# 884 Professional VA



Manual 8.884.8003EN / 2021-01-05





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# 884 Professional VA

Manual

8.884.8003EN / 2021-01-05

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# **1** Introduction

# 1.1 Instrument description

The **884 Professional VA** instrument is a computer-controlled voltammetric measuring instrument. The measuring instrument consists of an 884 Professional VA and a measuring head. Together with the **viva** computer software, it forms a measurement system specially designed for VA trace analysis (with MME, RDE and SPE measuring head) and the CVS determination of additives in galvanic baths (with RDE measuring head). The instrument makes it possible to verify or quantitatively determine materials in low concentrations using voltammetry/polarography. In the electroplating industry, the determination of additives (in combination with an RDE measuring head) is an indispensable step in the production process and plays a significant role in quality assurance. The additives can be quantitatively determined by means of CVS (Cyclic Voltammetric Stripping).

The 884 Professional VA instrument is very compact in size and requires little space for operation. The measuring head can be removed from the instrument and put back in place again with a simple hand movement. This allows you to exchange the measuring head with all the electrodes and tubing quickly. This allows you to quickly and easily switch the instrument's equipment over for another application. The measuring head arm can be tilted upwards, thus enabling convenient placement of the measuring vessel in the holder.

The measuring instrument uses the potentiostatic 3-electrode principle. This principle means that the potential at the working electrode is gauged to the predefined setpoint value with the aid of a reference electrode to which no current is applied. The current flows through a separate auxiliary electrode. The Multi-Mode Electrode pro (MME pro), which combines a dropping mercury electrode (DME/SMDE) and a stationary hanging mercury drop electrode (HMDE) in a single design, is used as the working electrode. A rotating disk electrode (RDE) or a screen-printed electrode (SPE) can be used in place of the MME pro. Unlike the MME and RDE, the SPE combines the working electrode, the reference electrode and the auxiliary electrode in one electrode. Not 3 separate electrodes are used.

The following documentation describes the measuring head with the MME pro, the measuring head with the RDE and the measuring head with the SPE.

The 884 Professional VA instrument can be used both for single determinations and sample series. Various sample processors are suitable for this measuring instrument. Furthermore, you can automate the addition of reagents and auxiliary solutions by using dosing devices of the type 800 Dosino.

The 884 Professional VA instrument must be started, operated and controlled via the **viva** computer software. The data transfer between the measuring instrument and the computer takes place via a USB connection. At the end of every determination, the data (voltammogram, results, etc.) is saved in a database.

This manual describes the hardware of the 884 Professional VA instrument (installation, start-up, etc.). Operation with the **viva** computer software is described in the software documentation (online help and VA/CVS tutorials). The *Multi-Mode Electrode pro* document (8.110.8018XX) describes how to handle the Multi-Mode Electrode pro (MME pro). The *Leaflet* 6.1241.090 Electrode shaft for screen-printed electrodes (SPE) document (8.0109.8014EN) describes how to handle the screen-printed electrode (SPE).

# 1.2 Instrument versions

The **884 Professional VA** instrument is available in the following versions:

2.884.0010	Professional VA	without measuring head
2.884.0110	Professional VA manual for MME	with MME measuring head
2.884.0210	Professional VA manual for CVS	with CVS measuring head
2.884.1110	Professional VA semiautomated for MME	with MME measuring head and 2 Dosinos
2.884.1210	Professional VA semiautomated for CVS	with CVS measuring head and 2 Dosinos

# 

The accessories for a given model version can be created as a PDF list on the Internet (*see chapter 10, page 184*).

# 1.3 Intended use

The 884 Professional VA, in combination with the MME measuring head, the RDE measuring head or the SPE measuring head is designed for voltammetric/polarographic determination of samples in the area of trace analysis. The following are potential application areas:

- Determination of transition metals using polarography or stripping voltammetry
- Speciation analysis



### NOTICE

SPEs are not resistant to solvents. Do not use organic solvents for measurements or cleaning.

The 884 Professional VA, in combination with the RDE measuring head, has been designed for use in the analysis of electroplating baths for the quantitative determination of organic additives. The main fields of application are the following electroplating bath types:

- Acidic copper baths
- Tin baths
- Tin-lead baths
- Alkaline zinc baths
- Nickel baths

This instrument is suitable for processing various chemicals and flammable samples. Usage of the 884 Professional VA therefore requires the user to have basic knowledge and experience in handling toxic and caustic substances. Knowledge regarding the application of fire prevention measures prescribed for laboratories is also mandatory.

Before performing an analysis, the user should get acquainted with the hazard and precautionary statements or the risk and safety phrases that are valid for the chemicals used. The user should also observe the precautionary measures prescribed.

# **1.4** About the documentation



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 Figures

The instrument can be used for both VA trace analysis (with an MME, RDE or SPE measuring head) and additive determination using CVS (with an RDE measuring head). The installation of all 3 measuring heads on the instrument is described separately in this documentation. For the sake of simplicity, in all other figures only one version of the measuring head is shown. In those cases, the content displayed also applies the same to the other measuring heads.

# 1.4.2 Further information and literature

Further information regarding the 884 Professional VA is available in the following publications:

- CVS tutorial (8.103.8010XX)
- VA tutorial (8.103.8033XX)
- "Electrodes in Voltammetry" multimedia guide (A.717.0003)
- Online help for computer software viva
- "Voltammetric analysis methods in electroplating" monograph (8.108.5002XX)
- Multi-Mode Electrode pro (8.110.8018XX)
- Mercury Handling Guidelines (8.000.5054XX)

# 1.4.3 Symbols and conventions

The following symbols and formatting may appear in this documentation:

(5- <b>12</b> )	Cross-reference to figure legend		
	The first number refers to the figure number, the sec- ond 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		
$\mathbf{A}$	WARNING		
	This symbol draws attention to a possible life-threat- ening 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.		

# 1.4.4 References

3 different measuring heads are described in this manual. In the chapters 3.5 Establishing the tubing connections, 4 Start-up, 5.1 884 Professional VA for VA trace analysis and 6.11 Servicing the stirrer, only one measuring head typ is shown as an example. The content displayed also applies the same to the other measuring heads.

References to figures or descriptions lead to the chapter of the measuring head that is explicitly shown. If you are using a different measuring head, navigate to the corresponding chapter for the measuring head used via the table of contents. The measuring head chapters all have the same structure. If the chapter 2.3.1 "MME measuring head overview" is referred to, for example, then the respective information for the RDE measuring head under 2.5.1.

# 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 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.

# 1.5.3 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.

# 1.5.4 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 884 Professional VA only with the measuring head in place and the measuring head arm lowered.

# 1.5.5 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).

# **1.5.6 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.

# 1.5.7 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

The following figures provide a detailed overview of the parts of the 884 Professional VA.

# 2.1 Front



Figure 1 Front 884 Professional VA

# 1 "Status" LED

Continuously on: instrument ready for operation. Blinking regularly: instrument operating. Blinking pattern "LED on a long time off a short time - on a long time - off a short time, etc.": Standby potential is being applied to the electrodes. Do not remove the electrode cables!

# 2 "On" LED

Illuminated if the 884 Professional VA is connected to the power grid.

- **3** Holder for measuring vessel For inserting the measuring vessel.
- 5 Measuring head arm (tiltable)
- 7 Decanting glass (6.2405.030) For the deposition of solid materials that may be present (such as electrode mercury) in fully automated mode.

# 2.2 Rear

- 4 **Connector plate measuring head arm** For inserting the measuring head.
- 6 Gas washing glass (6.2405.030) For wetting the inert gas.
- 8 Holder for drip pan For positioning the drip pan.



Figure 2 884 Professional VA rear

- **1 Temperature sensor connector (Temp.)** For connecting a temperature sensor of the type Pt1000. 2 B sockets, 2 mm.
- 2 Type plates With serial number.

#### 2.2 Rear

3	Calibrator				
	For service procedures carried out by				
	Metrohm.				

5 Tubing connector (measuring head arm)

*See Chapter 2.3.3, page 17.* 

# 7 "Controller" connector For connecting to a PC with the **viva** computer software installed. Mini DIN, 8-pin.

9 Power socket

4 Fan Runs during operation.

6 USB connectors (USB 1 and USB 2), type A For connecting barcode readers, keyboards,

For connecting barcode readers, keyboards, etc.

# 8 MSB connectors (MSB 1 to 4) Metrohm Serial Bus. For connecting dosing devices (800 Dosino) and Remote Boxes. Mini DIN, 8-pin.

# 2.3 MME measuring head

# 2.3.1 MME measuring head overview



Figure 3 MME measuring head - Overview

Measuring head insert
With openings for inserting electrodes and
tubing connections (see figure 5, page 16).
 Measuring head connector plate
For connecting the MME measuring head to
the connector plate of the measuring head
arm (1-4).
For connecting the electrodes and tubing

For connecting the electrodes and tubing (see figure 4, page 14).

- **3** Measuring head cover For shielding against electromagnetic interference.
- 5 Stopper (6.2709.100) For closing the pipetting opening (5-22).

# 2.3.2 MME measuring head connector plate and measuring head insert

4

Slide lock

For measuring head cover.



