

# 851 Titrande



## Manual

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

# **851 Titrando**

## **Manual**

Technical Communication  
Metrohm AG  
CH-9100 Herisau

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# Table of contents

<b>1</b>	<b>Introduction</b>	<b>1</b>
1.1	The Titrand system .....	1
1.2	Device description .....	2
1.3	Titration modes – Measuring modes – Dosing commands .....	2
1.4	Displaying accessories .....	3
1.5	Symbols and conventions .....	4
<b>2</b>	<b>Safety</b>	<b>6</b>
2.1	Intended use .....	6
2.2	Responsibility of the operator .....	6
2.3	Personnel requirement .....	7
2.4	Safety instructions .....	7
2.4.1	Electrical safety .....	7
2.4.2	Tubing and capillary connections .....	8
2.4.3	Flammable solvents and chemicals .....	8
<b>3</b>	<b>Overview of the instrument</b>	<b>9</b>
<b>4</b>	<b>Installation</b>	<b>11</b>
4.1	Setting up the device .....	11
4.1.1	Packaging .....	11
4.1.2	Checks .....	11
4.1.3	Location .....	11
4.2	Connecting a controller .....	11
4.2.1	Operation .....	11
4.3	Connecting MSB devices .....	16
4.3.1	Connecting a dosing device .....	17
4.3.2	Connecting a stirrer or titration stand .....	18
4.3.3	Connecting a Remote Box .....	19
4.4	Connecting USB devices .....	20
4.4.1	General .....	20
4.4.2	Connecting a USB hub .....	21
4.4.3	Connecting a printer .....	21
4.4.4	Connecting a balance .....	22
4.4.5	Connecting a keyboard (only for operation with Touch Control) .....	23
4.4.6	Connecting a barcode reader .....	24

<b>4.5</b>	<b>Titration vessel for coulometric KF titration</b>	<b>25</b>
4.5.1	Mounting the titration cell (coulometry)	25
4.5.2	Titration cell (coulometry) – standard configuration	26
4.5.3	Titration cell (coulometry) with addition and aspiration tube – Utilization with Ti Stand	30
4.5.4	Titration cell (coulometry) with equipment for reagent replacement – Utilization with Dosino	31
4.5.5	Titration cell (coulometry) with Karl Fischer oven	32
4.5.6	Titration cell (coulometry) with sample changer	32
<b>4.6</b>	<b>Connecting sensors</b>	<b>33</b>
4.6.1	Connecting a generator electrode	33
4.6.2	Connecting an indicator electrode	33
4.6.3	Connecting a temperature sensor	34
<b>5</b>	<b>Coulometric titration</b>	<b>35</b>
<b>5.1</b>	<b>Principle of coulometry according to Karl Fischer</b>	<b>35</b>
<b>5.2</b>	<b>Working with water standards</b>	<b>36</b>
5.2.1	Certified water standards	36
5.2.2	Practical recommendations	36
<b>5.3</b>	<b>Sample addition</b>	<b>38</b>
5.3.1	Size of the sample size	38
5.3.2	Working with liquid samples	38
5.3.3	Working with solid samples	39
<b>5.4</b>	<b>Optimum working conditions</b>	<b>40</b>
5.4.1	General	40
5.4.2	Drift	40
5.4.3	Reagent replacement	41
5.4.4	Indicator electrode	41
<b>6</b>	<b>Operation and maintenance</b>	<b>42</b>
<b>6.1</b>	<b>General notes</b>	<b>42</b>
6.1.1	Care	42
6.1.2	Maintenance by Metrohm Service	42
<b>6.2</b>	<b>Generator electrode</b>	<b>43</b>
6.2.1	Generator electrode without diaphragm	43
6.2.2	Generator electrode with diaphragm	43
<b>7</b>	<b>Troubleshooting</b>	<b>45</b>
<b>7.1</b>	<b>General</b>	<b>45</b>
<b>7.2</b>	<b>Karl Fischer titration</b>	<b>45</b>
7.2.1		45
<b>8</b>	<b>Appendix</b>	<b>47</b>
<b>8.1</b>	<b>Remote interface</b>	<b>47</b>
8.1.1	Pin assignment of the remote interface	47

<b>9 Recycling and disposal</b>	<b>51</b>
<b>10 Technical specifications</b>	<b>52</b>
<b>10.1 Measuring interface</b> .....	<b>52</b>
10.1.1 Generator electrode .....	52
10.1.2 Indicator electrode .....	52
10.1.3 Temperature .....	52
<b>10.2 Power connection</b> .....	<b>53</b>
<b>10.3 Ambient conditions</b> .....	<b>53</b>
<b>10.4 Dimensions</b> .....	<b>53</b>
<b>10.5 Interfaces</b> .....	<b>54</b>
<b>Index</b>	<b>55</b>



## Table of figures

Figure 1	The Titrand system .....	1
Figure 2	Front 851 Titrand .....	9
Figure 3	Rear 851 Titrand .....	10
Figure 4	Connecting the Touch Control .....	12
Figure 5	Connecting the computer .....	14
Figure 6	MSB connections .....	16
Figure 7	Connecting a dosing device .....	18
Figure 8	Connecting an MSB stirrer .....	19
Figure 9	Connecting the propeller stirrer to the titration stand .....	19
Figure 10	Connecting the Remote Box .....	20
Figure 11	Connecting a printer .....	22
Figure 12	Mounting the titration cell (coulometry) .....	25
Figure 13	Filling the adsorber tube .....	26
Figure 14	Equipping the titration cell (coulometry) .....	27
Figure 15	Unscrewing the lid from the indicator electrode .....	29
Figure 16	Unscrewing the lid from the generator electrode .....	29
Figure 17	Screwing the electrode cable to the electrodes .....	29
Figure 18	Mounting the addition and aspiration tube .....	30
Figure 19	Connect generator electrode .....	33
Figure 20	Connect indicator electrode .....	34
Figure 21	Connecting a temperature sensor .....	34
Figure 22	Connectors of the Remote Box .....	47
Figure 23	Pin assignment of remote socket and remote plug .....	47

# 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 devices. The following figure provides an overview of the peripheral devices you can connect to the 851 Titrando.

