place the stopper with the attached aspiration tip into the ground-joint opening with the ground-joint sleeve.

Insert the aspiration tip into the coulometer cell until it touches the vessel base.

3.5.2.5 Coulometer cell with Karl Fischer oven

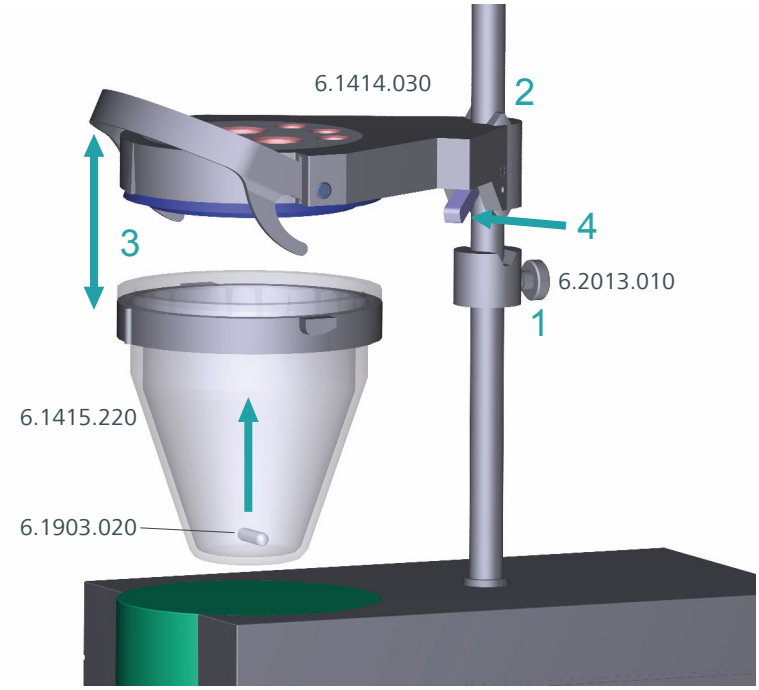
When samples release their water only slowly or only at higher temperatures, the oven method is used. The sample is heated in a KF oven (e.g. *860 KF Thermoprep*) and the water that is released is transferred to the coulometer cell with a carrier gas. A detailed description of setting up the coulometer cell with the KF oven can be found in the respective manual.

3.5.2.6 Coulometer cell with 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. *874 Oven Sample Processor*). A detailed description of setting up the coulometer cell with the sample changer can be found in the respective manual.

3.5.3 Titration vessel for volumetric KF titration

Mounting the KF titration cell

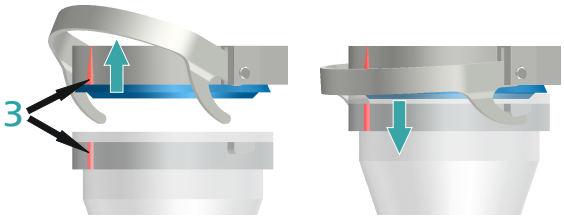


Proceed as follows:

1 Screw the 6.2013.010 clamping ring tightly to the support rod.

2 Fix the 6.1414.030 vessel lid of the KF titration cell (with correctly inserted sealing ring from the 6.1244.040 sealing set) to the support rod. Keep the locking lever pressed down until it can be released at the desired position.

3 Fasten the 6.1415.220 (or 6.1415.250) titration vessel with a 6.1903.020 (or 6.1903.030) stirring bar inside on the vessel lid. Fold back the holding bracket upwards while doing so. The markings on the vessel lid and on the plastic ring must be aligned above one another. Afterwards, press the holding bracket downwards in order to fix the titration vessel. The levers of the holding bracket must enclose the pins of the plastic ring on the titration vessel in order to ensure a secure hold.



4 Adjust the height of the KF titration cell by pressing the locking lever. It should almost touch the surface of the stirrer. The position can now be fixed by readjusting the clamping ring.

Once the height of the KF titration cell has been adjusted correctly, the entire cell can be raised and swiveled as required by pressing the locking lever.

