

663 VA Stand



Manual

8.663.1003 / 2021-12-08



Metrohm AG

CH-9100 Herisau

Switzerland

Phone +41 71 353 85 85

Fax +41 71 353 89 01

info@metrohm.com

www.metrohm.com

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

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1 Introduction

1.1 Instrument description

The 663 VA Stand is a voltammetric measuring stand for Metrohm Autolab potentiostats.

The 663 VA Stand can be used to perform polarographic and voltammetric measurements on the following electrodes:

- Multi-Mode Electrode pro (MME)
- Rotating disk electrode
- scTRACE Gold

The scope of delivery of the 663 VA Stand comprises all electrodes that are required for a complete measurement system, that is the Multi-Mode Electrode pro (MME), Ag/AgCl reference electrode and glassy carbon auxiliary electrode.

Alternative electrodes, cables and the power supply unit have to be ordered separately.

An interface for Metrohm 663 VA Stand (IME663) is required to connect the 663 VA Stand to the Metrohm Autolab potentiostats.

1.2 About the documentation



CAUTION

Please read through this documentation carefully before putting the instrument into operation. The documentation contains information and warnings which the user must follow in order to ensure safe operation of the instrument.







1.2.1 Further information and literature

You can find further information regarding the 663 VA Stand in the following publications:

- "Electrodes in Voltammetry" multimedia guide (A.717.0002)
- Multi-Mode Electrode pro (8.110.8018XX)
- Mercury Handling Guidelines (8.000.5054XX)

1.2.2 Symbols and conventions

The following symbols and formatting may appear in this documentation:

<i>(5-12)</i>	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 Perform the steps one after the other.
Method	Dialog text, parameter in the software
File ► New	Menu or menu item
[Continue]	Button or key
	WARNING This symbol draws attention to a possible life-threatening hazard or risk of injury.
	WARNING This symbol draws attention to a possible hazard due to electrical current.
	WARNING This symbol draws attention to a possible hazard due to heat or hot instrument parts.
	WARNING This symbol draws attention to a possible biological hazard.
	CAUTION This symbol draws attention to possible damage to instruments or instrument parts.
	NOTE This symbol highlights additional information and tips.

1.3 Safety instructions

1.3.1 General notes on safety



WARNING

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

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

1.3.2 Electrical safety

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



WARNING

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



WARNING

Never open the housing of the instrument. The instrument could be damaged by this. There is also a risk of serious injury if live components are touched.

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

Protection against electrostatic charges



WARNING

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

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

1.3.3 Personnel safety



WARNING

Handling hazardous substances

Hazardous substances may result in injuries.

Wear protective glasses and work clothes suitable for laboratory work.



WARNING

Uncontrolled splashing of reagents

Splashing reagents may result in injuries.

Operate the 663 VA Stand only with the measuring head arm folded down.

1.3.4 Metallic liquid mercury



WARNING

Mercury is a heavy liquid metal. Highly toxic mercury vapor forms even at room temperature. Breathing in mercury vapor poses the risk of chronic mercury poisoning. Therefore, observe the following points when handling a MME pro containing mercury:

- Do not inhale mercury vapor.
- Only work with open containers of mercury while under a fume cupboard.
- Avoid skin contact with mercury.
- Never store mercury in open containers.
- Keep the laboratory area for work involving mercury well-ventilated.
- Use amalgamation to bind any spilled mercury. Never use a broom or vacuum.
- Only have mercury disposed of by a professional. Never dispose of mercury in household or domestic waste.



NOTICE

For detailed information, observe the *Mercury Handling Guidelines* (8.000.5054XX).

1.3.5 Tubing and capillary connections

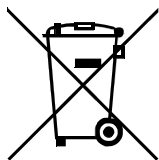


CAUTION

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

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

1.3.6 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.

The MME pro (Multi-Mode Electrode pro) contains toxic mercury; never dispose of it in domestic waste. For more information on the recycling and disposing of mercury, observe the *Mercury Handling Guidelines* (8.000.5054XX).