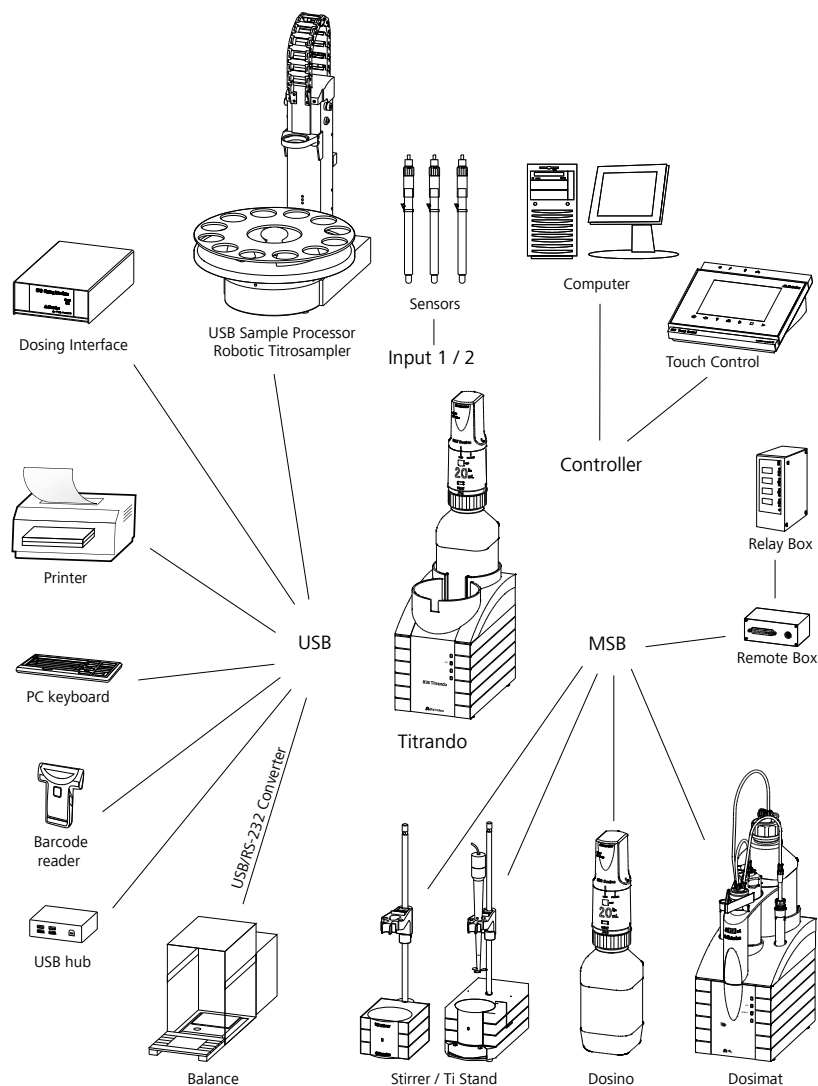


Figure 1 The Titrando system




- **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:
  - **T** (temperature measurement)
- **Dosing commands**  
The following commands for dosing can be selected:
  - **PREP** (rinse the cylinder and tubings)
  - **EMPTY** (empty the cylinder and tubings)
  - **ADD** (dosing a specified volume)
  - **LQH** (carrying out complex dosing tasks with a Dosino)

## 1.4 Displaying accessories

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

### 1 Searching for a product on the website

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

The search result is displayed.

### 2 Displaying product information

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

Detailed information regarding the product is displayed.

### 3 Displaying accessories and downloading the accessories list







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

**NOTE**

Metrohm recommends keeping the accessories list for reference purposes.

## 1.5 Symbols and conventions

The following symbols and formatting may appear in this documentation:

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



**NOTICE**

This symbol highlights additional information and tips.

---



## 2.3 Personnel requirement

Only qualified personnel may operate the product. Qualified personnel are persons who meet the following requirements:

- Knowledge of basic regulations on occupational safety and accident prevention.
- Knowledge regarding the application of fire prevention measures for laboratories.
- In-depth knowledge of handling hazardous chemicals.
- Personnel have been instructed and are able to operate the product safely and independently recognize and avoid potential dangers.
- The user documentation has been read and understood. The personnel operate the product according to the instructions in the user documentation.

## 2.4 Safety instructions

### 2.4.1 Electrical safety

Electrical safety when working with the device 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 device. The device could become damaged. There is a considerable risk of injury if live components are touched.

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

### Supply voltage



#### WARNING

---

An incorrect supply voltage can damage the device.

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

### 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 setting up or disconnecting electrical plug connections at the rear of the device.

## 2.4.2 Tubing and capillary connections



#### CAUTION

---

Leaks in tubing connections 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. Suitable tools can be used for disconnecting connections.

The leak-tightness of the connections must be checked regularly. If the device is used mainly in unattended operation, then weekly inspections are mandatory.

## 2.4.3 Flammable solvents and chemicals



#### WARNING

---

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

- Set up the device in a well-ventilated location (e.g., fume cupboard).
- Keep all sources of ignition far from the workplace.
- Clean up spilled liquids and solids immediately.
- Follow the safety instructions of the chemical manufacturer.