Filling the adsorber tube

Before insertion, the 6.1403.040 adsorber tube must be filled with the 6.2811.000 molecular sieve. Proceed as follows:

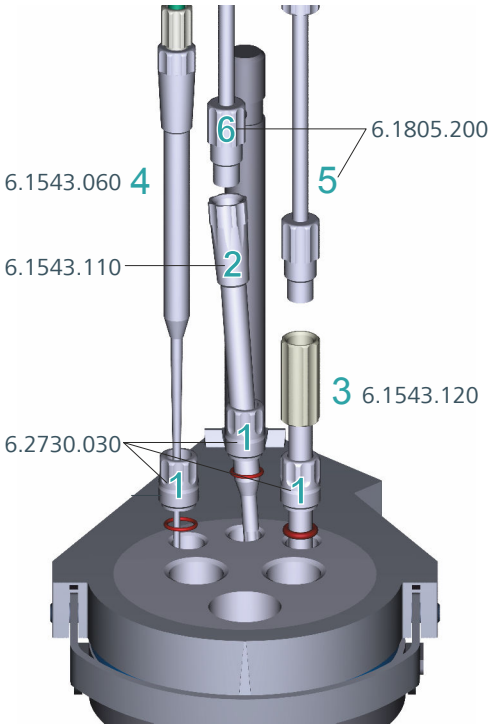
1 Insert a small cotton plug into the bottom of the adsorber tube. Do not pack the cotton too tightly.

2 Fill the molecular sieve up to the ¾ level.

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.

Inserting the dosing tip, aspiration tip and buret tip



Proceed as follows:

1 Place the three screw nipples from 6.2730.030 (including O-rings, but without stoppers) in the rear openings of the vessel lid.

2 Insert the 6.1543.110 dosing tip through the screw nipple in the middle rear opening.

3 Insert the 6.1543.120 aspiration tip through the screw nipple in the right rear opening.

When solvent is aspirated, the end of the aspiration tip must touch the vessel base, but it must not inhibit the action of the stirring bar.

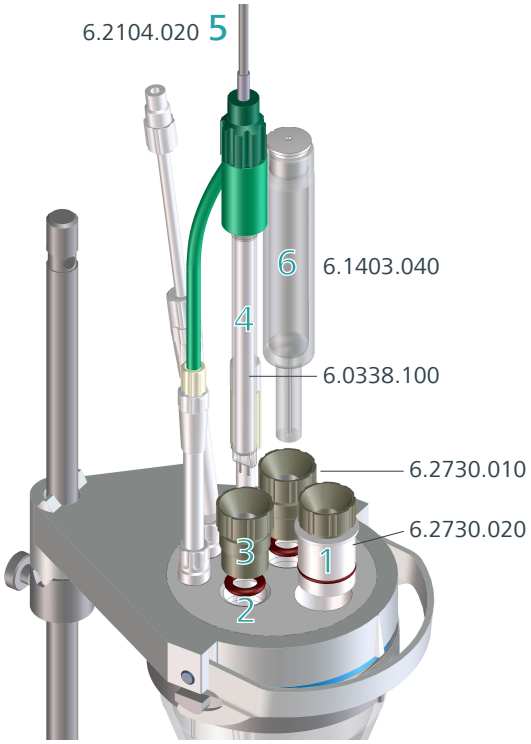
The aspiration tip can, if needed, be pulled out of the solvent.

4 Insert the 6.1543.060 buret tip through the screw nipple in the left rear opening.

5 Screw the 6.1805.200 PTFE M8 tubing of the aspiration bottle onto the aspiration tip.

6 Screw the 6.1805.200 PTFE M8 tubing of the solvent bottle onto the dosing tip.

Inserting electrode, adsorber tube and septum stoppers



Proceed as follows:

1 Introduce the 6.2730.020 septum stopper (with septum inserted) into the front opening of the vessel lid.

2 Insert the O-rings of the electrode and of the adsorber tube into the middle openings of the vessel lid.

3 Screw the two 6.2730.010 screw nipples into the openings with the O-rings. Do not screw too tightly.

4 Introduce the 6.0338.100 double Pt electrode into the left-hand opening and then tighten the screw nipple until it seals.