*Figure 4 MME measuring head connector plate* 

1	<b>M6 threaded opening (3)</b> For adding solutions. Can be connected to one of the openings <b>24</b> - <b>27</b> (FEP tubing from 6.1829.070).	2	<b>M6 threaded opening (2)</b> For adding solutions. Can be connected to one of the openings <b>24</b> - <b>27</b> (FEP tubing from 6.1829.070).
3	<b>M6 threaded opening (N<sub>2</sub>)</b> For connecting the inert gas supply (N <sub>2</sub> ). Connected directly to the working electrode. The tubing comes pre-installed.	4	M6 threaded opening (WASTE) For aspirating the measuring solution. Can be connected to one of the openings <b>24</b> - <b>27</b> (FEP tubing from 6.1829.070).

- 5 UNF 10/32 threaded opening (SMP) For the automated sample addition. Must be connected to opening *28* (PEEK capillary 6.1831.020).
- 7 Electrode connector (AE, RE, WE) With electrode cables, for connecting electrodes.
- **9** Flexible drive shaft for the stirrer
- **11 M6 threaded opening (OUT)** With preinstalled tubing connection to opening *18* - gas outlet.
  - 14 30: see next figure

- 6 M6 threaded opening (1) For adding solutions. Can be connected to one of the openings **24** - **27** (FEP tubing from 6.1829.070).
- 8 Threaded opening (PURGE) For tubing connection to opening **19** - gas inlet in the measuring solution.
- **12 M6 threaded opening (TAP)** With preinstalled tubing connection to threaded opening **17** - gas inlet to the tapping mechanism.



*Figure 5 MME measuring head insert* 

	righte 5 Wilvie measuring neue insert						
14	<b>Opening</b> For feeding through a four-way micro dos- ing tip (6.1824.000) from below.		15	<b>Opening</b> For positioning the stirrer.			
16	<b>Threaded opening</b> With preinstalled screw nipple and stopper. Can be equipped with a four-way micro dos- ing tip (6.1824.000).		17	<b>M6 threaded opening</b> With preinstalled tubing connection to threaded opening <b>12</b> (TAP) - gas inlet to the tapping mechanism.			
18	<b>Opening</b> With preinstalled tubing connection to threaded opening <b>11</b> (OUT) - gas outlet.		19	<b>Opening</b> For tubing connection to threaded opening <b>8</b> (PURGE) - gas inlet in the measuring solu- tion.			

- **20 Opening for electrode** For inserting the reference electrode (RE).
- 22 Pipetting opening

For manually dosing solutions. Is closed with a 6.2709.100 stopper (3-**5**).

### 24 Opening

For adding or aspirating solutions. Can be connected with threaded opening **1**, **2**, **3** or **WASTE** (FEP tubing from 6.1829.070).

#### 26 Opening

For adding or aspirating solutions. Can be connected with threaded opening **1**, **2**, **3** or **WASTE** (FEP tubing from 6.1829.070).

### 28 Opening

For the automated sample addition. Must be connected to threaded opening **5** (SMP) (PEEK capillary 6.1831.020).

### 30 Opening

For feeding through the temperature sensor cable from above.

21 Opening for electrode

For inserting the Multi-Mode Electrode (working electrode - WE).

**23** Opening for electrode

For inserting the auxiliary electrode (AE).

### 25 Opening

For adding or aspirating solutions. Can be connected with threaded opening **1**, **2**, **3** or **WASTE** (FEP tubing from 6.1829.070).

### 27 Opening

For adding or aspirating solutions. Can be connected with threaded opening **1**, **2**, **3** or **WASTE** (FEP tubing from 6.1829.070).

### 29 Opening for sensor

For inserting a temperature sensor (Pt1000).

# 2.3.3 Tubing connector (measuring head arm)



*Figure 6 Tubing connector (measuring head arm)* 

## 1 Nipple (N<sub>2</sub>) For connecting the inert gas supply. Is connected to threaded openings TAP, PURGE

and  $\mathbf{N_2}$  via the measuring head arm.

**3 M6 threaded opening (2)** For connecting tubing for adding solutions. 2 M6 threaded opening (3) For connecting tubing for adding solutions.

4 UNF 10/32 threaded opening (SMP) For connecting a capillary for automated sample addition.

# 5 M6 threaded opening (1)

For connecting tubing for adding solutions.

### 7 M6 threaded opening (OUT) For pressure compensation in the measuring vessel.

6 M6 threaded opening (WASTE) For connecting tubing for aspirating the measuring solution.

# 2.4 RDE measuring head

# 2.4.1 RDE measuring head overview



Figure 7 RDE measuring head - Overview

Measuring head insert
 With openings for inserting electrodes and tubing connections (see figure 9, page 22).
 Measuring head connector plate
 For connecting the RDE measuring head to the connector plate of the measuring head arm (1-4).

For connecting the electrodes and tubing (see figure 8, page 20).

- **3** Measuring head cover For shielding against electromagnetic interference.
- 5 Stopper (6.2709.100) For closing the pipetting opening (9-22).

# 2.4.2 RDE measuring head connector plate and measuring head insert

4

Slide lock

For measuring head cover.



*Figure 8 RDE measuring head connector plate* 

- M6 threaded opening (3) For adding solutions. Can be connected to one of the openings 24 - 27 (FEP tubing from 6.1829.070).
- **3 M6 threaded opening (N<sub>2</sub>)** With preinstalled stopper. Not applicable to CVS analyses and RDE applications in VA trace analysis.
- 2 M6 threaded opening (2) For adding solutions. Can be connected to one of the openings 24 - 27 (FEP tubing from 6.1829.070).
- M6 threaded opening (WASTE)
   For aspirating the measuring solution. Can be connected to one of the openings 24 27 (FEP tubing from 6.1829.070).

- 5 UNF 10/32 threaded opening (SMP) For the automated sample addition. Must be connected to opening 28 (PEEK capillary 6.1831.020).
- 7 Electrode connector (AE, RE, WE) With electrode cables, for connecting electrodes.
- 9 Drive shaft for rotating disk electrode (RDE)

### **11** M6 threaded opening (OUT)

With preinstalled tubing connection to opening **18** - gas outlet. *For voltammetric applications, not applicable to CVS analyses.* 

### 13 Guide roller

Transfers the rotary movement of the motor to the driving axle of the working electrode.

6 M6 threaded opening (1) For adding solutions. Can be connected to one of the openings 24 - 27 (FEP tubing from 6.1829.070).

### 8 Threaded opening (PURGE)

For tubing connection to opening **19** - gas inlet in the measuring solution. *For voltammetric applications, not applicable to CVS analyses*.

## **10 M6 threaded opening (N<sub>2</sub>)** With preinstalled stopper. Not applicable to CVS analyses and RDE applications in VA trace analysis.

- **12 M6 threaded opening (TAP)** With preinstalled tubing connection to threaded opening **17** - gas inlet via the measuring solution for keeping out oxygen during the measurement. *For voltammetric applications, not applicable to CVS analyses.* 
  - 14 30: see next figure



*Figure 9 RDE measuring head insert* 

# 14 Opening

For feeding through a four-way micro dosing tip (6.1824.000) from below.

# **16** Threaded opening

With preinstalled screw nipple and stopper. Can be equipped with a four-way micro dosing tip (6.1824.000).

#### **15 Opening** For positioning the driving

For positioning the driving axle.

## **17** M6 threaded opening

With preinstalled tubing connection to threaded opening **12** (TAP) - gas inlet via the measuring solution for keeping out oxygen during the measurement. *For voltammetric applications, not applicable to CVS analyses*.

# 18 Opening

With preinstalled tubing connection to threaded opening **11** (OUT) - gas outlet. For voltammetric applications, not applicable to CVS analyses.

### 20 Opening for electrode

For inserting the reference electrode (RE).

### 22 Pipetting opening

For manually dosing solutions. Is closed with a 6.2709.100 stopper (7-**5**).

#### 24 Opening

For adding or aspirating solutions. Can be connected with threaded opening **1**, **2**, **3** or **WASTE** (FEP tubing from 6.1829.070).

#### 26 Opening

For adding or aspirating solutions. Can be connected with threaded opening **1**, **2**, **3** or **WASTE** (FEP tubing from 6.1829.070).

### 28 Opening

For the automated sample addition. Must be connected to threaded opening **5** (SMP) (PEEK capillary 6.1831.020).

#### 30 Opening

For feeding through the temperature sensor cable from above.

### 19 Opening

For tubing connection to threaded opening **8** (PURGE) - gas inlet in the measuring solution. *For voltammetric applications, not applicable to CVS analyses.* 

# **21** Opening for electrode

For inserting the driving axle for the RDE (working electrode - WE).

#### 23 Opening for electrode

For inserting the auxiliary electrode (AE).

#### 25 Opening

For adding or aspirating solutions. Can be connected with threaded opening **1**, **2**, **3** or **WASTE** (FEP tubing from 6.1829.070).

### 27 Opening

For adding or aspirating solutions. Can be connected with threaded opening **1**, **2**, **3** or **WASTE** (FEP tubing from 6.1829.070).

### 29 Opening for sensor

For inserting a temperature sensor (Pt1000).