2 Overview of the instrument

2.1 Front

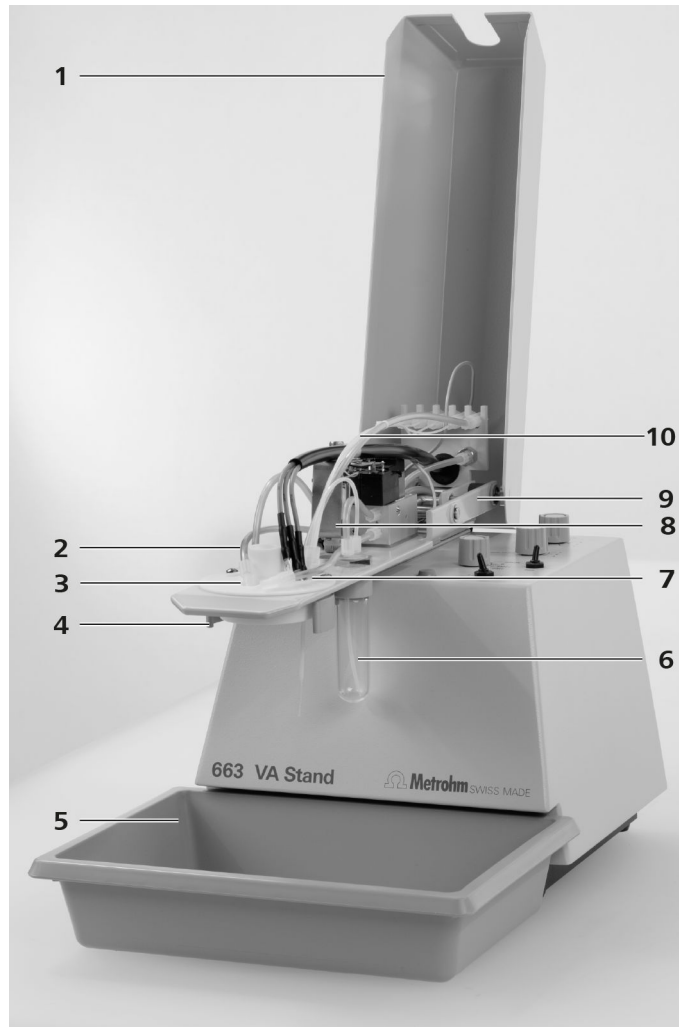


Figure 1 Front

1 Covering

Can be opened.

3 Top of measuring vessel

5 Drip pan (6.2711.030)

2 FEP tubing

Inert gas inlet to the MME - Multi-Mode Electrode pro.