### 3 Overview of the instrument

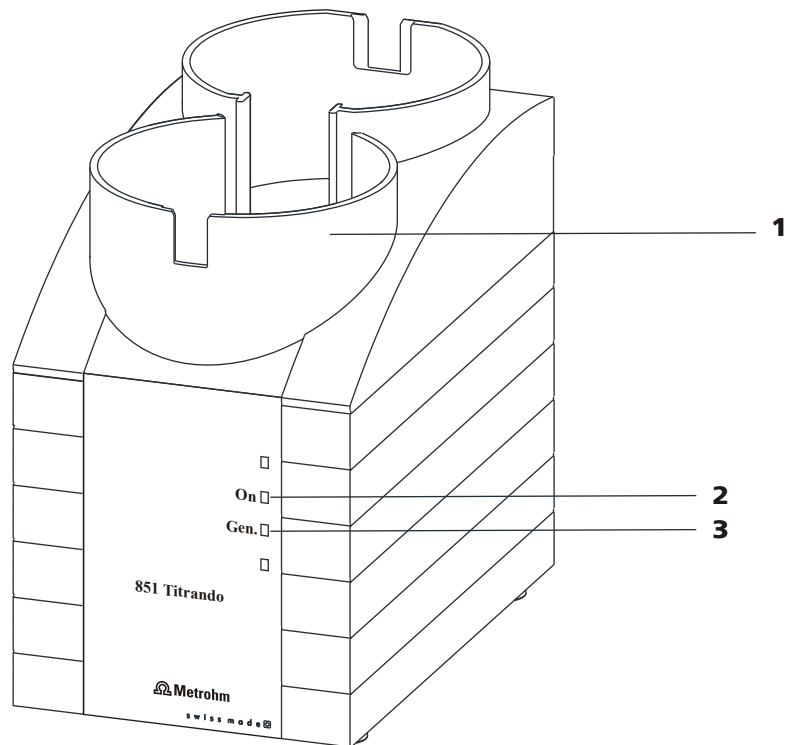


Figure 2 Front 851 Titrando

**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.

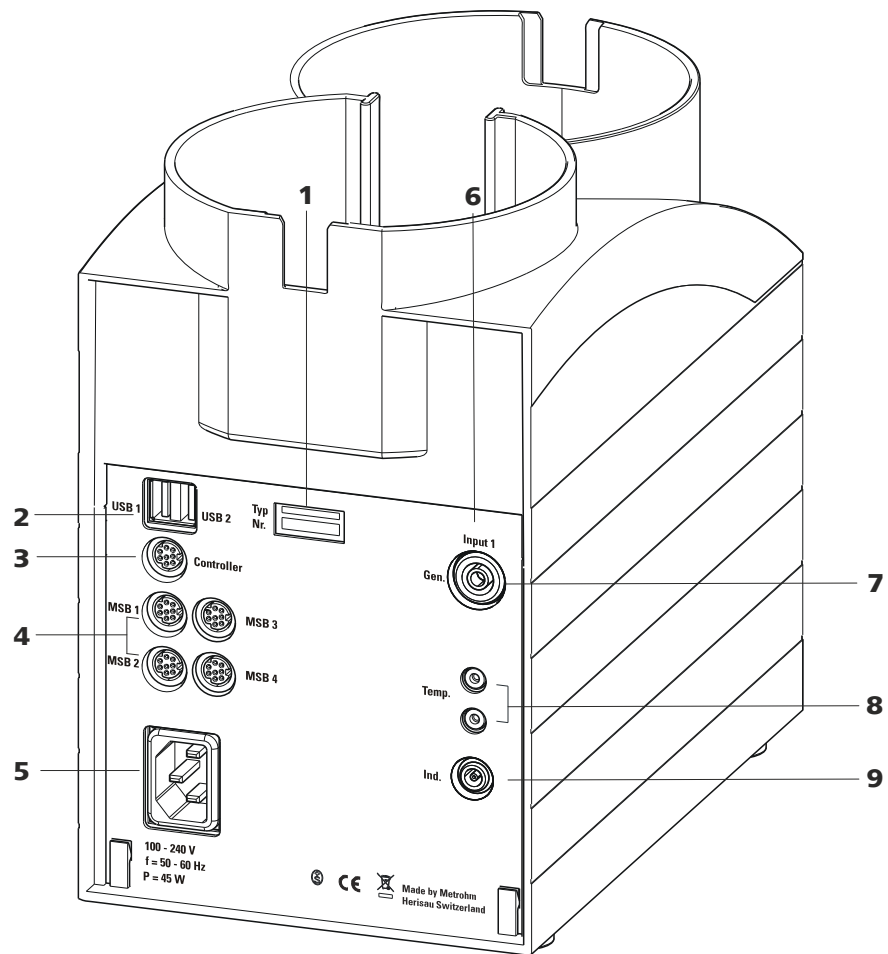


Figure 3 Rear 851 Titrando

**1 Type plate**

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

**3 Connector (Controller)**

For connecting a Touch Control or a PC with installed PC software. Mini DIN, 9-pin.

**5 Power socket**

**7 Electrode connector (Gen.)**

For connecting a generator electrode.

**9 Electrode connector (Ind.)**

For connecting a double Pt electrode. Socket F.

**2 USB connector (USB 1 and USB 2)**

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

**4 MSB connector (MSB 1 to MSB 4)**

Metrohm Serial Bus. For connecting external dosing devices, stirrers or Remote Boxes. Mini DIN, 9-pin.

**6 Measuring interface 1 (Input 1)**

**8 Temperature sensor connector (Temp.)**

For connecting temperature sensors (Pt1000 or NTC). Two B sockets, 2 mm.

## 4 Installation

### 4.1 Setting up the device

#### 4.1.1 Packaging

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

#### 4.1.2 Checks

Inspect the delivery immediately upon receipt:

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

#### 4.1.3 Location

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

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

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

### 4.2 Connecting a controller

#### 4.2.1 Operation

Two different versions are available for operating the 851 Titrando:

- A Touch Control with touch-sensitive screen. It forms a "stand-alone instrument" together with the 851 Titrando.
- A computer enables operation of the 851 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.



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.

#### 4.2.1.2 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<sup>2</sup> / 18 AWG
- Power plug:
  - according to customer requirement (6.2122.XX0)
  - min. 10 A



##### NOTE

Do not use a not permitted power cord!

##### 1 Plugging in the power cord

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



### 3 Follow the instructions of the installation wizard.

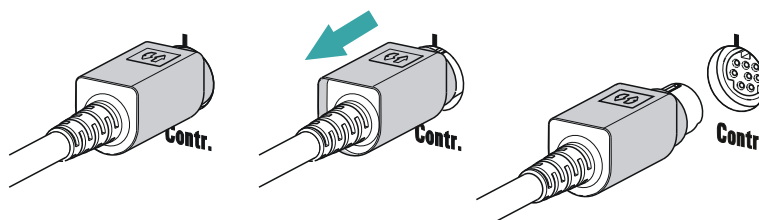
The "On" LED on the 851 Titrando lights up when the driver installation has been completed and the instrument is ready for operation.

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



#### NOTE

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 the instrument according to your requirements.

### 1 Setting up the instrument

- Start up 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 instrument configuration can be found in the documentation for the respective computer software.

## 4.3 Connecting MSB devices

In order to connect MSB devices, e.g., stirrers or dosing devices, Metrohm devices are equipped with up to a maximum of 4 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.

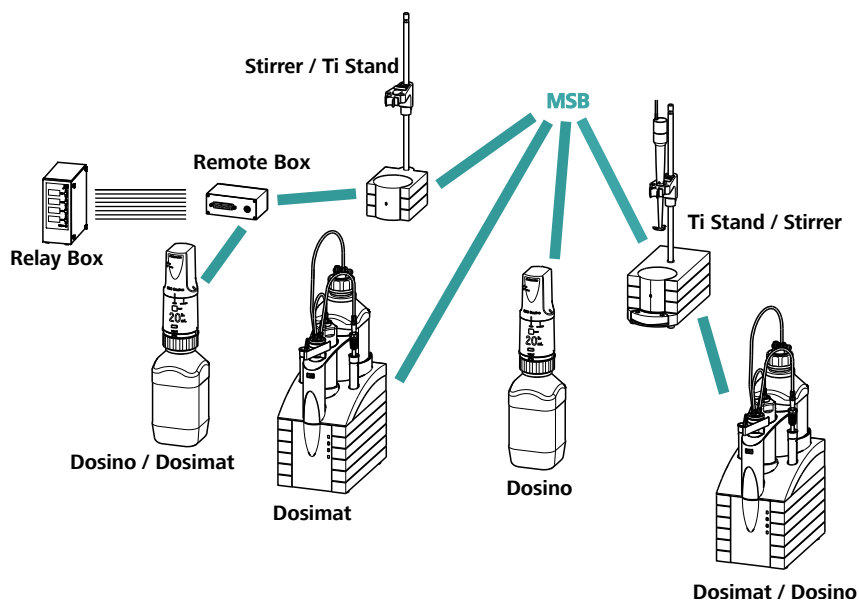


Figure 6 MSB connections

The control instrument determines which peripheral devices are supported.



### NOTE

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

### 4.3.1 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

#### Connecting a dosing device

##### 1 Connecting a dosing device

- Exit the control software.
- Connect the connection cable of the dosing device to 1 of **MSB** sockets on the rear of the control instrument.
- Start the control software.