5 Screw the 6.2104.020 electrode cable tightly onto the electrode.

6 Insert the filled 6.1403.040 adsorber tube on the right of the electrode into the remaining opening and then tighten the screw nipple until it seals.

3.6 Connecting sensors

The measuring interface contains the following measuring inputs:

- Measuring interface 1:
  - **Gen.** for a generator electrode
  - **Ind.** for a double Pt electrode
  - **Temp.** for a temperature sensor (Pt1000 or NTC)
- Measuring interface 2:
  - **Temp.** for a temperature sensor (Pt1000 or NTC)
  - **Pol.** for a polarizable electrode

3.6.1 Connecting a generator electrode (KFC)

Connect the generator electrode as follows:

1 Plug the electrode plug into the **Gen.** socket of the 852 Titrando.

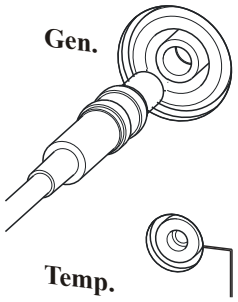


Figure 20 Connecting a generator electrode

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.

3.6.2 Connecting an indicator electrode (KFC)

Connect the indicator electrode as follows:

1 Plug the electrode plug into the **Ind.** socket of the 852 Titrando.

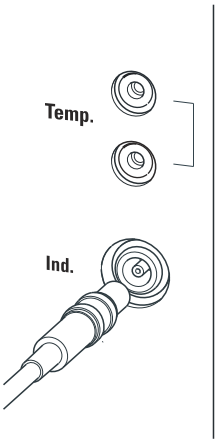


Figure 21 Connecting an indicator electrode

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.

3.6.3 Connecting a polarizable electrode (KFT)

Connect the polarizable electrode as follows:

1 Plug the electrode plug into the **Pol.** socket of the 852 Titrando.

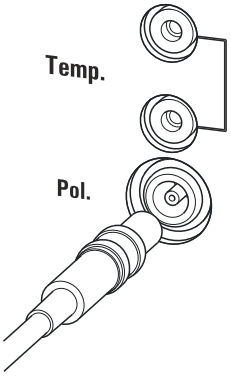


Figure 22 Connecting a polarizable electrode

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.

3.6.4 Connecting a temperature sensor

A temperature sensor of the Pt1000 or NTC 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 852 Titrando.

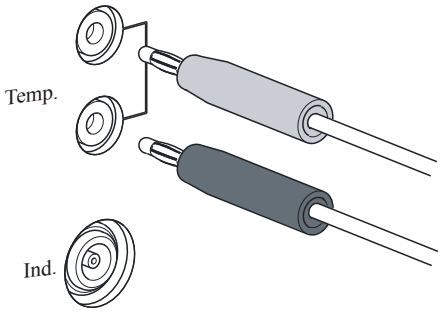


Figure 23 Connecting a temperature sensor

NOTICE

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

4 Karl Fischer titration

4.1 Coulometric titration

4.1.1 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.

4.1.2 Working with water standards

4.1.2.1 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

4.1.2.2 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 852 Titrando is stable, take the syringe in your hand, press **[START]** and inject approx. 1 mL of the water standard through the septum.

There are 2 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 If you have connected a balance to the Titrando, 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.

4.1.3 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

4.1.3.1 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%	10–100 mg	100–1,000 µg
1,000 ppm = 0.1%	100 mg–1 g	100–1,000 µg
100 ppm = 0.01%	1 g	100 µg
10 ppm = 0.001%	5 g	50 µg

4.1.3.2 Working with liquid samples

Liquid samples are added with a syringe. The samples can be injected 2 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.

4.1.3.3 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.

4.1.4 Optimum working conditions

4.1.4.1 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.

4.1.4.2 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.

4.1.4.3 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.

4.1.4.4 Indicator electrode

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.