4 Holder for measuring vessel

6 Gas washing bottle

7 FEP tubing
Inert gas inlet to the MME - Multi-Mode Electrode pro.

8 Motor drive
For stirrer.

9 Measuring head arm
Tilttable.

10 Tubing connections

2.2 Rear

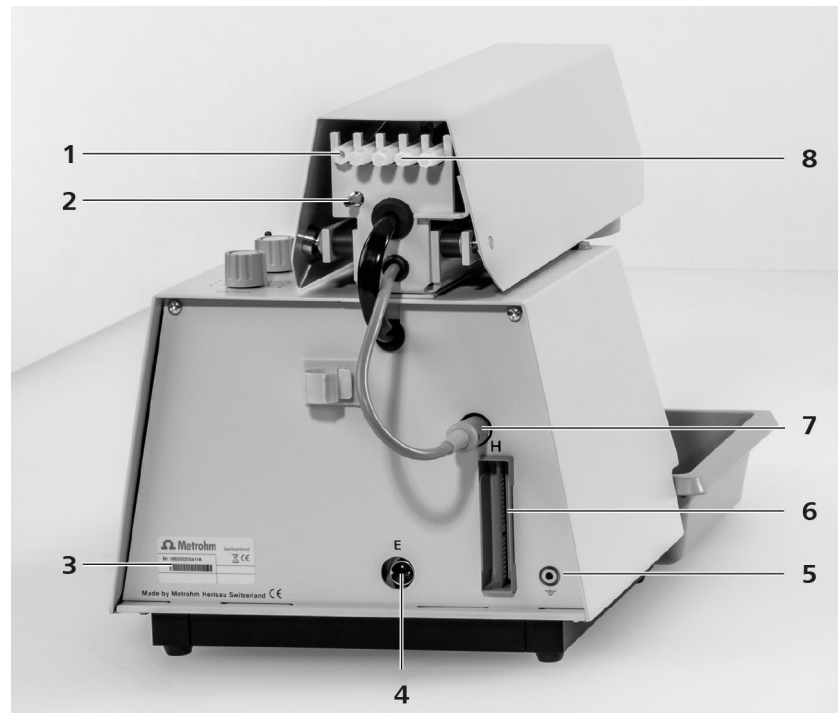


Figure 2 Rear

1 Connector
For inert gas outlet.

2 Connector
For inert gas inlet.

3 Type plate

4 Connection socket
For stirrer control cable.

5 Grounding socket

6 Connection socket
For instrument control cable.

7 Valve control

8 Connectors
For a maximum of four burets. Sealed with stoppers.



2.3 Controls

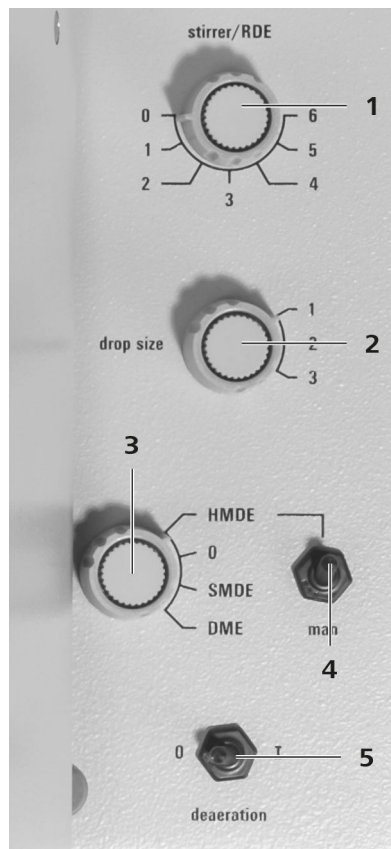


Figure 3 Controls

1 Control dial

For setting the stirring rate.

2 Control dial

For setting the drop size. For static mercury drop electrode (SMDE) and hanging mercury drop electrode (HMDE).

3 Control dial

For selecting the electrode type.

4 Switch

For knocking off the mercury drop of a hanging mercury drop electrode (HMDE).

5 Switch

To deaerate.

2.4 Top of measuring vessel

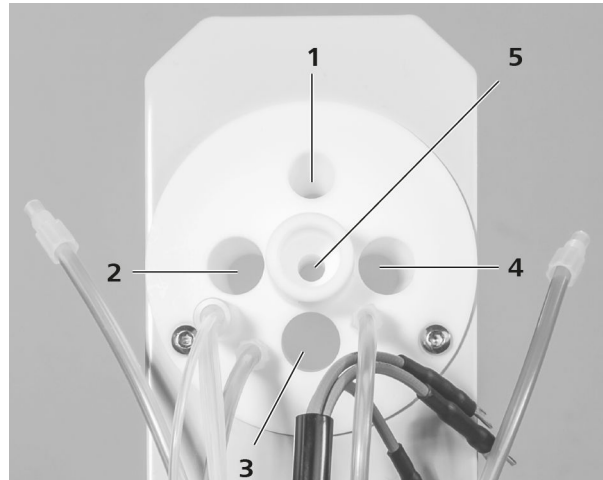


Figure 4 Top of measuring vessel

1 Pipetting opening

Seal with stopper (6.2709.030).

3 Opening

For stirrer (6.1204.200).

5 Opening

For MME - Multi-Mode Electrode pro (6.1246.120).

2 Opening

For reference electrode (6.0728.120 with 6.1245.010).