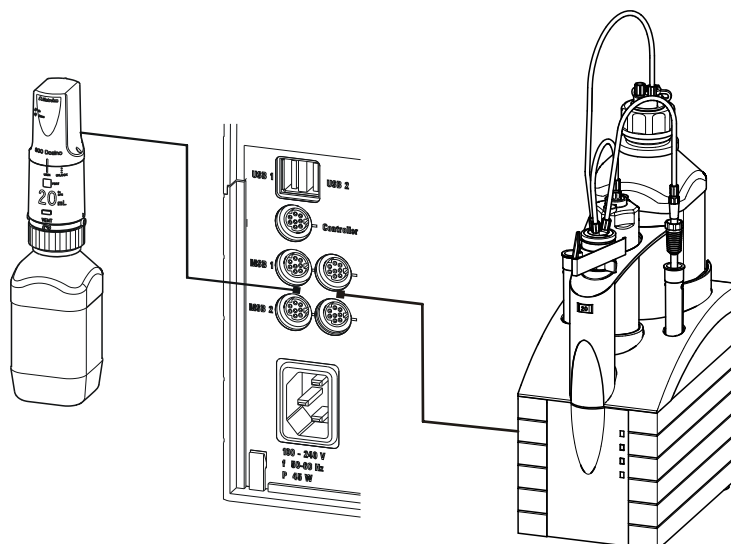


Figure 7 Connecting a dosing device

### 4.3.2 Connecting a stirrer or titration stand

You can use the following devices:

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 propeller stirrer 802 Stirrer

#### Connecting the stirrer or titration stand

- 1 Exit the control software.
- 2 Connect the connection cable of the magnetic stirrer or titration stand to an **MSB** socket on the rear of the control instrument.  
804 Ti Stand only: Connect the propeller stirrer to the stirrer connector (socket with stirrer symbol) of the titration stand.
- 3 Start the control software.

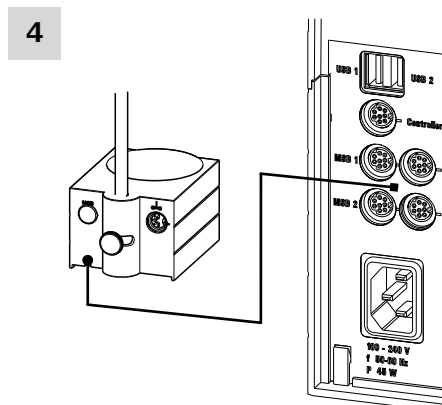


Figure 8 Connecting an MSB stirrer

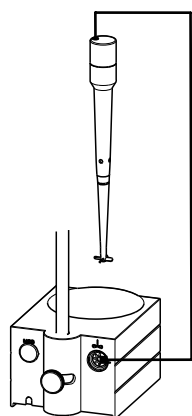


Figure 9 Connecting the propeller stirrer to the titration stand

### 4.3.3 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.

You can connect the following devices 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 pre-

cise information concerning the pin assignment of the interface on the Remote Box in the *Appendix*.

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. Runs on a variety of instruments and can thus be coordinated in a single complex automation system. However, no data exchange is possible.

### Connecting the Remote Box

- 1 Exit the control software.
- 2 Connect the connection cable of the Remote Box to an **MSB** socket on the rear of the control instrument.
- 3 Start the control software.

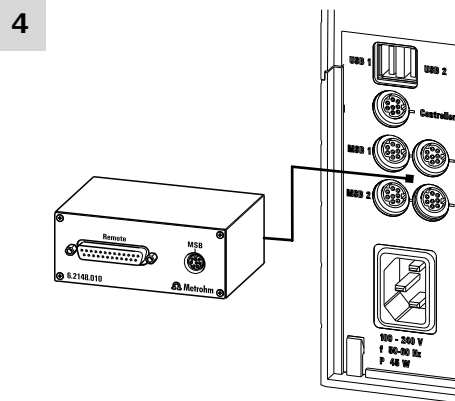


Figure 10 Connecting the Remote Box

## 4.4 Connecting USB devices

### 4.4.1 General

The 851 Titrando has 2 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 2 devices to the USB, you can also use an additional, commercially available USB hub.

**CAUTION**

If you operate the 851 Titrande with the aid of the Touch Control, then take care to ensure that the Touch Control is switched off when you set up or disconnect connections between the various devices. If you use a computer software to control the 851 Titrande, then you should exit the program before you set up or disconnect the USB connections.

**4.4.2 Connecting a USB hub**

If you wish to connect more than 2 devices to the USB connector of the 851 Titrande, then you can also use an additional commercially available USB hub (distributor). If you operate the 851 Titrande with the help of the Touch Control, then you should use a USB hub with its own energy supply.

- 1 Switch off the Touch Control and/or exit the computer software.
- 2 With the aid of the 6.2151.020 cable, connect the USB connector of the 851 Titrande (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.

**4.4.3 Connecting a printer**

Printers that are connected to the 851 Titrande 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.

- 1 Switch off the Touch Control.
- 2 With the aid of the 6.2151.020 cable, connect the USB connector of the 851 Titrande (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).

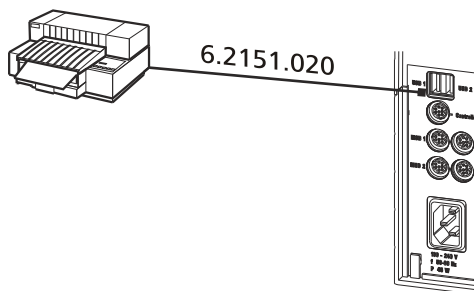


Figure 11 Connecting a printer

#### 4.4.4 Connecting a balance

- Operation with a computer 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 851 Titrando and of which cable you will need for connection to the RS-232 interface:

Balance	Cables
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	Cables
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

- 1** Plug in the USB plug of the USB/RS-232 adapter at the USB connector of the 851 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).

#### 4.4.5 Connecting a keyboard (only for operation with Touch Control)

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

- 1** Insert the USB plug of the keyboard into 1 of the USB port of the 851 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).

#### 4.4.6 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

- 1 Insert the USB plug of the barcode reader into 1 of the USB ports of the 851 Titrande.
- 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:

- 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.