4.2 Volumetric titration

4.2.1 Principle of the volumetric Karl Fischer titration

The **volumetric Karl Fischer titration** is the classic method of water content determination. It 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, I_2 reacts quantitatively with H_2O. This chemical equation serves as a basis for the water content determination.

The classic Karl Fischer method has undergone continuous further development in recent years. This development did not concern itself solely with the refining and automation of reagent dosing, but also with improving endpoint indication and reagents. The disadvantage of this method is that the reagents are not completely stable. This means that the titer has to be re-determined on a regular basis.

4.2.2 Endpoint determination

The titration endpoint is determined by an electrometric indication method. Double Pt wire electrodes or double Pt ring electrodes are used for this purpose. A distinction is made between the following two indication methods:

Biamperometric indication (Upol)

A constant potential is applied to the electrodes and the resulting current is measured.

Bivoltametric indication (Ipol)

A direct or alternating current is applied between the two electrodes and the resulting potential is measured.

4.2.3 Karl Fischer reagents

One-component reagents

They contain all the reactive parts in a single solution – iodine, sulfur dioxide and a base, dissolved in an appropriate alcohol.

Two-component reagents

The reactive parts are distributed among two separated solutions. The titration reagent contains iodine in methanol. The KF solvent is a solution of sulfur dioxide and a base in methanol. It is used as a working medium in the KF titration cell.

4.2.4 Application of the Karl Fischer titration

The volumetric Karl Fischer titration is the method of choice for determining quantities of water between 0.1 and 100%. It has the advantage that even solid and pasty samples can be added directly to the titration vessel. In addition, various organic solvents can be used that are tailored to the respective samples.

4.2.5 Working with water standards

4.2.5.1 Certified water standards

Commercially available, certified water standards with a water content of 10.0 ± 0.1 mg/g should be used for validating the instrument as a whole, integrated system.

4.2.5.2 Practical recommendations

(see chapter 4.1.2.2, page 43)

4.2.6 Sample addition

This chapter contains a few notes concerning sample addition. Further notes can be found in the publications of the reagent manufacturers and in the Karl Fischer monograph published by Metrohm.

4.2.6.1 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 in order to keep the titration time short. However, ensure that the sample contains at least 50 µg of H_2O. The following tables provide clues for the sample size.

Table 3 Approximate sample size in grams (5 mL buret)

Water content of the sample	KF reagent 1	KF reagent 2	KF reagent 5
0.5%	0.1–0.9	0.2–1.8	0.5–4.5

Water content of the sample	KF reagent 1	KF reagent 2	KF reagent 5
1.0%	0.05–0.45	0.1–0.9	0.25–2.25
5.0%		0.02–0.18	0.05–0.45
10.0%			0.03–0.22
25.0%			
50.0%			

Table 4 Approximate sample size in grams (10 mL buret)

Water content of the sample	KF reagent 1	KF reagent 2	KF reagent 5
0.5%	0.2–1.8	0.4–3.6	
1.0%	0.1–0.9	0.2–1.8	0.5–4.5
5.0%	0.02–0.18	0.04–0.36	0.1–0.9
10.0%		0.02–0.18	0.05–0.45
25.0%			0.02–0.18
50.0%			0.02–0.09

Table 5 Approximate sample size in grams (20 mL buret)

Water content of the sample	KF reagent 1	KF reagent 2	KF reagent 5
0.5%	0.4–3.6		
1.0%	0.2–1.8	0.4–3.6	
5.0%	0.04–0.36	0.08–0.72	0.2–1.8
10.0%	0.02–0.18	0.04–0.36	0.1–0.9
25.0%		0.02–0.14	0.04–0.36
50.0%			0.02–0.18

KF reagent 1: 1 mL KF reagent reacts with around 1 mg H_2O

KF reagent 2: 1 mL KF reagent reacts with around 2 mg H_2O

KF reagent 5: 1 mL KF reagent reacts with around 5 mg H_2O

4.2.6.2 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 form. 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 blank value.

Viscous samples can be added to the measuring cell with a syringe without needle. You can use the ground-joint opening for this. The best way for you to determine the added sample amount is by reweighing the sample.