# 2.4.3 **Tubing connector (measuring head arm)**



*Figure 10 Tubing connector (measuring head arm)* 

1	Nipple (N <sub>2</sub> ) Is connected to threaded openings TAP, PURGE and N <sub>2</sub> via the measuring head arm. For voltammetric applications, not applica- ble to CVS analyses.	2	<b>M6 threaded opening (3)</b> For connecting tubing for adding solutions.
3	<b>M6 threaded opening (2)</b> For connecting tubing for adding solutions.	4	<b>UNF 10/32 threaded opening (SMP)</b> For connecting a PEEK capillary for the auto- mated sample addition.
5	<b>M6 threaded opening (1)</b> For connecting tubing for adding solutions.	6	<b>M6 threaded opening (WASTE)</b> For connecting tubing for aspirating the measuring solution.
7	<b>M6 threaded opening (OUT)</b> For pressure compensation in the measuring vessel.		
# 2.5 SPE measuring head

#### 2.5.1 SPE measuring head overview



Figure 11 SPE measuring head - Overview

# Measuring head insert With openings for inserting electrodes and tubing connections (see figure 13, page 28). Measuring head connector plate For connecting the SPE measuring head to the connector plate of the measuring head arm (1-4).

For connecting the electrodes and tubing (see figure 12, page 26).

- **3** Measuring head cover For shielding against electromagnetic interference.
- 5 Stopper (6.2709.100) For closing the pipetting opening (13-20).

#### 2.5.2 SPE measuring head connector plate and measuring head insert

4

Slide lock

For measuring head cover.



Figure 12 SPE measuring head connector plate

- 1 M6 threaded opening (3) For adding solutions. Can be connected to one of the openings *21* - *24* (FEP tubing from 6.1829.070).
- **3 M6 threaded opening (N<sub>2</sub>)** With preinstalled stopper. Not applicable to SPE applications.
- 2 M6 threaded opening (2) For adding solutions. Can be connected to one of the openings **21** - **24** (FEP tubing from 6.1829.070).
- M6 threaded opening (WASTE)
   For aspirating the measuring solution. Can be connected to one of the openings 21 24 (FEP tubing from 6.1829.070).

- 5 UNF 10/32 threaded opening (SMP) For the automated sample addition. Must be connected to opening 25 (PEEK capillary 6.1831.020).
- 7 Electrode connector (AE, RE, WE) With electrode cables, for connecting electrodes.
- **9** Flexible drive shaft for the stirrer
- **11 M6 threaded opening (OUT)** With preinstalled tubing connection to opening **17** - gas outlet.

- 6 M6 threaded opening (1) For adding solutions. Can be connected to one of the openings **21** - **24** (FEP tubing from 6.1829.070).
- 8 Threaded opening (PURGE) For tubing connection to opening **18** - gas inlet in the measuring solution.
- **10 M6 threaded opening (N<sub>2</sub>)** With preinstalled stopper. *Not applicable to SPE applications*.
- **12 M6 threaded opening (TAP)** With preinstalled tubing connection to threaded opening **16** - gas inlet via the measuring solution for keeping out oxygen during the measurement. *For voltammetric applications, not applicable to CVS analyses.*

13 - 27: see next figure



*Figure 13* SPE measuring head insert

#### 13 Opening

For feeding through a four-way micro dosing tip (6.1824.000) from below.

#### **15** Threaded opening

With preinstalled screw nipple and stopper. Can be equipped with a four-way micro dosing tip (6.1824.000).

#### 17 Opening

With preinstalled tubing connection to threaded opening **11** (OUT) - gas outlet.

#### 14 Opening

For positioning the stirrer.

#### **16** M6 threaded opening

With preinstalled tubing connection to threaded opening **12** (TAP) - gas inlet via the measuring solution for keeping out oxygen during the measurement.

#### 18 Opening

For tubing connection to threaded opening **8** (PURGE) - gas inlet in the measuring solution.

#### **19** Opening for electrode

For inserting the 6.1241.090 SPE electrode shaft with the screen-printed electrode (SPE).

Optional: 6.2709.130 adapter for use of the scTRACE Gold (6.1258.000) with the electrode shaft for the scTRACE Gold (6.1241.080) in the SPE measuring head.

#### 21 Opening

For adding or aspirating solutions. Can be connected with threaded opening **1**, **2**, **3** or **WASTE** (FEP tubing from 6.1829.070).

#### 23 Opening

For adding or aspirating solutions. Can be connected with threaded opening **1**, **2**, **3** or **WASTE** (FEP tubing from 6.1829.070).

#### 25 Opening

For the automated sample addition. Must be connected to threaded opening **5** (SMP) (PEEK capillary 6.1831.020).

#### 27 Opening

For feeding through the temperature sensor cable from above.

#### 2.5.3 **Tubing connector (measuring head arm)**





#### 1 Nipple (N<sub>2</sub>)

For connecting the inert gas supply. Is connected to threaded openings **TAP**, **PURGE** and  $N_2$  via the measuring head arm.

## 2 M6 threaded opening (3)

For connecting tubing for adding solutions.

**20 Pipetting opening** For manually dosing solutions. Is closed with a 6.2709.100 stopper (*11-5*).

#### 22 Opening

For adding or aspirating solutions. Can be connected with threaded opening **1**, **2**, **3** or **WASTE** (FEP tubing from 6.1829.070).

#### 24 Opening

For adding or aspirating solutions. Can be connected with threaded opening **1**, **2**, **3** or **WASTE** (FEP tubing from 6.1829.070).

#### 26 Opening for sensor

For inserting a temperature sensor (Pt1000).

- **3 M6 threaded opening (2)** For connecting tubing for adding solutions.
- 5 M6 threaded opening (1) For connecting tubing for adding solutions.
- 7 M6 threaded opening (OUT) For pressure compensation in the measuring vessel.
- 4 UNF 10/32 threaded opening (SMP) For connecting a capillary for automated sample addition.
- 6 M6 threaded opening (WASTE) For connecting tubing for aspirating the measuring solution.

# **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. Do not place the instrument in the vicinity of an air-conditioning unit outlet.

# 3.2 Equipping the MME measuring head



#### CAUTION

The measuring head insert (*see figure 5, page 16*) is made of PTFE. Do not use sharp objects around it to ensure the material is not damaged.

#### 3.2.1 Preparing the MME measuring head



#### NOTICE

For equipping, Metrohm recommends placing the MME measuring head in the measuring head holder and only then inserting it onto the measuring head arm.

**1** Remove the stopper (3-5) from the pipetting opening to remove the measuring head cover.



*Figure 15 Removing the stopper from the pipetting opening* 

2 Pull the slide lock (3-4) on the top of the measuring head cover towards you and, at the same time, tilt the measuring head cover to an angle of approx. 45° and remove it.



*Figure 16 Removing the measuring head cover* 

## **3** Connecting the gas inlet

- Insert the PTFE tubing for adding gas to the solution (6.1829.030) through the opening (5-19).
- Pull the transparent inner tubing through as far as it will go.
- Ensure that the green kink protection is protecting the entire piece of tubing.



*Figure 17* Inserting the gas inlet

- Connect the tubing to the PURGE threaded opening (4-8) and tighten it hand-tight.
- Finally, tighten the tubing nipple using the wrench provided (6.2739.000).



*Figure 18 Connecting the gas inlet* 

#### 4 Inserting the stirrer

- Screw one end of the flexible shaft to the stirrer using the clamping screw. While fastening the flexible shaft, push it through as far as it will go.
- Tighten the flexible shaft. To do so, screw the clamping screw for the flexible shaft into the stirrer using 2 wrenches (6.2739.000). In the process, ensure that the flexible shaft is not kinked.



*Figure 19 Screwing in the flexible shaft* 

 Insert the stirrer into the opening (5-15) and press it down as far as it will go.

- Fasten the other end of the flexible shaft to the drive shaft (4-9) using the second clamping screw. While doing so, push the flexible shaft through far enough so that the course is as straight as possible.
- Screw the clamping screw for the flexible shaft to the drive shaft using 2 wrenches (6.2739.000). In the process, ensure that the flexible shaft is not kinked.



*Figure 20* Inserting and connecting the stirrer

#### **5** Inserting the stopper

• If the openings (5-24)-(5-29) are not needed (for manual operation), seal them with the stoppers provided (6.2709.110).



#### NOTICE

The stopper shown in light blue in the figure has a smaller diameter than the others. Keep this in mind when inserting the stopper.



*Figure 21 Inserting the stopper* 

# **3.2.2 Preparing electrodes and inserting them in the MME measuring head**

The 884 Professional VA uses the 3-electrode principle. The following electrodes are used:

- Multi-Mode Electrode pro (MME pro) as the working electrode (WE)
- Reference electrode (RE)
- Auxiliary electrode (AE)



#### NOTICE

Also observe the notes contained in the electrode leaflets that can be downloaded from the *Metrohm website* using the corresponding article number. In addition, you can learn how to best handle the electrodes from the multimedia guide (A.717.0003).

For detailed information about handling the Multi-Mode Electrode pro (MME pro), observe the *Multi-Mode Electrode pro* document (8.110.8018XX).

#### 3.2.2.1 Working electrode (WE)

#### Preparing and inserting the working electrode

Proceed as follows:

#### **1** Preparing the working electrode

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 working electrode into the measuring head insert

- Place an empty measuring vessel in the holder (1-3).
- Carefully insert the working electrode into the opening (5-21) of the measuring head insert. The bottom of the capillary must not touch the measuring head while being inserted.



*Figure 22* Inserting the working electrode

#### **3** Connecting the working electrode



#### CAUTION

The electrode cables for the reference, working and auxiliary electrode look identical. Observe the markings on the plugs, because the 3 cables must not be mixed up.

Plug the electrode cable (4-**7**) with the **WE** marking on the plug onto the metal contact of the working electrode.