## 4.5 Titration vessel for coulometric KF titration

### 4.5.1 Mounting the titration cell (coulometry)

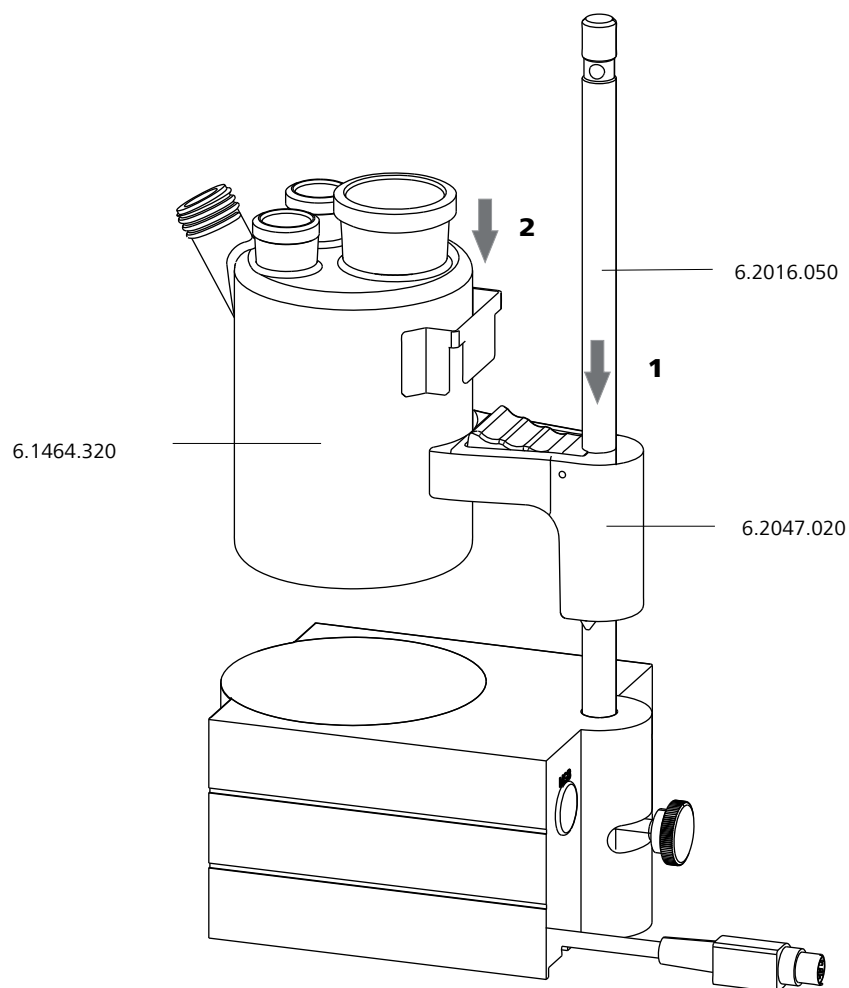


Figure 12 Mounting the titration cell (coulometry)

- 1** Fasten 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.

## 4.5.2 Titration cell (coulometry) – standard configuration

### Filling the adsorber tube

#### Required accessories:

- 6.1403.030 adsorber tube
- 6.2811.000/6.2811.010 molecular sieve

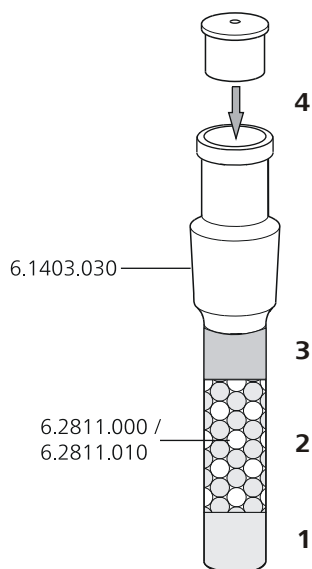


Figure 13 Filling the adsorber tube

- 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  $\frac{3}{4}$  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 associated lid.



#### NOTE

The molecular sieve must be replaced at regular intervals. Each time the adsorber tube is filled, write the date directly on the adsorber tube, for example.

### Equipping the titration cell (coulometry)

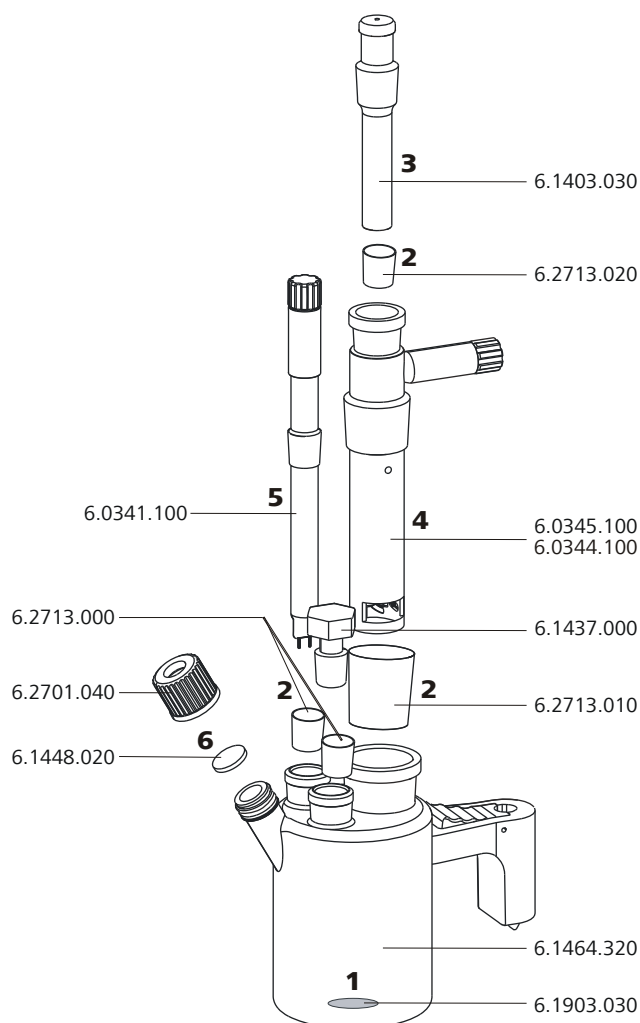


Figure 14 Equipping the titration cell (coulometry)

- 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.).

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 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 for it to seal. The septum must not bend.

#### **Filling the titration cell (coulometry) – Generator electrode with diaphragm**

- 1 Fill approximately 5 mL of catholyte into the generator electrode.
- 2 Fill approximately 100 mL of anolyte into the titration cell using the 6.2738.000 funnel. The level of the anolyte should be roughly 1 to 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 (coulometry) – Generator electrode without diaphragm**

- 1 Fill approximately 100 mL of reagent into the titration cell using 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**

- 1 Unscrew the lid of the indicator electrode.