4.2.6.3 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 have to be directly added to the titration cell, they can be inserted through the ground-joint 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 surface of the electrode is not covered by the sample substance (incomplete KF reaction!).
- the Pt wires of the indicator electrode do not become damaged.

4.2.7 Optimum working conditions

4.2.7.1 Drift

A constant drift in the range of ≤ 10 µL/min is all right. Lower values are, however, quite possible. Higher but stable values will still produce good results, because the drift can be compensated.

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

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

4.2.7.2 Reagent replacement

The electrolyte solution must be replaced in the following cases:

- The titration vessel is too full.
- The drift is too high, and cannot be reduced by shaking the titration vessel.

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 vessel does not have to be disassembled.

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

4.2.7.3 Indicator electrode

A new indicator electrode can take a certain warm-up time for forming the surface. During this time unexpectedly long titration times and high measurement results can occur. This phenomenon will, however, disappear after a short time of use. In order to accelerate the setting of a new indicator electrode, the 852 Titrando 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 and maintenance

5.1 General notes

5.1.1 Care

The 852 Titrando requires appropriate care. Excess contamination of the instrument may result in functional disruptions and a reduction in the service life of the otherwise sturdy mechanics and electronics.

Spilled chemicals and solvents should be removed immediately. Above all, the plug connections on the rear of the instrument (in particular the power socket) should be protected from contamination.

CAUTION

Although this is extensively prevented by design measures, the power plug should be unplugged immediately if aggressive media have penetrated the inside of the instrument, so as to avoid serious damage to the instrument electronics. In such cases, Metrohm Service must be informed.

5.1.2 Maintenance by Metrohm Service

Maintenance of the 852 Titrando is best carried out as part of an annual service, which is performed by specialist personnel of the Metrohm company. A shorter maintenance interval may be necessary if you frequently work with caustic and corrosive chemicals.

Metrohm Service offers every form of technical advice for maintenance and service of all Metrohm instruments.

5.2 Generator electrode

5.2.1 Generator electrode without diaphragm

The 6.0345.100 generator electrode without diaphragm is easy to handle and clean. It needs only one reagent and is quickly ready for use (no moisture deposits in the diaphragm!). The generator electrode without diaphragm is suitable for most applications. It should be specifically used for severely contaminated samples.

5.2.1.1 Reagents

Use only reagents that are specifically intended for generator electrodes without diaphragms. Details can be found in the documentation of the reagent manufacturers.

5.2.1.2 Cleaning

As a rule, the electrolyte solution can be exchanged without special cleaning of the parts. If cleaning is necessary anyway, ensure that the Pt grid of the generator electrode is not damaged.

- **Contaminations containing oil**
  First clean with a solvent (e.g. hexane), then rinse with ethanol.
- **Saline depositions**
  First clean with water, then rinse with ethanol.

Thoroughly dry all parts after cleaning. A hair dryer can be used for this. If the parts are dried in the drying oven the temperature must not exceed 70 °C (plastic parts!).

5.2.2 Generator electrode with diaphragm

The 6.0344.100 generator electrode with diaphragm should be used for samples containing ketones, because special reagents for ketones are only available for generator electrodes with diaphragm. If your reagent has a low conductivity, e.g. because you have to add chloroform to the sample due to its solubility, you should prefer the generator electrode with diaphragm. It is also recommended when you have to rely on a high accuracy in the lowest trace range.

5.2.2.1 Reagents

Reagents for the coulometric water content determination with generator electrodes with diaphragm consist of the anode solution (anolyte), which is filled in the titration vessel, and the cathode solution (catholyte), which is filled in the generator electrode.

For the water content determinations in ketones, special reagents have to be used (see the documentation of the reagent manufacturers).

5.2.2.2 Cleaning

As a rule, the electrolyte solution can be exchanged without special cleaning of the parts. If cleaning is necessary anyway, ensure that the Pt grid of the generator electrode is not damaged.