4 Opening

For auxiliary electrode (6.1241.120 with 6.1247.000).



2.5 Construction of the MME pro

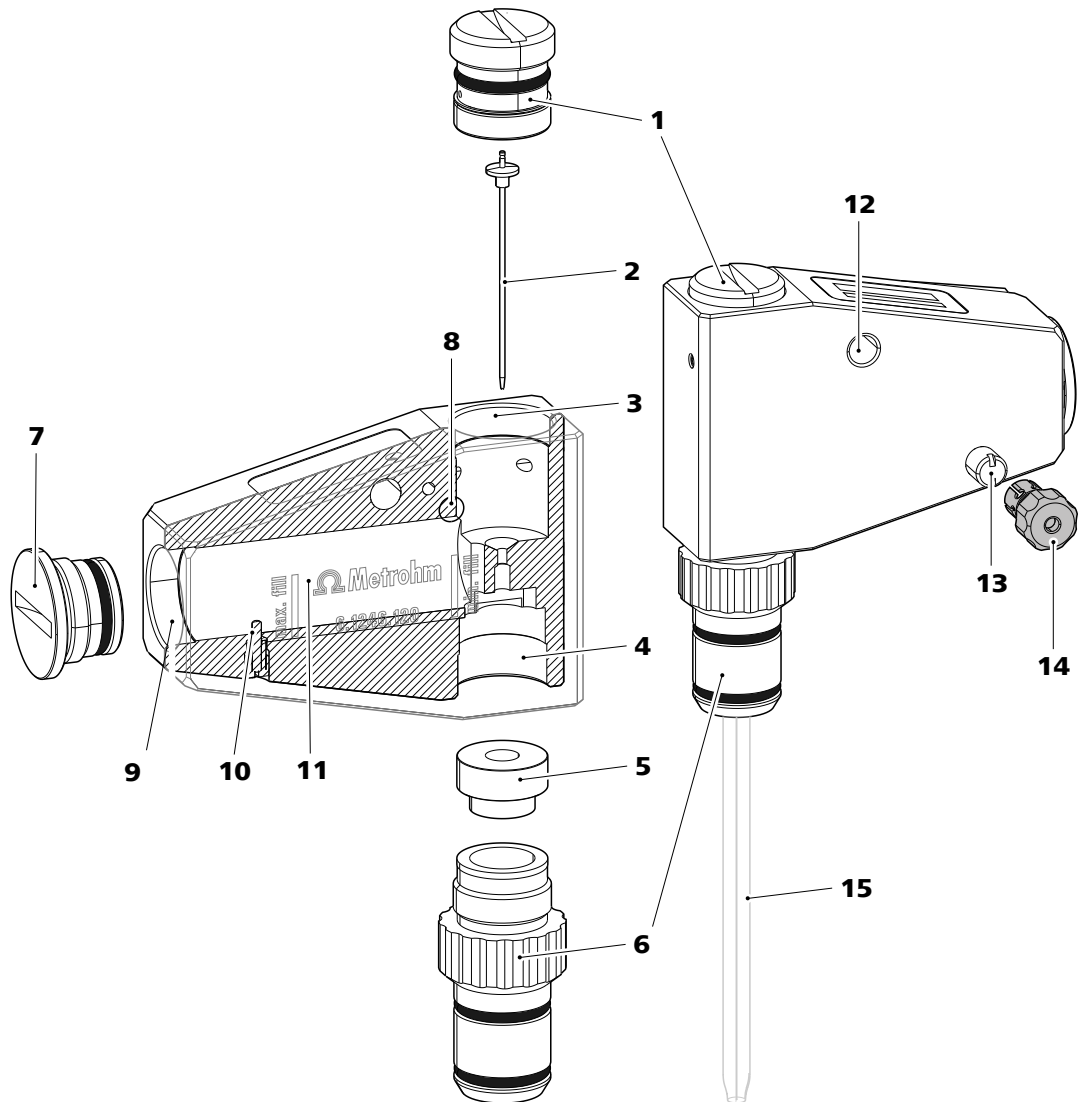


Figure 5 MME pro – Construction

1	Needle holder (6.1247.140)	2	Needle (6.1247.020)
3	Thread For the needle holder (5- 1).	4	Thread For the retaining screw (5- 6).
5	Silicone seal (6.1247.150)	6	Retaining screw (6.1247.120) For the capillary.
7	Screw cap (6.1247.110) For the mercury filler opening.	8	Connector For inert gas supply to control the needle valve.