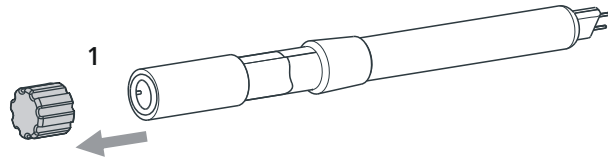


Figure 15 Unscrewing the lid from the indicator electrode

**2** Unscrew the lid of the generator electrode.

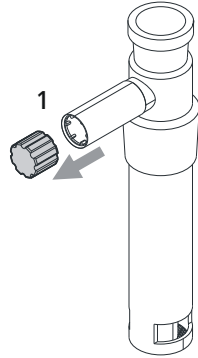


Figure 16 Unscrewing the lid from the generator electrode

**3** Tighten the 6.2104.020 electrode cable to the indicator electrode.

**4** Tighten the 6.2104.120 electrode cable to the generator electrode.

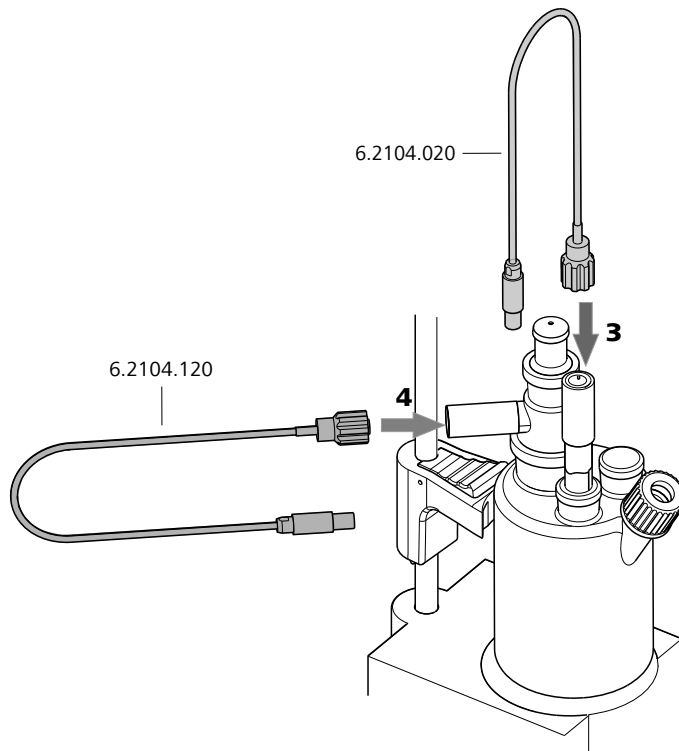


Figure 17 Screwing the electrode cable to the electrodes

**NOTE**

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

### 4.5.3 Titration cell (coulometry) with addition and aspiration tube – Utilization with Ti Stand

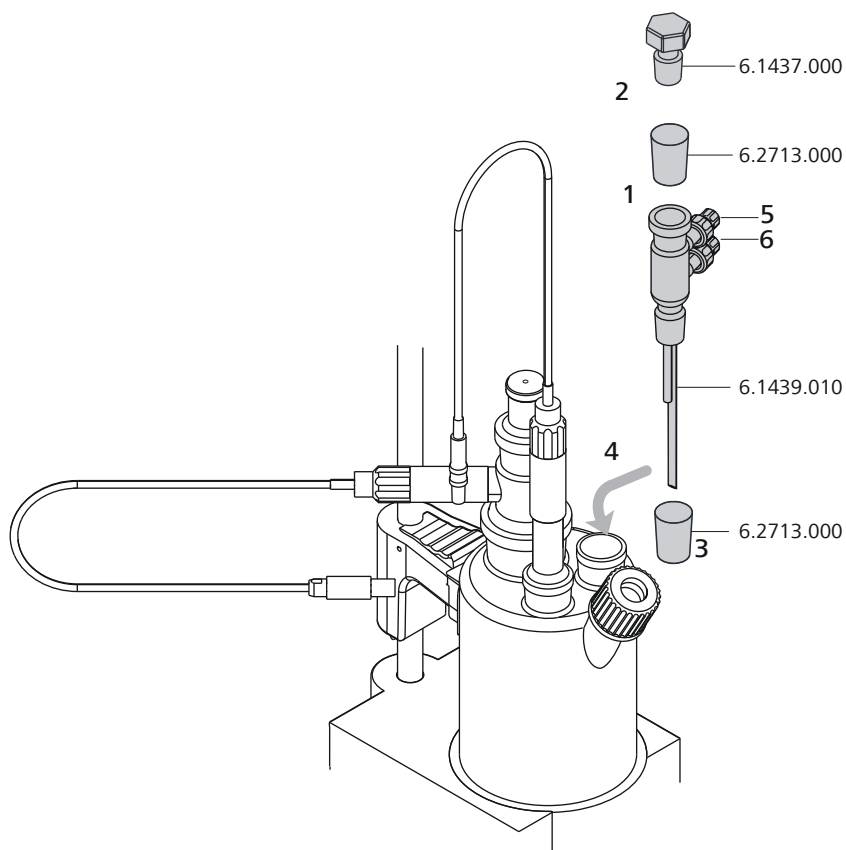


Figure 18 Mounting the addition and aspiration tube

- 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).

#### 4.5.4 Titration cell (coulometry) with equipment for reagent replacement – Utilization with Dosino

A Dosino allows the automatic replacement of reagents.

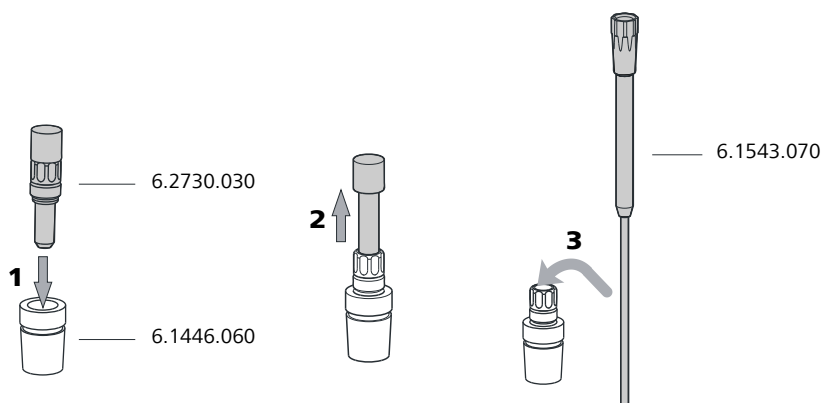
##### Required accessories:

- Equipment for reagent replacement (6.5617.000)
- 807 Dosing Unit

Metrohm recommends the following cylinder volumes for the 807 Dosing Unit:

Use case	Cylinder volume
General	50 mL
Aspirating greasy samples when only the sample and not the whole reagent is aspirated	20 mL
High-viscosity samples	10 mL

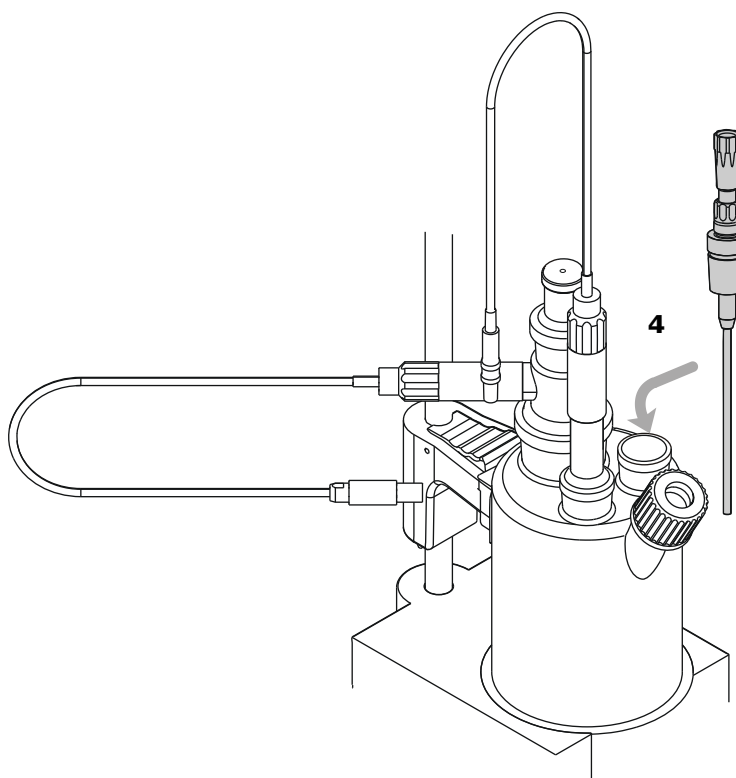
##### Mounting the aspiration tip



- 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 titration cell until it touches the vessel base.