- **Resinous residues on the diaphragm**
  Hang the generator electrode vertically on a support rod, fill with concentrated HNO_3 and leave overnight. First rinse with water, then with ethanol.
- **Contaminations containing oil**
  First clean with a solvent (e.g. hexane), then rinse with ethanol.
- **Saline depositions**
  First clean with water, then rinse with ethanol.
- **Cleaning (rinsing) the diaphragm**
  Fill the cathode chamber of the generator electrode with methanol and let the contents flow out. Repeat this procedure two or three times. This procedure should also be carried out after the cleaning described above.

Thoroughly dry all parts after cleaning. A hair dryer can be used for this. If the parts are dried in the drying oven the temperature must not exceed 70 °C (plastic parts!).

6 Troubleshooting

6.1 General

Problem	Cause	Remedy
The "On" LED is not illuminated, even though the instrument is connected to the power supply.	*The Touch Control or the computer has not been switched on yet or the plugs are not correctly plugged in.*	1. Check the plug connections. 2. Switch on the Touch Control or the computer.

6.2 Karl Fischer titration

Problem	Cause	Remedy
The drift is very high during conditioning.	*The titration cell is leaking.*	▪ Check the seals and the septum. Replace if necessary. ▪ Replace the molecular sieve.
The drift becomes greater after each titration.	*The sample releases water very slowly.*	▪ Adjust the method. ▪ Add solubility promoter. ▪ Increase the temperature (possibly using a KF oven). ▪ See technical literature.
	A side reaction is taking place.	▪ Use special reagents. ▪ Adjust the method (increase/decrease the temperature, external extraction). ▪ See technical literature.
	The pH value is no longer in the optimum range.	Add buffer (see technical literature).
The titration will not be finished.	*The titration cell is leaking.*	▪ Check the seals and the septum. Replace if necessary. ▪ Replace the molecular sieve.
	The minimum increment is too low.	Select the user-defined titration rate and increase the minimum volume increment (see manual/help of the software used).
	The stop criterion is unsuitable.	Adjust the control parameters (see manual/help of the software used): ▪ Increase the stop drift.

Problem	Cause	Remedy
		▪ Select a short delay time.
	See also: The drift becomes greater after each titration.	
The sample is over-titrated.	*The increments at the end of the titration are too high.*	▪ Select the user-defined titration rate and reduce the dosing rate (see manual/help of the software used). The following experiment provides a clue for the optimum dosing rate: During conditioning, display the drift and add sample without starting the titration. Select a value below the highest drift as dosing rate. ▪ Stir faster.
	The amount of methanol in the working medium is too low.	▪ Replace the working medium. ▪ Reduce the amount of solubility promoter, if working with solvent mixtures, see technical literature.
	The electrode may be covered.	Wipe off the electrode with ethanol or a suitable solvent.
The solution becomes darker after each titration.		Replace the working medium.
	The electrode may be covered.	Wipe off the electrode with ethanol or a suitable solvent.
	The electrode has a short circuit.	1. Check the Pt wires. 2. Activate the electrode check.
The endpoint is reached too quickly.	*The dosing rate outside the control range is too high.*	Select the user-defined titration rate and reduce the dosing rate (see manual/help of the software used).
The titration times with volumetric titration are constantly longer.	*The buffer capacity of the solvent may be exhausted with two-component reagents.*	Replace the working medium.

6.3 SET titration

Problem	Cause	Remedy
The titration will not be finished.	*The minimum dosing rate is too low.*	Select the user-defined titration rate and increase the minimum rate (see manual/help of the software used).
	The stop criterion is unsuitable.	Adjust the control parameters (see manual/help of the software used): ▪ Increase the stop drift. ▪ Select a short delay time.
The sample is overtitrated.	*The control parameters are unsuitable.*	Adjust the control parameters (see manual/help of the software used): ▪ Select Titration rate = **slow**. ▪ Select the user-defined titration rate and increase the control range. ▪ Select the user-defined titration rate and reduce the maximum rate. ▪ Select the user-defined titration rate and reduce the minimum rate. ▪ Stir faster. ▪ Arrange the electrode and buret tip to an optimum.
	The electrode responds too slowly.	Replace the electrode.
The titration time is too long.	*The control parameters are unsuitable.*	Adjust the control parameters (see manual/help of the software used): ▪ Select Titration rate = **optimal** or **fast**. ▪ Select the user-defined titration rate and decrease the control range. ▪ Select the user-defined titration rate and increase the maximum rate. ▪ Select the user-defined titration rate and increase the minimum rate.
The results are spread widely.	*The minimum dosing rate is too high.*	Select user-defined titration rate and decrease the minimum rate (see manual/help of the software used).
	The electrode responds too slowly.	Replace the electrode.