9 **Thread**
For the screw cap (5-7).

11 **Mercury reservoir**

13 **Quick connector**
For WE cable.

15 **Glass capillary**
6.1226.030 or 6.1226.050

10 **Contact pin**
Electrical contact with mercury.

12 **Connector**
For inert gas supply.

14 **Adapter (6.2112.130)**
The adapter is only needed to connect the working electrode cable with terminal to the MME pro.



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 Inserting the stirrer

Installing the stirrer

Accessories

- Stirrer (6.1204.200)
- Drive belt (6.1241.020)



Figure 6 Installed stirrer

1 Driving wheel
On the motor drive (1-8).

2 Drive belt

3 Stirrer

1 Inserting the stirrer

Insert the stirrer in the measuring head opening (4-3) at the rear and push it back until it stops.

2 Connecting the stirrer

Strain the drive belt of the stirrer between the driving wheel (6-1) and the stirrer.

3.3 Inserting the Multi-Mode Electrode pro

Preparing and inserting the Multi-Mode Electrode pro

Accessories

- Multi-Mode Electrode pro (6.1246.120)

1 Preparing the Multi-Mode Electrode pro

Prepare the working electrode in accordance with the instructions in the *Multi-Mode Electrode pro* document (8.110.8018XX). In preparation, carry out the following tasks:

- Mount the capillary
- Insert the needle
- Top up the mercury



2 Inserting the Multi-Mode Electrode pro



CAUTION

When inserting the Multi-Mode Electrode pro, the tip of the capillary must not touch the measuring head.

- Raise the measuring head arm (1-9).
- Place an empty measuring vessel in the holder (1-4).
- Lower the measuring head arm.
- Carefully place the Multi-Mode Electrode pro in the center opening (4-5) of the measuring head insert and insert it as far as it will go.



3 Connecting the inert gas inlet

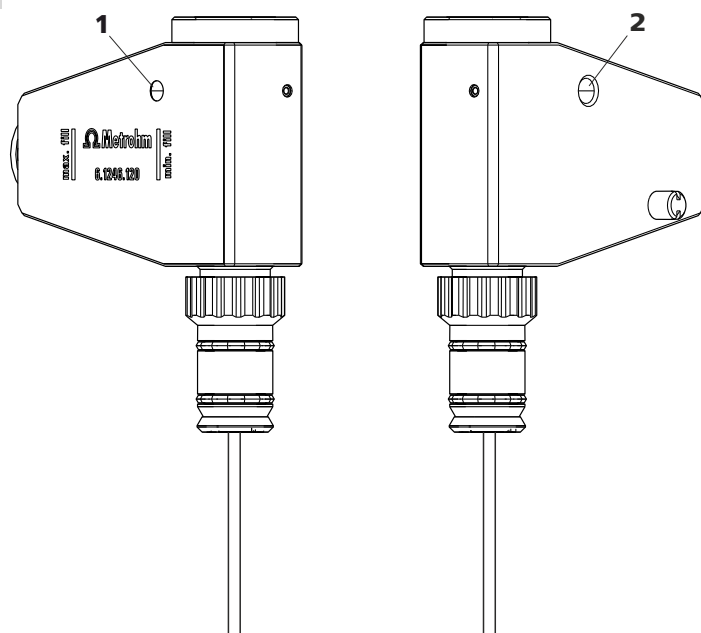


Figure 7 Connecting the inert gas inlet

1 Inert gas connection, left

2 Inert gas connector, right

- Screw the FEP tubing for the inert gas inlet (N₂) (1-2) into the connector (7-1) of the MME pro.
- Screw the FEP tubing for the inert gas inlet (N₂) (1-7) into the connector (7-2) of the MME pro.