*Figure 23 Connecting the working electrode* 





1 Inert gas connection, left	2 Inert gas connection, right
------------------------------	-------------------------------

- Screw in the FEP tubing to the inert gas inlet (N<sub>2</sub>, (4-3)) in the connection (24-1) for the MME pro.
- Screw in the FEP tubing to the inert gas inlet (N<sub>2</sub>, (4-10)) in the connection (24-2) for the MME pro.



#### NOTICE

Observe (*see chapter 3.2.4, page 50*) for information on connecting the inert gas supply.

#### 5 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.717.0003).

### **6** Testing the electrode function

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

#### 3.2.2.2 Reference electrode (RE)

The reference electrode consists of the following 2 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

Proceed as follows:

**1** Remove the reference electrode from the storage vessel.

The reference electrode that is part of the accessories is already filled with reference electrolyte (c(KCI) = 3 mol/L).

- **2** Fill the electrolyte vessel with bridge electrolyte (e.g. c(KCI) = 3 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.
- **4** Place the reference electrode in the filled electrolyte vessel and screw it in place.

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



trode look identical. Observe the markings on the plugs, because the 3 cables must not be mixed up.

Plug the electrode cable (4-**7**) with the **RE** marking on the plug onto the metal contact of the reference electrode.



*Figure 26 Connecting a reference electrode* 

#### 3.2.2.3 Auxiliary electrode (AE)

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

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

The Pt auxiliary electrode (6.0343.000) included in standard delivery can be inserted directly into the measuring head. The optionally available GC auxiliary electrode has to be put together first.



*Figure 27* Structure of the auxiliary electrodes

- **1** Auxiliary electrode
- **3** Pt auxiliary electrode (6.0343.100)
- 5 Electrode holder (6.1241.120)
- 2 Electrical connection for the "AE" cable
  4 Pt rod (permanently mounted)
  6 Retaining ring
- **7** Glassy carbon rod (6.1247.000)

#### Assembling the GC auxiliary electrode

Proceed as follows to assemble the optionally available GC auxiliary electrode:



#### CAUTION

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

In the event the GC rod breaks, the remaining part in the holder can be removed by pulling out the retaining ring (27-**6**).

Insert the glassy carbon rod (27-7) through the retaining ring (27-6) into the electrode holder (27-5) as far as it will go.

# Inserting the auxiliary electrode (Pt auxiliary electrode or GC auxiliary electrode)

Proceed as follows to insert the auxiliary electrode into the measuring head:

**1** Insert the auxiliary electrode into the opening (5-**23**) of the measuring head insert.



## CAUTION

The electrode cables for the reference, working and auxiliary electrode look identical. Observe the markings on the plugs, because the 3 cables must not be mixed up.

Plug the electrode cable (4-**7**) with the **AE** marking on the plug onto the metal contact of the auxiliary electrode.



*Figure 28 Connecting the auxiliary electrode* 

#### 3.2.3 Inserting the MME measuring head

Once the MME measuring head has been fully equipped, it can be inserted on the measuring head arm. Proceed as follows:

#### **1** Installing the measuring head cover

Place the measuring head cover at an angle of approx. 45° in the guide bolt in the front part of the measuring head insert.

Fold back the measuring head cover and gently push it in place.



*Figure 29* Installing the measuring head cover

The measuring head cover must snap into place with an audible click.

## 2 Inserting the stopper

Insert the stopper (3-5) into the pipetting opening.



*Figure 30* Inserting the stopper in the pipetting opening

#### **3** Inserting the measuring head



#### WARNING

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.



#### CAUTION

Do not apply pressure to the drive disk on the connector plate of the measuring head arm. Otherwise, the stirrer motor may be damaged.



*Figure 31* Do not touch the drive disk

Use one hand to hold the measuring head arm on the rear and use the other hand to insert the measuring head into the connector plate of the measuring head arm.

The measuring head must snap into place with an audible click.



Figure 32 Inserting the measuring head

#### **3.2.4 Connecting the inert gas supply**

Generally, nitrogen ( $N_2$ ) is used as inert gas in VA trace analysis for purging the measuring solution and for operating the MME pro, the SPE and the RDE. Only nitrogen of sufficient purity may be used for this.

For general polarography/voltammetry:

4.5 (w(N<sub>2</sub>) = 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<sub>2</sub>) = 99.999%)

#### 1 Filling the gas washing glass

- Unscrew the gas washing glass (1-6) from the measuring head arm.
- Fill the gas washing glass as follows:
  - Standard: Fill the gas washing glass halfway with distilled  $H_2O$ .
  - For long-term measurement with base electrolytes like acetic acid/acetate buffer solution or ammonia/ammonium chloride buffer solution, add the base electrolyte.
  - For measurements in organic solvents, fill with the solvent being used.
- Screw the gas washing glass back onto the measuring head arm.



*Figure 33 Gas washing glass* 

#### 2 Connecting the inert gas inlet

- Connect one end of the PVC tubing (6.1801.080) on the N<sub>2</sub> nipple of the 884 Professional VA.
- Connect the other end of the PVC tubing (6.1801.080) to the inert gas bottle connection.
- Set the inert gas pressure on the gas bottle to p = 1.0 to 1.2 bar (or 14.5 to 17.4 psi or 0.1 to 0.12 MPa) using the reducing valve.
- Open the gas inlet on the gas bottle.



*Figure 34 Nipple for inert gas supply* 

# **3.3 Equipping the RDE measuring head**

The RDE measuring head can be used for both VA trace analysis and CVS analysis. If the measuring head is used for the determination of organic additives with CVS, then chapter 3.3.1, step 3 and chapter 3.3.4 can be omitted.



#### CAUTION

The measuring head insert (7-**1**) is made of PTFE. Do not use sharp objects around it to ensure the material is not damaged.

#### 3.3.1 Preparing the RDE measuring head



## NOTICE

For equipping, Metrohm recommends placing the RDE measuring head in the measuring head holder and only then inserting it onto the measuring head arm.

**1** Remove the stopper (7-**5**) from the pipetting opening to remove the measuring head cover.



*Figure 35 Removing the stopper from the pipetting opening* 

2 Pull the slide lock (7-4) on the top of the measuring head cover towards you and, at the same time, tilt the measuring head cover to an angle of approx. 45° and remove it.



*Figure 36 Removing the measuring head cover* 

#### **3** Connecting the gas inlet



Only carry out this step if the RDE measuring head is used for VA trace analysis. If the RDE measuring head is used for CVS analysis, then no gas inlet is necessary.

- Insert the PTFE tubing for adding gas to the solution (6.1829.030) through the opening .
- Pull the transparent inner tubing through as far as it will go.
- Ensure that the green kink protection is protecting the entire piece of tubing.



*Figure 37* Inserting the gas inlet

- Connect the tubing to the PURGE threaded opening (4-8) and tighten it hand-tight.
- Finally, tighten the tubing nipple using the wrench provided (6.2739.000).



*Figure 38 Connecting the gas inlet* 

# **3.3.2 Preparing electrodes and inserting them in the RDE measuring head**

The 884 Professional VA uses the potentiostatic three-electrode principle. The following electrodes are used:

- Rotating disk electrode (RDE) as working electrode (WE)
- Reference electrode (RE)
- Auxiliary electrode (AE)



#### NOTICE

Also observe the notes contained in the electrode leaflets that can be downloaded from the *Metrohm website* using the corresponding article number. In addition, you can learn how to best handle the electrodes from the multimedia guide (A.717.0003).

#### 3.3.2.1 Working electrode (WE)

The working electrode consists of the following 2 articles:

- Electrode tip (e.g. 6.1204.610)
- Driving axle for rotating disk electrode (RDE) (e.g. 6.1204.510)

#### Preparing and inserting the working electrode

Proceed as follows:

#### **1** Preparing the electrode tip

Remove the protective cap from the electrode tip.



*Figure 39 Removing the protective cap from the electrode tip* 

#### 2 Assembling the working electrode

Hold the driving wheel by the driving axle and tighten the electrode tip to the driving axle.



*Figure 40 Tightening the electrode tip to the driving axle* 

# **3** Inserting the working electrode into the measuring head insert

Insert the working electrode into the opening (9-21) of the measuring head insert.

Make sure that the pin on the lower part of the driving axle is positioned in the opening (9-**15**) of the measuring head insert.



*Figure 41 Working electrode, installed* 

#### 4 Fastening the drive belt

- Slide the drive belt (6.1244.050) over the drive shaft (8-9),
- guide it on both sides over the guide roller (8-13) from below,
  - pull it over the working electrode and fasten it in the driving wheel of the driving axle.



*Figure 42 Fastening the drive belt* 



Make sure that the drive belt does not rub against the driving axle or surrounding components (tubing, cables, etc.).

#### **5** Connecting the working electrode



CAUTION

The electrode cables for the reference, working and auxiliary electrode look identical. Observe the markings on the plugs, because the 3 cables must not be mixed up.



Plug the electrode cable (8-7) with the **WE** marking on the plug onto the metal contact of the driving axle.

*Figure 43 Connecting the working electrode* 

#### 3.3.2.2 Reference electrode (RE)

The reference electrode consists of the following 2 articles:

- Reference electrode filled with reference electrolyte (e.g. 6.0728.130)
- Electrolyte vessel filled with bridge electrolyte (e.g. 6.1245.010)

#### Preparing and inserting the reference electrode

Proceed as follows:

**1** Remove the reference electrode from the storage vessel.

The reference electrode that is part of the accessories is already filled with reference electrolyte (c(KCI) = 3 mol/L).