#### 4.5.5 Titration cell (coulometry) with Karl Fischer oven

If samples release their water only slowly or only at higher temperatures, then 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 (coulometry) with a carrier gas. A detailed description can be found in the respective manual.

#### 4.5.6 Titration cell (coulometry) with sample changer

If a large number of samples need to be processed, then 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 can be found in the respective manual.

## 4.6 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 (Pt1000 or NTC)

### 4.6.1 Connecting a generator electrode

- 1 Plug the electrode plug into the **Gen.** socket of the 851 Titrand.

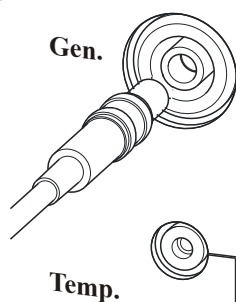


Figure 19 Connect generator electrode



#### NOTE

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.

### 4.6.2 Connecting an indicator electrode

- 1 Plug the electrode plug into the **Ind.** socket of the 851 Titrand.



## 5 Coulometric titration

### 5.1 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:



According to the equation above the  $\text{I}_2$  reacts quantitatively with  $\text{H}_2\text{O}$ . 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.

## 5.2 Working with water standards

### 5.2.1 Certified water standards

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



#### NOTE

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

### 5.2.2 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.



#### NOTE

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 851 Titrande 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** Read off the value displayed by the balance and enter it as sample size on the Touch Control or in the computer software (e.g. *tiamo*).  
If you have connected a balance to the Titrande, 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.

## 5.3 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

### 5.3.1 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<sub>2</sub>O. 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

### 5.3.2 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.

### 5.3.3 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.



### 5.4.3 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.

### 5.4.4 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. This phenomenon will, however, disappear after a short time of use. In order to accelerate the setting of a new indicator electrode, the 851 Titrand 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.



## 6.2 Generator electrode

### 6.2.1 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.

#### 6.2.1.1 Reagents

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

#### 6.2.1.2 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!).

### 6.2.2 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.

#### 6.2.2.1 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).



### 6.2.2.2 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  $\text{HNO}_3$  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  $70^\circ\text{C}$  (plastic parts!).

## 7 Troubleshooting

### 7.1 General

Problem	Cause	Remedy
The "On" LED is not illuminated, even though the instrument is connected to the power supply.	<i>The Touch Control or the computer has not been switched on yet or the plugs are not correctly plugged in.</i>	<ol style="list-style-type: none"> <li>1. Check the plug connections.</li> <li>2. Switch on the Touch Control or the computer.</li> </ol>

### 7.2 Karl Fischer titration

Problem	Cause	Remedy
The drift is very high during conditioning.	<i>The titration cell is leaking.</i>	<ul style="list-style-type: none"> <li>▪ Check the seals and the septum. Replace if necessary.</li> <li>▪ Replace the molecular sieve.</li> </ul>
The drift becomes greater after each titration.	<i>The sample releases water very slowly.</i>	<ul style="list-style-type: none"> <li>▪ Adjust the method.</li> <li>▪ Add solubility promoter.</li> <li>▪ Increase the temperature (possibly using a KF oven).</li> <li>▪ See technical literature.</li> </ul>
	<i>A side reaction is taking place.</i>	<ul style="list-style-type: none"> <li>▪ Use special reagents.</li> <li>▪ Adjust the method (increase/decrease the temperature, external extraction).</li> <li>▪ See technical literature.</li> </ul>
	<i>The pH value is no longer in the optimum range.</i>	Add buffer (see technical literature).
The titration will not be finished.	<i>The titration cell is leaking.</i>	<ul style="list-style-type: none"> <li>▪ Check the seals and the septum. Replace if necessary.</li> <li>▪ Replace the molecular sieve.</li> </ul>
	<i>The minimum increment is too low.</i>	Select the user-defined titration rate and increase the minimum volume increment (see manual/help of the software used).
	<i>The stop criterion is unsuitable.</i>	Adjust the control parameters (see manual/help of the software used): <ul style="list-style-type: none"> <li>▪ Increase the stop drift.</li> </ul>



## 8 Appendix

### 8.1 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.

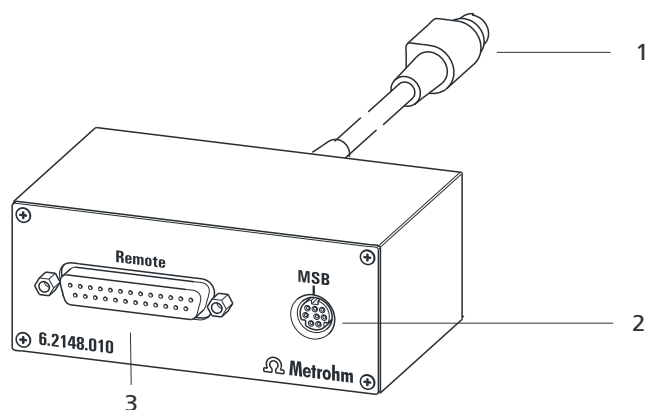


Figure 22 Connectors of the Remote Box

#### 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.

#### 8.1.1 Pin assignment of the remote interface

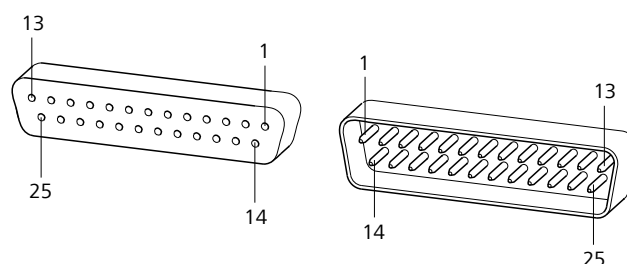
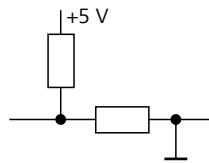


Figure 23 Pin assignment of remote socket and remote plug

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

### Inputs

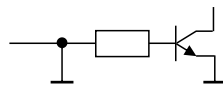


approx. 50 k $\Omega$  Pull-up

$t_p > 20$  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 3 Inputs and outputs of the remote interface

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

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.