4 Adjusting the needle valve

Adjust the needle valve in accordance with the instructions in the *Multi-Mode Electrode pro* document (8.110.8018XX) and the *Electrodes in Voltammetry* multimedia guide (A.717.0002).

5 Testing the electrode function

Test the electrode function in accordance with the instructions in the *Multi-Mode Electrode pro* document (8.110.8018XX).

3.4 Reference electrode (RE)

The reference electrode consists of the following two articles:

- Reference electrode filled with reference electrolyte (e.g. 6.0728.120)
- Electrolyte vessel filled with bridge electrolyte (e.g. 6.1245.010)

Preparing and inserting the reference electrode

1

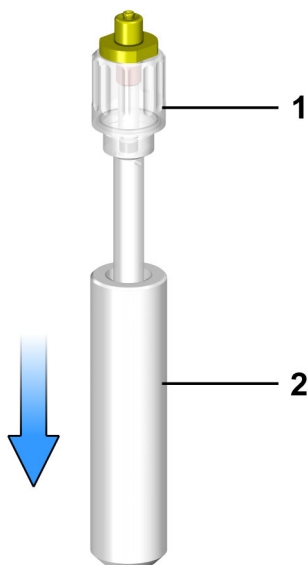


Figure 8 Preparing the reference electrode 1

1 Reference electrode

2 Storage vessel

Remove the reference electrode (8-**1**) from the storage vessel (8-**2**).

The reference electrode that is part of the accessories is already filled with reference electrolyte ($c(\text{KCl}) = 3 \text{ mol/L}$).

- 2** Fill the electrolyte vessel with bridge electrolyte (e.g. $c(\text{KCl}) = 3 \text{ mol/L}$) in accordance with the information in the electrode leaflet.
- 3** Allow the bridge electrolyte to react in the electrolyte vessel until the diaphragm is soaked with bridge electrolyte.

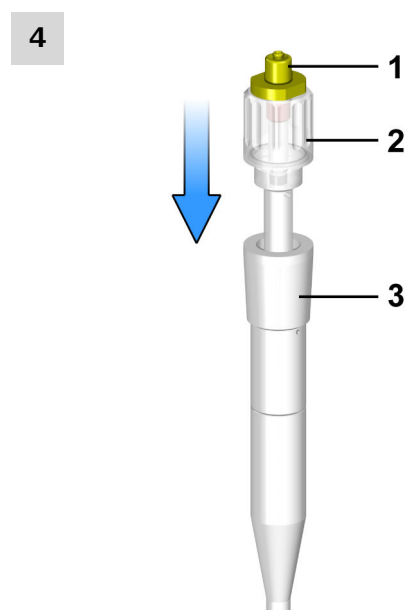


Figure 9 Preparing the reference electrode 2

1 Quick connector (for connection of RE)

2 Reference electrode

3 Electrolyte vessel

Place the reference electrode (9-**2**) in the filled electrolyte vessel (9-**3**) and screw it in place.

The electrolyte solution that is displaced in the electrolyte vessel is forced out of the deaeration openings.

5 Rinse the installed reference electrode with ultrapure water.

6 Insert the installed reference electrode into the opening (4-**2**) of the measuring head insert.



3.5 Auxiliary electrode (AE)

The following electrodes can be used as auxiliary electrodes (AE):

- **Electrode holder (6.1241.120) and glassy carbon rod (6.1247.000)**: Together they form the glassy carbon auxiliary electrode. Included in standard delivery.
- **Pt auxiliary electrode (6.0343.100)**

The provided GC auxiliary electrode has to be put together first. The optionally available Pt auxiliary electrode (6.0343.100) can be placed directly in the measuring head.