- 2 Fill the electrolyte vessel with bridge electrolyte (e.g.  $c(KNO_3) = 1$  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.
- **4** Place the reference electrode in the filled electrolyte vessel and screw it in place.

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



6 Insert the installed reference electrode into the opening (9-20) of the measuring head insert.



#### CAUTION

The electrode cables for the reference, working and auxiliary electrode look identical. Observe the markings on the plugs, because the 3 cables must not be mixed up.

Plug the electrode cable (8-7) with the **RE** marking on the plug onto the metal contact of the reference electrode.



*Figure 45 Connecting a reference electrode* 

#### 3.3.2.3 Auxiliary electrode (AE)

The auxiliary electrode (e.g. 6.0343.100) can be placed directly in the measuring head.
#### Inserting the auxiliary electrode

Proceed as follows:

**1** Insert the auxiliary electrode into the opening (9-23) of the measuring head insert.



## CAUTION

The electrode cables for the reference, working and auxiliary electrode look identical. Observe the markings on the plugs, because the 3 cables must not be mixed up.

Plug the electrode cable (8-7) with the **AE** marking on the plug onto the metal contact of the auxiliary electrode.



*Figure 46 Connecting the auxiliary electrode* 

#### 3.3.3 Inserting the RDE measuring head

Once the RDE measuring head has been fully equipped, it can be inserted on the measuring head arm. Proceed as follows:

## **1** Installing the measuring head cover

Place the measuring head cover at an angle of approx. 45° in the guide bolt in the front part of the measuring head insert.

Fold back the measuring head cover and gently push it in place.



Figure 47 Installing the measuring head cover

The measuring head cover must snap into place with an audible click.



## 2 Inserting the stopper

Insert the stopper (3-5) into the pipetting opening.



*Figure 48* Inserting the stopper in the pipetting opening

## 3 Inserting the measuring head



#### WARNING

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.



## CAUTION

Do not apply pressure to the drive disk on the connector plate of the measuring head arm. Otherwise, the stirrer motor may be damaged.



Figure 49 Do not touch the drive disk

Use one hand to hold the measuring head arm on the rear and use the other hand to insert the measuring head into the connector plate of the measuring head arm.

The measuring head must snap into place with an audible click.



Figure 50 Inserting the measuring head

## 3.3.4 Connecting the inert gas supply



#### NOTICE

Only connect the inert gas supply if the RDE measuring head is used for VA trace analysis.

If the RDE measuring head is used for CVS analysis, then no inert gas supply is necessary.

Generally, nitrogen ( $N_2$ ) is used as inert gas in VA trace analysis for purging the measuring solution and for operating the MME pro, the SPE and the RDE. Only nitrogen of sufficient purity may be used for this.

For general polarography/voltammetry:

4.5 (w(N<sub>2</sub>) = 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<sub>2</sub>) = 99.999%)

#### 1 Filling the gas washing glass

- Unscrew the gas washing glass (1-6) from the measuring head arm.
- Fill the gas washing glass as follows:
  - Standard: Fill the gas washing glass halfway with distilled  $H_2O$ .
  - For long-term measurement with base electrolytes like acetic acid/acetate buffer solution or ammonia/ammonium chloride buffer solution, add the base electrolyte.
  - For measurements in organic solvents, fill with the solvent being used.
- Screw the gas washing glass back onto the measuring head arm.



*Figure 51 Gas washing glass* 

#### 2 Connecting the inert gas inlet

- Connect one end of the PVC tubing (6.1801.080) on the N<sub>2</sub> nipple of the 884 Professional VA.
- Connect the other end of the PVC tubing (6.1801.080) to the inert gas bottle connection.
- Set the inert gas pressure on the gas bottle to p = 1.0 to 1.2 bar (or 14.5 to 17.4 psi or 0.1 to 0.12 MPa) using the reducing valve.
- Open the gas inlet on the gas bottle.



*Figure 52* Nipple for inert gas supply

## **3.4 Equipping the SPE measuring head**



The measuring head insert (*see figure 13, page 28*) is made of PTFE. Do not use sharp objects around it to ensure the material is not damaged.

#### 3.4.1 Preparing the SPE measuring head



#### NOTICE

For equipping, Metrohm recommends placing the SPE measuring head in the measuring head holder and only then inserting it onto the measuring head arm.

**1** Remove the stopper (11-5) from the pipetting opening to remove the measuring head cover.



*Figure 53 Removing the stopper from the pipetting opening* 

2 Pull the slide lock (11-4) on the top of the measuring head cover towards you and, at the same time, tilt the measuring head cover to an angle of approx. 45° and remove it.



*Figure 54 Removing the measuring head cover* 

## **3** Connecting the gas inlet

- Insert the PTFE tubing for adding gas to the solution (6.1829.030) through the opening (13-18).
- Pull the transparent inner tubing through as far as it will go.
- Ensure that the green kink protection is protecting the entire piece of tubing.



*Figure 55 Inserting the gas inlet* 

- Connect the tubing to the PURGE threaded opening (12-8) and tighten it hand-tight.
- Finally, tighten the tubing nipple using the wrench provided (6.2739.000).



*Figure 56 Connecting the gas inlet* 

#### 4 Inserting the stirrer

- Screw one end of the flexible shaft to the stirrer using the clamping screw. While fastening the flexible shaft, push it through as far as it will go.
- Tighten the flexible shaft. To do so, screw the clamping screw for the flexible shaft into the stirrer using 2 wrenches (6.2739.000). In the process, ensure that the flexible shaft is not kinked.



*Figure 57 Screwing in the flexible shaft* 

• Insert the stirrer into the opening (13-**14**) and press it down as far as it will go.

- Fasten the other end of the flexible shaft to the drive shaft (12-9) using the second clamping screw. While doing so, push the flexible shaft through far enough so that the course is as straight as possible.
- Screw the clamping screw for the flexible shaft to the drive shaft using 2 wrenches (6.2739.000). In the process, ensure that the flexible shaft is not kinked.



*Figure 58* Inserting and connecting the stirrer

#### **5** Inserting the stopper

• If the openings (13-21)-(13-26) are not needed (for manual operation), seal them with the stoppers provided (6.2709.110).



#### NOTICE

The stopper shown in light blue in the figure has a smaller diameter than the others. Keep this in mind when inserting the stopper.



*Figure 59 Inserting the stopper* 

## **3.4.2 Preparing the electrode and inserting it in the SPE measuring head**

The 884 Professional VA uses the 3-electrode principle. The following electrodes are located on the screen-printed electrode (SPE):

- Working electrode (WE)
- Reference electrode (RE)
- Auxiliary electrode (AE)



#### NOTICE

Also observe the notes contained in the electrode leaflets that can be downloaded from the *Metrohm website* using the corresponding article number.

#### Preparing and inserting the electrode

NOTICE

#### **1** Preparing the electrode



Only hold the electrode by its edges. Contaminations on the electrode surface may falsify the measurement curves.

Prepare the electrode holder and the electrode according to the instructions in the *Electrode shaft for screen-printed electrodes (SPE)* document (8.0109.8014XX). Carry out the following tasks:

- Preparing the electrode holder
  - Insert the silicone seal in the electrode shaft
  - Position the supporting ring
  - Close the electrode shaft with the nut
- Inserting the electrode
  - Align the electrode with the symbol on the electrode shaft
  - Insert the electrode in the electrode shaft
  - Tighten the nut
- 2 Inserting the electrode holder in the measuring head insert

Insert the electrode holder with the electrode from above into the opening for the electrode (13-19).





**3** Align the printed side of the electrode with the stirrer.



*Figure 61 Aligning the electrode* 

## **Connecting electrodes**



#### CAUTION

The electrode cables for the reference, working and auxiliary electrode look identical. Observe the markings on the plugs. The 3 cables must not be mixed up.

## **1** Connecting the working electrode

Plug the electrode cable (12-7) with the **WE** marking on the plug onto the corresponding metal contact **WE** of the electrode shaft.



*Figure 62 Connecting the working electrode* 

## 2 Connecting a reference electrode

Plug the electrode cable (12-7) with the **RE** marking on the plug onto the corresponding metal contact **RE** of the electrode shaft.



Figure 63 Connecting a reference electrode

## **3** Connecting the auxiliary electrode

Plug the electrode cable (12-7) with the **AE** marking on the plug onto the corresponding metal contact **AE** of the electrode shaft.



*Figure 64 Connecting the auxiliary electrode* 

## 3.4.3 Inserting the SPE measuring head

Once the SPE measuring head has been fully equipped, it can be inserted on the measuring head arm. Proceed as follows:

#### **1** Installing the measuring head cover

Place the measuring head cover at an angle of approx. 45° in the guide bolt in the front part of the measuring head insert.

Fold back the measuring head cover and gently push it in place.



*Figure 65 Installing the measuring head cover* 

The measuring head cover must snap into place with an audible click.

## 2 Inserting the stopper

Insert the stopper (3-5) into the pipetting opening.



*Figure 66* Inserting the stopper in the pipetting opening

#### **3** Inserting the measuring head



#### WARNING

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.



## CAUTION

Do not apply pressure to the drive disk on the connector plate of the measuring head arm. Otherwise, the stirrer motor may be damaged.