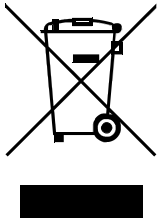
Table 4 Explanation of the individual functions

Function	Explanation
<b>Start</b>	The current method is started at the time of activation. $t_{\text{pulse}} > 100 \text{ ms}$
<b>Stop</b>	The current method is canceled (Stop) at the time of activation. $t_{\text{pulse}} > 100 \text{ ms}$
<b>Quit</b>	The current command in the determination run will be canceled at the time of activation. $t_{\text{pulse}} > 100 \text{ ms}$
<b>Ready</b>	The instrument is ready to receive a start signal.
<b>Conditioning OK</b>	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 <b>[START]</b> .
<b>Determination</b>	The instrument performs a data-generating determination.
<b>EOD</b>	<b>End of Determination.</b> Pulse ( $t_{\text{pulse}} = 200 \text{ ms}$ ) after a determination or after a buffer/standard solution during calibration using a Sample Processor.
<b>Error</b>	The line is set for error message display.



Function	Explanation
<b>Warning</b>	The line is set for warning message display.

## 9 Recycling and disposal



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

## 10 Technical specifications

### 10.1 Measuring interface

The 851 Titrande has one galvanically isolated measuring interface.

The measuring cycle is 100 ms for all measuring modes.

#### 10.1.1 Generator electrode

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

*I<sub>max</sub>*

400 mA

Continuous current and pulsed

#### 10.1.2 Indicator electrode

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

*Measuring mode*

Determination with adjustable polarization current

*I<sub>pol</sub>*

AC

5, 10, 20, and 30  $\mu$ A

DC

-125 to +125  $\mu$ A

#### 10.1.3 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  $\Omega$  and B (25/50) = 4,100 K)

*Resolution*

*Pt1000*

0.1 °C

*NTC*

0.1 °C

*Measuring accuracy*

*Pt1000*

$\pm$ 0.2 °C

(Applies for measuring range -20 to +150 °C;  $\pm$ 1 digit; without sensor error, under reference conditions)

*NTC*

$\pm$ 0.6 °C

(Applies for measuring range +10 to +40 °C;  $\pm 1$  digit; without sensor error, under reference conditions)

## 10.2 Power connection

<i>Supply voltage</i>	100–240 V ( $\pm 10\%$ )
<i>Frequency</i>	50–60 Hz ( $\pm 3\%$ )
<i>Power consumption</i>	max. 45 W
<i>Fuse</i>	Electronic overload protection

## 10.3 Ambient conditions

<i>Automatic interior temperature monitoring</i>	
<i>Nominal function range</i>	+5 to +45 °C at max. 80% relative humidity, non-condensing
<i>Storage</i>	+5 to +45 °C at max. 80% relative humidity, non-condensing
<i>Altitude / Pressure range</i>	max. 2,000 m. above sea level / min. 780 mbar
<i>Overvoltage category</i>	II
<i>Pollution degree</i>	2

## 10.4 Dimensions

<i>Width</i>	142 mm
<i>Height</i>	227 mm
<i>Depth</i>	231 mm
<i>Weight</i>	2.9 kg (without accessories)
<i>Material (housing)</i>	Polybutylene terephthalate (PBT)



## 10.5 Interfaces

### USB connectors

*USB ports* 2 USB downstream ports (type A sockets), 500 mA, for connecting peripheral devices such as printers, keyboards, barcode readers or RS-232/USB boxes (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 851 Titrande.

*Touch Control* With integrated Touch Control cable.

*Computer* With 6.2151.000 cable

### MSB connectors (Metrohm Serial Bus)

*Dosing device* Connection of a max. of 4 external dosing devices of the Dosimat or Dosino (MSB 1 to MSB 4) types.

*Stirrer* Connection of a max. 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 max. of 4 Remote Boxes. Remote Boxes can be used to actuate and monitor external devices.

# Index

6.2151.000 controller cable .....	14
685 Dosimat plus .....	17
700 Dosino .....	17
800 Dosino .....	17
801 Stirrer .....	18
803 Ti Stand .....	18
804 Ti Stand .....	18
805 Dosimat .....	17

## A

ADD .....	3
Addition and aspiration tube	
Mount .....	30
Adsorber tube	
Fill .....	26
Ambient conditions .....	53
Application bulletin .....	38

## B

Balance .....	22
Barcode reader	
Connect .....	24
BRC .....	3

## C

Catholyte	
Replace .....	41
Computer	
Connect .....	14
Computer keyboard	
Connect .....	23
Connect	
Balance .....	22
Barcode reader .....	24
Computer .....	14
Computer keyboard .....	23
Dosing device .....	17
MSB devices .....	16
Power grid .....	13
Printer .....	21
Remote Box .....	19
Stirrer .....	18
Titration stand .....	18
Touch Control .....	12
USB hub .....	21
Connector	
MSB .....	2
USB .....	2
Controller	
Connector .....	10

Coulometry	
Drift .....	40
Principle .....	35
Sample size .....	38
Tips for daily practice .....	36
Water standard .....	36
Working conditions .....	40

## D

Device description .....	2
Device software	
Update .....	2
Dosing command .....	2
ADD .....	3
EMPTY .....	3
LQH .....	3
PREP .....	3
Dosing device	
Connect .....	17
Driver software	
Install .....	14

## E

Electrode	
Connect .....	33
Electrostatic charge .....	8
EMPTY .....	3

## G

Generator electrode	
Fill .....	28

## H

Humidity .....	53
----------------	----

## I

Indicator electrode	
Warm-up time .....	41
Installation	
Driver software .....	14
Instrument type .....	10

## K

Karl Fischer	
Coulometry .....	35
Karl Fischer oven	
Use .....	32
Keyboard	
Connect .....	23
KFC .....	2

## L

LED	
On .....	45
LQH .....	3

## M

Maintenance .....	42
MEAS .....	3
Measuring interface .....	2, 10
Measuring mode .....	2
MEAS .....	3
Metrohm Serial Bus MSB, see also "MSB" .....	16
Molecular sieve	
Replace .....	26
MSB	
Connect devices .....	16
Connector .....	10
MSB connector .....	2

## O

Operation .....	53
Oven module .....	32
Overview of the instrument .....	9
Overvoltage category .....	53

## P

Pin assignment .....	47
Power connection .....	10, 13
PREP .....	3
Preparation time	
Generator electrode with dia- phragm .....	40
Generator electrode without diaphragm .....	40
Printer .....	21

## R

Reagent replacement .....	41
Remote	
Interface .....	47
Pin assignment .....	47
Remote Box	
Connect .....	19

## S

Safety instructions .....	6
Sample	
Liquid .....	38
Solid .....	39

## Index

Sample changer	
With oven module .....	32
Sample size	
Size .....	38
Sea level .....	53
Sensor	
Connect .....	33
Serial number .....	10
Service .....	7
Stirrer	
Connect .....	18
Storage .....	53
Supply voltage .....	8
System test .....	12

## T

---

Temperature .....	53
-------------------	----

Temperature sensor	
Connect .....	33
Titrando system .....	1
Titration cell	
Equip .....	27
Fill .....	28
Titration cell (coulometry)	
Equip .....	27
Fill .....	28
Titration mode .....	2
BRC .....	3
KFC .....	2
Titration stand	
Connect .....	18
Touch Control	
Connect .....	12

## U

---

Update	
Device software .....	2
USB	
Connector .....	10
USB connector .....	2
USB hub	
Connect .....	21

## W

---

Water standard	
Certified .....	36