7 Appendix

7.1 Remote interface

The 6.2148.010 Remote Box allows devices to be controlled which cannot be connected directly to the MSB interface of the Titrando.

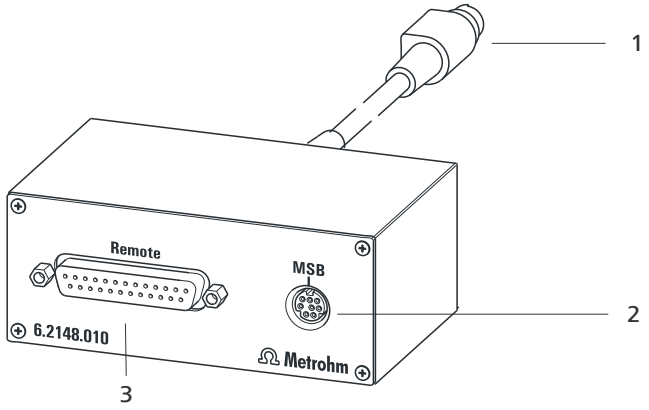


Figure 24 Connectors of the Remote Box

1 Cable
For connecting to the Titrando.

2 MSB connector
Metrohm Serial Bus. For connecting external dosing devices or stirrers.

3 Remote connector
For connecting instruments with a remote interface.

7.1.1 Pin assignment of the remote interface

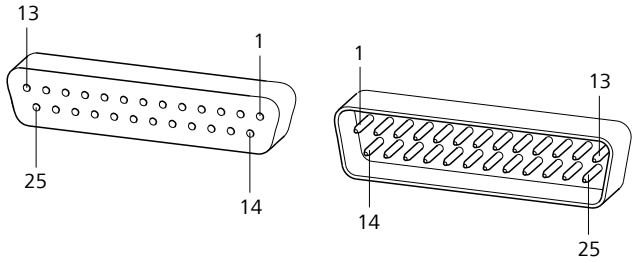
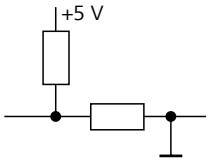


Figure 25 Pin assignment of remote socket and remote plug

The above figure of the pin assignment applies for all Metrohm instruments with 25-pin D-Sub remote connector.

Inputs

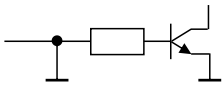


approx. 50 kΩ Pull-up

t_p > 20 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

The following tables offer information concerning the assignment of the individual pins and their function:

Table 6 Inputs and outputs of the remote interface

Assignment	Pin No.	Function*
Input 0	21	**Start**
Input 1	9	**Stop**
Input 2	22	
Input 3	10	**Quit**
Input 4	23	**–**
Input 5	11	
Input 6	24	
Input 7	12	
Output 0	5	**Ready**
Output 1	18	**Conditioning OK**
Output 2	4	**Determination**
Output 3	17	**EOD**
Output 4	3	
Output 5	16	**Error**
Output 6	1	
Output 7	2	**Warning**

Assignment	Pin No.	Function*
Output 8	6	
Output 9	7	
Output 10	8	
Output 11	13	
Output 12	19	
Output 13	20	
0 volts / GND	14	
+5 volts	15	
0 volts / GND	25	

* Signal activated only for operation with Touch Control.