*Figure 67* Do not touch the drive disk

Use one hand to hold the measuring head arm on the rear and use the other hand to insert the measuring head into the connector plate of the measuring head arm.

The measuring head must snap into place with an audible click.



Figure 68 Inserting the measuring head

#### 3.4.4 Connecting the inert gas supply

Generally, nitrogen ( $N_2$ ) is used as inert gas in VA trace analysis for purging the measuring solution and for operating the MME pro, the SPE and the RDE. Only nitrogen of sufficient purity may be used for this.

For general polarography/voltammetry:

4.5 (w(N<sub>2</sub>) = 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<sub>2</sub>) = 99.999%)

#### 1 Filling the gas washing glass

- Unscrew the gas washing glass (1-6) from the measuring head arm.
- Fill the gas washing glass as follows:
  - Standard: Fill the gas washing glass halfway with distilled  $H_2O$ .
  - For long-term measurement with base electrolytes like acetic acid/acetate buffer solution or ammonia/ammonium chloride buffer solution, add the base electrolyte.
  - For measurements in organic solvents, fill with the solvent being used.
- Screw the gas washing glass back onto the measuring head arm.



Figure 69 Gas washing glass

#### 2 Connecting the inert gas inlet

- Connect one end of the PVC tubing (6.1801.080) on the N<sub>2</sub> nipple of the 884 Professional VA.
- Connect the other end of the PVC tubing (6.1801.080) to the inert gas bottle connection.
- Set the inert gas pressure on the gas bottle to p = 1.0 to 1.2 bar (or 14.5 to 17.4 psi or 0.1 to 0.12 MPa) using the reducing valve.
- Open the gas inlet on the gas bottle.



*Figure 70 Nipple for inert gas supply* 

## **3.5 Establishing the tubing connections**

Depending on the peripheral devices you are using with the measuring instrument (manual, semiautomated or automated determinations), different tubing connections have to be established. All tubing connections that can be used are described below.

#### 3.5.1 Removing the measuring head cover

Remove the measuring head cover to establish the tubing connections.



#### CAUTION

The measuring head insert (*see figure 5, page 16*) is made of PTFE. Do not use sharp objects around it to ensure the material is not damaged.

#### Removing the measuring head cover

**1** Remove the stopper (3-5) from the pipetting opening to remove the measuring head cover.





2 Pull the slide lock (3-4) on the top of the measuring head cover towards you and, at the same time, tilt the measuring head cover to an angle of approx. 45° and remove it.



*Figure 72 Removing the measuring head cover* 

## 3.5.2 Installing the 4-way micro dosing tip

The 4-way micro dosing tip (6.1824.000) can be used to connect the 884 Professional VA to dosing devices of the type 800 Dosino and to add auxiliary solutions and standard solutions automatically. Information on the electrical connection of dosing devices can be found in *Chapter 3.6.3, page 97*.

#### Installing a 4-way micro dosing tip in the measuring head

Proceed as follows:

- **1** Remove the stopper from the screw nipple of the threaded opening *(5-16)*.
- **2** Loosen the screw nipple in the threaded opening a little.

This slightly loosens the O-ring located on the bottom of the screw nipple.

- **3** Feed the 4-way micro dosing tip through the opening from below (5-**14**).
- 4 Insert the 4-way micro dosing tip into the screw nipple of the threaded opening (5-**16**) until it stops.



Figure 73 Inserting the 4-way micro dosing tip

**5** Tighten the screw nipple in the threaded opening again.

# Connecting the PTFE capillaries of the 4-way micro dosing tip to a dosing unit

Proceed as follows:

**1** Screw the PTFE capillaries to the dosing units (port 1).





Sealing unused PTFE capillaries of the 4-way micro dosing tip

To prevent accidental aspiration of solution from the measuring vessel, unused PTFE capillaries must be sealed. Proceed as follows:

**1** Screw a coupling (6.1808.000) to each unused PTFE capillary.

**2** Screw a threaded stopper (6.1446.040) to each coupling (6.1808.000).



Figure 75 Sealing the PTFE capillary of a 4-way micro dosing tip

#### 3.5.3 Installing capillaries

You can add the samples from a Sample Processor in an automated way by using capillaries. The capillaries are connected to the peristaltic pump installed on the Sample Processor and/or a dosing unit *(see figures in Chapter 5.1.3.2, page 115)*. Several pressure screws (e.g. 6.2744.010) are required to attach the capillaries.

In order to achieve optimum analysis results, the capillary connections have to be as tight as possible and free of dead volume. Dead volume occurs if 2 capillary ends connected to each other do not fit exactly, thus allowing liquid to escape. There are 2 possible causes for this:

- The capillary ends do not have exactly flat edges.
- The 2 capillary ends do not completely meet.

We recommend using the capillary cutter (6.2621.080) to obtain exactly flat edges of capillaries.

#### Installing the capillary in the measuring head

We recommend using a PEEK capillary (e.g. 6.1831.020) in the measuring head. Proceed as follows:



Figure 76 Inserting a PEEK capillary into the measuring head

- **1** Feed the capillary through the opening (5-**28**) of the measuring head insert.
- 2 Slide the pressure screw over the capillary. Ensure that the capillary protrudes 1 to 2 mm from the tip of the pressure screw.
- **3** Push the capillary into the threaded opening **SMP** (4-**5**) of the measuring head connector plate until it stops.
- **4** Only then start turning the pressure screw, while holding the capillary firmly in place.
- **5** Cut the capillary to the desired length using the capillary cutter.

In order to prevent diffusion between the solution in the capillary and the solution in the measuring vessel, make sure that the end of the capillary is positioned above the measuring solution.

#### Installing the capillary on the tubing connector of the measuring head arm

We recommend using PTFE capillaries (e.g. 6.1803.020) between the tubing connector of the measuring head arm *(see chapter 2.3.3, page 17)* and the dosing unit and/or peristaltic pump. Proceed as follows:

- **1** Slide the pressure screw over the capillary. Ensure that the capillary protrudes 1 to 2 mm from the tip of the pressure screw.
- **2** Push the capillary into the threaded opening **SMP** (6-**4**) of the tubing connector of the measuring head arm as far as it will go.
- **3** Only then start turning the pressure screw, while holding the capillary firmly in place.

#### Connecting a capillary to a dosing unit

To attach the capillary to the dosing unit using a pressure screw, you need an adapter for the thread on the dosing unit. Proceed as follows:

- 1 Screw the adapter (6.2744.080) to port 2 of the dosing unit.
- Slide the pressure screw (e.g. 6.2744.010) over the capillary (e.g. 6.1803.020). Ensure that the capillary protrudes 1 to 2 mm from the tip of the pressure screw.
- **3** Push the capillary into the threaded opening of the adapter until it stops.
- **4** Only then start turning the pressure screw, while holding the capillary firmly in place.



#### Connecting a capillary to the peristaltic pump

Proceed as follows:

**1** Install the peristaltic pump with all tubing connections (including capillaries).

Proceed in accordance with the information contained in the manual of the Sample Processor.

#### 3.5.4 Installing FEP tubing

With FEP tubing, you can add or aspirate the following solutions:

- Rinsing solution adding via 843 Pump Station and
- Waste solution aspirating via 843 Pump Station and
- Auxiliary solutions (e.g. buffer, electrolyte, VMS, etc.) adding via 800 Dosino with dosing unit

#### Installing FEP tubing in the measuring head



NOTICE

Use the appropriate FEP tubing from the tubing set (6.1829.070): For threaded openings **1** and **WASTE**, use the tubing with the long kink protection; and for threaded openings **2** and **3**, use the tubing with the short kink protection.

Proceed as follows:

- Insert the conical end of the FEP tubing into one of the openings (24
  27) in the measuring head insert.
- 2 Carefully pull the conical end of the tubing protruding on the bottom side of the measuring head insert through the opening using the sandpaper supplied.

Pull the tubing through the opening until the kink protection of the tubing sits flush on the opening of the measuring head insert.

- **3** Screw the tubing nipple of the FEP tubing into the required threaded opening (**1**, **2**, **3** or **WASTE**) of the measuring head connector plate.
- 4 Cut the FEP tubing on the underside of the measuring head insert to the required length.

In order to prevent diffusion between the solution in the tubing and the solution in the measuring vessel, make sure that the end of the tubing is positioned above the measuring solution. This does not apply to the tubing for draining the measuring solution; this tubing must touch the bottom of the measuring vessel.



*Figure 78* Inserting the FEP tubing into the measuring head

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## NOTICE

If more than 1 piece of tubing is to be installed, we recommend pulling in all of the pieces of tubing into the measuring head insert first and shortening them to the required length.

In order to simplify fastening the tubing nipples to the threaded openings, remove the measuring head from the instrument and screw the tubing nipples in place from bottom to top (in the order **3**, **2**, **WASTE**, **1**).

**5** Seal the unneeded openings using the stoppers (6.2709.110) provided (*see figure 21, page 37*).

#### Installing the measuring head cover

The measuring head cover must be on the measuring head during determinations. Proceed as follows:



Figure 79 Installing the measuring head cover

**1** Place the measuring head cover at an angle of approx. 45° in the guide bolt in the front part of the measuring head insert.

2

#### WARNING

If the measuring head cover is installed without due care, this may result in injury to the hand.

Make sure that your fingers do not get caught between the measuring head cover and the measuring head.

Fold back the measuring head cover and gently push it in place.