Assembling the GC auxiliary electrode

Accessories

- Electrode holder (6.1241.120)
- Glassy carbon rod (6.1247.000)

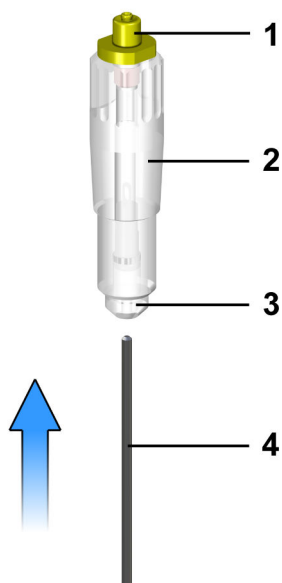


Figure 10 GC auxiliary electrode

1 Quick connector (for AE cable)

2 Electrode holder

3 Mounting ring

4 Glassy carbon rod

1



CAUTION

Glassy carbon is a brittle, very fragile material; proceed with caution when inserting it into the electrode holder.

Should the glassy carbon rod break, remove the remaining part from the holder by pulling the mounting ring (10-3) out.

Insert the glassy carbon rod (10-4) through the mounting ring (10-3) into the electrode holder (10-2) as far as it will go.

Inserting the auxiliary electrode

- 1 Insert the auxiliary electrode into the opening (4-4) of the measuring head insert.

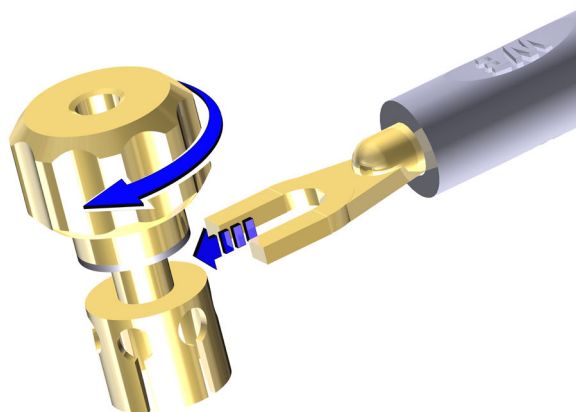
3.6 Connecting the electrodes

Connecting the electrodes

The electrode cable of the potentiostat has three banana plugs:

- red for the working electrode (WE)
- blue for the reference electrode (RE)
- black for the auxiliary electrode (AE)

Metrohm Autolab provides an adapter cable to connect the electrode cable to the respective electrode. The adapter cable has sockets for the banana plugs on one end and on the other end, it has terminals to connect to the electrode. Additionally, one 6.2112.130 adapter per electrode is necessary for electrodes with quick connector.



1 Connecting the working electrode

- Screw the 6.2112.130 adapter onto the terminal of the adapter cable for the working electrode (red).
- Plug the adapter onto the electrode connector (5-13).
- Plug the working electrode banana plug of the electrode cable into the working electrode socket of the adapter cable.

2 Connecting a reference electrode

- Screw the 6.2112.130 adapter onto the terminal of the adapter cable for the reference electrode (blue).
- Plug the adapter onto the electrode connector (9-1).
- Plug the reference electrode banana plug of the electrode cable into the reference electrode socket of the adapter cable.

3 Connecting the auxiliary electrode

- Screw the 6.2112.130 adapter onto the terminal of the adapter cable for the auxiliary electrode (black).
- Plug the adapter onto the electrode connector (10-1).
- Plug the auxiliary electrode banana plug of the electrode cable into the auxiliary electrode socket of the adapter cable.

3.7 Connecting the inert gas supply

Generally, nitrogen (N_2) is used as inert gas for deaerating the measuring solution and for operating the MME pro. Only nitrogen of sufficient purity may be used for this.

For general polarography/voltammetry:

- 4.5 (w(N_2) = 99.995%)

For analyses in organic solvents; for determinations that result in very high current strengths (such as for determining the smallest concentrations without preceding deposition).

- 5.0 (w(N_2) = 99.999%)

1 Filling the gas washing glass



Figure 11 Gas washing bottle

1 Gas washing bottle

- Unscrew the gas washing glass (11-1) from the measuring head arm.