Table 7 Explanation of the individual functions

Function	Explanation
Start	The current method is started at the time of activation. t_{pulse} > 100 ms
Stop	The current method is canceled (Stop) at the time of activation. t_{pulse} > 100 ms
Quit	The current command in the determination run will be canceled at the time of activation. t_{pulse} > 100 ms
Ready	The instrument is ready to receive a start signal.
Conditioning OK	The line is set when Conditioning with SET titration and KFT titration is at OK. The line remains set until the determination is started with **[START]**.
Determination	The instrument performs a data-generating determination.
EOD	**E**nd **o**f **D**etermination. Pulse (t_{pulse} = 200 ms) after a determination or after a buffer/standard solution during calibration using a Sample Processor.
Error	The line is set for error message display.

Function	Explanation
Warning	The line is set for warning message display.

8 Technical specifications

8.1 Measuring interface

The measuring cycle is 100 ms for all measuring modes.

8.1.1 Generator electrode

One measuring input (**Gen.**) for a generator electrode.

I_{max}	400 mA Continuous current and pulsed

8.1.2 Indicator electrode

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

Measuring mode Ipol	Determination with adjustable polarization current
AC	5, 10, 20 and 30 µA
DC	–125 to +125 µA

8.1.3 Temperature

A measuring input (**Temp.**) for temperature sensors of the Pt1000 or NTC type with automatic temperature compensation.

R (25 °C) and B value can be configured for NTC sensors.

Measuring range	
Pt1000	–150 to +250 °C
NTC	–5 to +250 °C (R (25 °C) = 30,000 Ω and B (25/50) = 4,100 K)
Resolution	
Pt1000	0.1 °C
NTC	0.1 °C
Measuring accuracy	
Pt1000	±0.2 °C (Applies for measuring range –20 to +150 °C; ±1 digit; without sensor error, under reference conditions)
NTC	±0.6 °C (Applies for measuring range +10 to +40 °C; ±1 digit; without sensor error, under reference conditions)

8.1.4 Polarizer

	One measuring input (**Pol.**) for polarizable electrodes.
Measuring mode Ipol	Determination with adjustable polarization current
Polarization current	–122.5 to +122.5 µA (increment: 0.5 µA) –125.0 to +125.0 µA: non-guaranteed values, dependent on reference voltage +2.5 V
Measuring range	–1,200 to +1,200 mV
Resolution	0.1 mV
Measuring accuracy	±0.2 mV (±1 digit, without sensor error, under reference conditions)
Measuring mode Upol	Determination with adjustable polarization voltage
Polarization voltage	–1,225 to +1,225 mV (increment: 25 mV) –1,250 to +1,250 mV: non-guaranteed values, dependent on reference voltage +2.5 V
Measuring range	–120 to +120 µA
Resolution	0.1 µA

8.2 Power connection

Supply voltage	100–240 V
Frequency	50–60 Hz
Power consumption	Maximum 45 W
Fuse	Electronic overload protection

8.3 Ambient temperature

Nominal function range	+5 to +45 °C at max. 80% relative humidity non-condensing
Storage	+5 to +45 °C

8.4 Dimensions

Width	142 mm
Height	227 mm
Depth	231 mm
Weight	3.1 kg (without accessories)
Material (housing)	Poly(butylene terephthalate) (PBT)

8.5 Interfaces

USB connectors

USB ports	2 USB downstream ports (type A sockets), 500 mA each, for connecting peripheral devices such as printers, keyboards, barcode readers or RS-232/USB boxes (Metrohm order no. 6.2148.020).

"Controller" connector

Controller port	USB upstream port with auxiliary power supply (Mini DIN socket) for connecting Touch Control or computer for controlling the 852 Titrando.
Touch Control	With integrated Touch Control cable.
Computer	With 6.2151.000 cable.

MSB connectors (Metrohm Serial Bus)

Dosing device	Connection of a maximum of 4 external dosing devices, models Dosimat or Dosino (MSB 1 to MSB 4).
Stirrer	Connection of a maximum of 4 stirrers. Stirrer control: Switching on/off manually or coordinated with the titration sequence. Speed in 15 steps and shift direction can be selected.
Remote Box	Connection of a maximum of 4 Remote Boxes. Remote Boxes can be used to actuate and monitor external devices.

9 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. **852**) 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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