The measuring head cover must snap into place with an audible click.

**3** Insert the stopper (*3-5*) into the pipetting opening.



*Figure 80* Inserting the stopper in the pipetting opening

## Installing a piece of FEP tubing on the tubing connector of the measuring head arm

Proceed as follows:

**1** Screw the tubing nipple of the FEP tubing (e.g. 6.1805.530) into the required threaded opening (**1**, **2**, **3** or **WASTE**) of the tubing connector of the measuring head arm (*see figure 95, page 119*).

#### **Connecting FEP tubing to an 843 Pump Station**

Proceed as follows:

**1** Install the inlet and outlet tubing on the 2 pumps or the 843 Pump Station (*see figure 108, page 133*).

#### **Connecting FEP tubing to a rinsing canister**

A bottle cap (6.1602.115) is required to connect a piece of FEP tubing to a rinsing canister. *Figure 95, page 119* provides an overview of the tubing between the rinsing canister, 843 Pump Station and 884 Professional VA.



*Figure 81* Installing a bottle cap with pieces of tubing on a rinsing canister

Proceed as follows:

- **1** Insert a piece of FEP tubing (e.g. 6.1819.110) from above into the smallest threaded opening of the bottle cap (6.1602.115) until the tubing flare of the FEP tubing sits flush in the threaded opening.
- **2** Screw the bottle cap with inserted FEP tubing onto the larger of the two openings of the rinsing canister.
- **3** Screw the tubing nipple of the FEP tubing (e.g. 6.1805.530) into the smallest threaded opening of the bottle cap.

#### **Connecting FEP tubing to the waste canister**

A 5-way tubing connector (6.1828.020) is required to connect FEP tubing to a waste canister. *Figure 95, page 119* provides an overview of the tub-

ing between the waste canister, 843 Pump Station and 884 Professional VA.



*Figure 82 Connecting the 5-way tubing connector to a waste canister* Proceed as follows:

1 Unscrew the cap with the hole in the center from the waste canister.

- **2** Feed each of the 5 tubings from the 5-way tubing connector with the couplings already installed from below through the hole of the unscrewed cap.
- **3** Screw the cap with the 5-way tubing connector inserted back onto the waste canister.
- Screw the tubing nipples of the pieces of FEP tubing (e.g. 6.1805.530) to the couplings of the pieces of tubing.



#### NOTICE

In order to ensure that liquid is transported smoothly from and to the canisters, the canisters must not be air-tight. If necessary, loosen the screw caps a little.

#### Connecting FEP tubing to a dosing unit with auxiliary solution

Proceed as follows:



*Figure 83 Connecting FEP tubing to the dosing unit (auxiliary solution)* 

**1** Screw the tubing nipple of the FEP tubing (e.g. 6.1805.120) to port 1 of the dosing unit containing the auxiliary solution (e.g. buffer, electrolyte, VMS, etc.).

## **3.6 Connecting instruments electrically**

## 3.6.1 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

# i

Do not use a not permitted power cord!

#### **1** Plugging in the power cord

NOTICE

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

#### 3.6.2 Connecting the 884 Professional VA

The 884 Professional VA is connected to the computer with the supplied controller cable.

#### **Connecting the computer**

**1** Connect the controller cable (6.2151.000) to the "Controller" connector of the 884 Professional VA.



#### NOTICE

The plug on the 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.

**2** Connect the USB plug of the controller cable to an available USB connector on the computer.

#### Initializing the 884 Professional VA in viva

1 Start viva.

The following dialog window is displayed:

009-108	3 Save device	23
2	The new device '884 Professional VA' with serial number '99003' has been detected.	
	Should it be saved in the device table?	
	Yes No	

## 2 Click on Yes.

The following dialog window is displayed:

Properties - 884 Professional VA - 884_2				
General GLP	General GLP			
Device name	884_1			
Device type	884 Professional VA			
Program version	5.884.0012			
Device serial number	02117			
FPGA version	122			
Set to work	2015-06-23 09:26:27 UTC+2			
Remarks				
	·]			

- **3** Change the suggested device name if required.
- 4 Confirm with **OK**.

The instrument will be automatically listed in the device table of the **Configuration** program part.
### 3.6.3 Connecting an 800 Dosino

Up to 4 dosing devices of the type 800 Dosino can be connected directly to the 884 Professional VA. As an alternative, the dosing devices can be connected via an 846 Dosing Interface, a sample changer or any other supported instrument that also has MSB outputs.



#### NOTICE

An 800 Dosino is connected to Metrohm instruments via an MSB connector. Make sure that the flat side of the plug matches the marking on the socket.



Figure 84 Connecting a dosing device to an MSB socket



NOTICE

Information regarding assembling a Dosino with the dosing unit as well as operation and maintenance is available in the manual of the 800 Dosino.

# Connecting an 800 Dosino with dosing unit directly to the 884 Professional VA

**1** Connect the connection cable of the 800 Dosino to one of the 4 MSB connectors (2-**8**) of the 884 Professional VA.

The following dialog window is displayed:



2 Confirm with **OK**.

### Initializing a dosing unit in viva

- 1 Select the 884 Professional VA in the device table of the **Configuration** section of the program.
- 2 In the device table, click on the **Edit** button and select **Initialize**.

If a brand-new dosing unit is connected, the following dialog window is displayed:



#### Or:

If a dosing unit is connected that has been previously configured, then the following dialog window is displayed:



3 Click on **OK** if a brand-new dosing unit is used.

The following dialog window is displayed:

osing unit -							2
Dosing unit GLP	]						
Hardware							
	Name						
c	Iomment						
Device name / dosin	ig device				]		
Order	r number	6.3032.3	120		]		
Serial	Inumber	1113038	39		]		
Cylinder	r volume			2	mL		
Cylinder serial	Inumber			7203	]		
Parameters for prepa	aration						
Dosing	g port Prej	p/Empty	Dosing port 1		-		
Dosing rate Dosing port 1		ig port 1			1.00 🔽	mL/min	
Dosing rate Dosing port 2		[	maxir	num 🔽	mL/min		
Dosing rate Fill port		[	maxir		ml (min		
Desire web General cont		-i-l	[	maxi			
Dosing	grate ope	iciai porc		maxir	num 📺	mL/min	
Tubing parameters	ort		Lepath		Diamete		
Dosing port 1	Port 1	Ţ	80.0	]		" 03	ro ro
Desing port 1	Jort 2		00.0	]		2.0	
	-orca		0.0	) un		2.0	Inn
Fill port	Port 2		25.0	] cm		2.0	mm
Special port F	Port 4	-	0.0	cm		2.0	mm
¥alve disk							
	Rotating (	direction	automatic		-		
	1	Not over	Port 4		-		
						Capca	

**4** Configure the new dosing unit in this dialog window.

i

### NOTICE

Under **Tubing parameters**, adapt the parameters **Length** and **Diameter** to the actual installation. This ensures that functions such as **Prepare** or **Empty** work properly in **viva**. The lengths and diameters of the tubing connections in the measuring head arm are available in *Chapter 8.1, page 178*.

5 Click on Yes if you are using the already configured dosing unit.

The dosing unit will be automatically displayed in the **Dosing units** subwindow of the **Configuration** program part.



Check the **Tubing parameters** and adapt them to the actual installation, if necessary.

### **3.6.4 Connecting a Sample Processor**

The following Sample Processors can be used in conjunction with the 884 Professional VA:

- 858 Professional Sample Processor
- 919 IC Autosampler plus
- 814 USB Sample Processor
- 815 Robotic USB Sample Processor XL

Sample Processors are connected to the power supply with a power supply cable and and to the PC with a controller cable.



### WARNING

An incorrect supply voltage can damage the instrument. Operate the instrument only with the supply voltage specified for it.

In order to ensure a constant power supply, we recommend using an uninterruptible power supply (UPS).

### **Connecting to power supply and PC**

Proceed as follows:

- **1** Connect the Sample Processor to the power supply with the power supply cable (6.2122.0x0).
- **2** Connect the controller cable (6.2151.000) to the "Controller" connector of the Sample Processor.

# 1

# NOTICE

The plug on the 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.

**3** Connect the USB plug of the controller cable to an available USB connector on the PC.



We recommend connecting the Sample Processor directly to the PC and not to one of the two USB connections on the rear of the 884 Professional VA.

The following dialog window is displayed:



4 Click on Yes.

The following dialog window is displayed:

Properties - 858.0020 Profe	essional Sample Processor - 858 Professional Sa	x
General GLP		
Device name	858 Professional Sample Processor 1	
Device type	858.0020 Professional Sample Processor	
Program version	5.858.0011	
Device serial number	03693	
Set to work	2013-03-28 14:23:57 UTC+1	
Remarks		
		-'
	OK Cancel	

- **5** Change the suggested instrument name if required.
- 6 Confirm with **OK**.

The instrument will be automatically listed in the device table of the **Configuration** section of the program.

### 3.6.5 Connecting an external pump

If solutions are to be added or aspirated via an external pump, we recommend using the 843 Pump Station with membrane pump. The 843 Pump Station is connected to the tower of the Sample Processor with the 6.2141.300 cable.

#### **Connecting an 843 Pump Station**

Proceed as follows:

- **1** Connect the 843 Pump Station to the power grid with the power cord (6.2122.0x0).
- **2** Insert the remote plug of the cable (6.2141.300) into the "Remote 2" socket of the 843 Pump Station.