- Fill the gas washing glass as follows:
 - Standard: Fill the gas washing glass halfway with distilled H₂O.
 - For long-term measurements with base electrolytes like acetic acid/acetate buffer or ammonia/ammonium chloride buffer, fill with base electrolyte.
 - For measurements in organic solvents, fill with the solvent being used.
- Screw the gas washing glass back onto the measuring head arm.

2 Connecting the inert gas inlet

- Connect one end of the PVC tubing (6.1801.080) onto the nipple (2-2) of the 663 VA Stand.
- Connect the other end of the PVC tubing (6.1801.080) onto the inert gas bottle connector.
- Set the inert gas pressure on the gas bottle to $p = 1.0\text{--}1.2$ bar (or 14.5–17.4 psi or 0.1–0.12 MPa) using the reducing valve.
- Open the gas inlet on the gas bottle.

3.8 Connecting the instruments

Connect all instruments that belong to the system according to the following diagram.

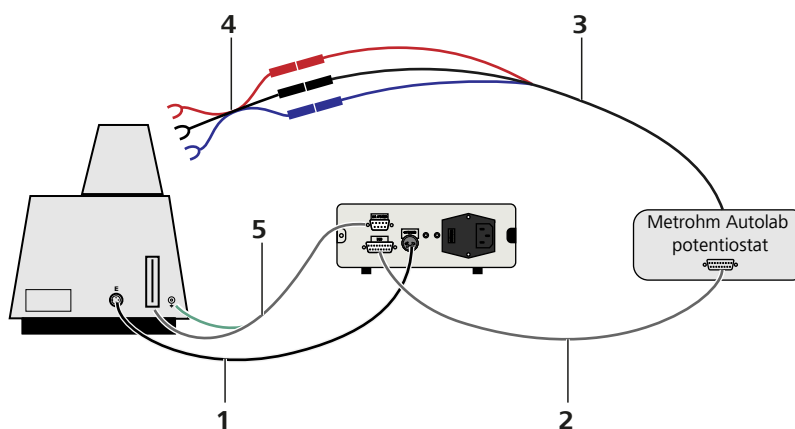


Figure 12 Cable connections

1 Control cable

For stirrer.

3 Electrode cable

5 Connecting cable

Between 663 VA Stand and IME663 interface.

2 Connecting cable

Between IME663 interface and Metrohm Autolab potentiostat.

4 Adapter cable

4 Start-up

Preparing the system for start-up



WARNING

Uncontrolled splashing of reagents

Splashing reagents may result in injuries.

Operate the 663 VA Stand only with the measuring head arm folded down.



WARNING

Careless folding of the measuring head arm

If the measuring head arm is folded down without due care, this may result in injuries to the hands.

Make sure that your fingers do not get caught between the measuring head arm and the instrument housing.

- 1 Insert the measuring vessel into the holder (1-4).
- 2 Fold the measuring head arm (1-9) down.
- 3 Position the drip pan (6.2711.030) (1-5) underneath the measuring vessel.
- 4 Switch on the potentiostat and the IME 663 interface.



5 Technical specifications

5.1 Ambient conditions

<i>Nominal function range</i>	+5 to +45 °C (at a maximum of 85% relative humidity)
<i>Storage and transport</i>	-20 to +70 °C

5.2 Energy supply

<i>Via control instrument</i>	5 V DC / 40 V DC
<i>Nominal voltage Stirrer</i>	11 V DC

5.3 Dimensions

<i>Measurements</i>	Width	290 mm
	Height	with covering closed 295 mm with covering opened 570 mm
	Depth	420 mm
	<i>Weight</i>	9.4 kg

5.4 Housing

<i>Materials</i>	Covering	Steel, coated
	Back panel	Steel
	Base	Steel, coated
	Housing	Steel, coated

5.5 Specifications stirrer / rotating disk electrode

Rotational speed settings 0, 500, 1,000, 1,500, 2,000, 2,500, 3,000 min⁻¹

6 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. **2.663.0020**) 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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