*Figure 85 Connecting an external pump using the remote cable* 

**3** Connect the 2 plugs of the cable (6.2141.300) for the pump connectors to the tower of the Sample Processor.

Make sure that the markings on the cables match the markings on the tower ("Ext. Pump 1" and "Ext. Pump 2").



*Figure 86 Connecting an external pump to the tower* 

# 3.6.6 Connecting USB devices directly to the measuring instrument

Additional USB devices can be incorporated using the USB interfaces on the measuring instrument. You can, for example, connect the following USB devices:

- Barcode reader
- Keyboard
- Mouse
- USB hub



### NOTICE

Use only USB connectors on the PC to connect Metrohm devices with a USB connector.

# 4 Start-up

The 884 Professional VA is operated exclusively via the **viva** computer software. You can find information on operating **viva** in the online help and in the VA and CVS tutorials.

Proceed as follows for the initial start-up of the 884 Professional VA:

### Preparing the system for starting up



### WARNING

### **Uncontrolled splashing of reagents**

Splashing reagents may result in injuries.

Operate the 884 Professional VA only with the measuring head in place and the measuring head arm lowered.

- **1** Insert the measuring head as described in "*Preparing the RDE measuring head*", page52.
- **2** Equip the measuring head with the electrodes (*see chapter 3.3.2, page 55*).
- **3** Establish the tubing connections in the measuring head and those between the 884 Professional VA and the peripheral devices (*see chapter 3.5, page 82*).
- **4** Attach the measuring head cover and the stopper (3-**5**) to the measuring head as described in "Installing the measuring head cover", page 90.
- **5** Insert the measuring vessel into the holder (1-3).

6	

### WARNING

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.

Lower the measuring head arm.

7 Place the drip pan (6.2711.090) into the drip pan holder (1-8).

8 Connect the instruments electrically (see chapter 3.6, page 94).

### Activating the calibrator

The calibrator built into the 884 Professional VA guarantees a very high measuring accuracy for 2 years after the initial start-up. You can find more detailed information about the calibrator in *Chapter 6.10, page 147*.

In viva, proceed as follows:

- **1** Select the 884 Professional VA in the device table of the **Configuration** section of the program.
- 2 In the device table, click on the Edit button and select Properties....
- 3 Open the **Calibrator** tab.

Properties - 884 Professional	I VA - 884_4
General MSB 1 MSB	B 2 MSB 3 MSB 4 Calibrator GLP
Calibrator serial number	
Set to work	. not activated Activate calibrator
Remarks	
Monitor calibrator —	
Exchange interval	700 days
Next check	
Message	
Message by e-ma	ail E-mail
🗌 Acoustic signal	
Action	
O Record message	<u>}</u>
O Display message	3
O Cancel determina	ation
	OK Cancel

# 4 Click on Activate calibrator and then on OK.

The set-to-work date of the calibrator and the calibrator's serial number are displayed on the **Calibrator** tab after the dialog window has been closed and opened again.

Properties - 884 Professional	VA - 884_4
General MSB 1 MSB	2 MSB 3 MSB 4 Calibrator GLP
Calibrator serial number	0815
Set to work	2015-08-18 09:34:41 UTC+2
Remarks	
Monitor calibrator	
Replacement interval	700 days
Next check	2017-07-18
Message	
Message by e-ma	il E-mail
🗌 Acoustic signal	
Action	
O Record message	
Display message	
Cancel determina	tion
	OK Cancel



# NOTICE

The calibration certificate is available online. Go to *http://www.metrohm.com/en/support-and-service/certificate-finder* and enter the serial number of the calibrator and download the certificate.

**5** If required, adjust the settings on the **Calibrator** tab (e.g. **Message** or **Action**).

# **5** System configuration

The following chapters contain figures of possible system configurations for the following operating modes:

- Manual operation
- Semiautomated operation
- Automated operation

The parts that are required in addition to the 884 Professional VA and the VA or CVS electrode kit are listed for each operating mode. In addition, the viva software is required for controlling the instrument and evaluating the measurement.

# 5.1 884 Professional VA for VA trace analysis

# 5.1.1 Manual operation for VA trace analysis

In manual operating mode, all solutions are added via the pipetting opening (5-**22**). Therefore, only the 3 electrodes have to be inserted and connected; no tubing is required other than that described in *Chapter 3.2*.

You will need the 884 Professional VA (2.884.0110) and a VA electrode kit (6.5339.0x0).



*Figure 87 Manual operating mode – Electrical connection* 

# 5.1.2 Semiautomated operating mode for VA trace analysis

In semiautomated operating mode, samples as well as standard and auxiliary solutions can be added either automatically via dosing units or manually via the pipetting opening.

For the system configurations shown in the following figures, you will need a VA electrode kit (6.5339.0x0) in addition to the 884 Professional VA semiautomated (2.884.1110).

### 5.1.2.1 **Connecting instruments electrically**

The following figure shows an exemplary electrical connection of the devices in semiautomated operating mode:





# 5.1.2.2 Establishing the tubing connections

The following figures provide an overview of the tubing setups in semiautomated operating mode:

Figure 89	Semiautomated operating mode – Tubing setup with 2 dosing units
Figure 90	Semiautomated operating mode – Tubing setup – 884 Professional VA details



*Figure 89 Semiautomated operating mode – Tubing setup with 2 dosing units* 







a FEP tubing (6.1829.070 tubing set) Use the tubing with the short kink protection.

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### 5.1.3 Automated operating mode for VA trace analysis

The 884 Professional VA can be operated in conjunction with the following Sample Processors:

- 858 Professional Sample Processor
- 919 IC Autosampler plus
- 814 USB Sample Processor
- 815 Robotic USB Sample Processor XL

For the system configurations shown in the following figures, you will need the following options in addition to the 884 Professional VA semiautomated (2.884.1110) and a VA electrode kit (6.5339.0x0):

- 919 IC Autosampler plus (2.919.0020)
- 843 Pump Station (2.843.0240)
- Remote cable (6.2141.300)

### 5.1.3.1 Connecting instruments electrically

The following figure shows an exemplary electrical connection of the devices in automated operating mode:



### NOTICE

We recommend connecting all controller cables (6.2151.000) directly to the PC and not cascading them through the USB ports on the rear of the instrument.





# 5.1.3.2 Establishing the tubing connections

The following figures provide an overview of the tubing setup in automated operating mode:

Figure 92	Automated operating mode – Tubing setup with 2 Dosinos
Figure 93	Automated operating mode – Tubing setup – 884 Professional VA details
Figure 94	Automated operating mode – Tubing setup – 800 Dosino details
Figure 95	Automated operating mode – Tubing setup – Add- ing rinsing solution and pumping out the waste solution
Figure 96	Automated operating mode – Tubing setup – Rins- ing and waste solutions – 884 Professional VA details
Figure 97	Automated operating mode – Tubing configuration – Measuring head details









*Figure 93 Automated operating mode – Tubing setup – 884 Professional VA details* 



*Figure 94 Automated operating mode – Tubing setup – 800 Dosino details* 



Figure 95 Automated operating mode – Tubing setup – Adding rinsing solution and pumping out the waste solution





*Figure 96* Automated operating mode – Tubing setup – Rinsing and waste solutions – 884 Professional VA details

- a FEP tubing (6.1829.070 tubing set) Use the tubing with the long kink protection.
- **b FEP tubing (6.1829.070 tubing set)** Use the tubing with the short kink protection.



*Figure 97* Automated operating mode – Tubing configuration – Measuring head details

# 5.2 884 Professional VA for CVS

# 5.2.1 Manual operating mode for CVS

In manual operating mode, all solutions are added via the pipetting opening (9-22). Therefore, only the 3 electrodes must be inserted and connected on the measuring head; no tubing is required.

You will need the 884 Professional VA for CVS (2.884.0210) and CVS electrode kit (6.5339.0x0).





# 5.2.2 Semiautomated operating mode for CVS

In semiautomated operating mode, samples, additives and the VMS can be added either automatically via dosing units or manually via the pipetting opening.

For the system configurations shown in the following figures, depending on the application you will also need 2 800 Dosinos (2.800.0020) and the equipment with 2 dosing units in addition to the 884 Professional VA semiautomated for CVS (2.884.1210) and a CVS electrode kit (6.5339.0x0).

### 5.2.2.1 Connecting instruments electrically

The following figure shows an exemplary electrical connection of the devices in semiautomated operating mode:



*Figure 99 Semiautomated operating mode – Electrical connections* 

### 5.2.2.2 Establishing the tubing connections

The following figures provide an overview of the tubing setup options in semiautomated operating mode:

Figure 100	Semiautomated operating mode – Tubing setup with 2 dosing units
Figure 101	Semiautomated operating mode – Tubing setup with 4 dosing units
Figure 102	Semiautomated operating mode – Tubing setup – 884 Professional VA details



*Figure 100* Semiautomated operating mode – Tubing setup with 2 dosing units



*Figure 101* Semiautomated operating mode – Tubing setup with 4 dosing units



*Figure 102* Semiautomated operating mode – Tubing setup – 884 Professional VA details

### a FEP tubing (6.1829.070 tubing set)

Use the tubing with the long kink protection.

# 5.2.3 Automated operating mode for CVS

The 884 Professional VA can be operated in conjunction with the following Sample Processors:

- 858 Professional Sample Processor
- 919 IC Autosampler plus
- 814 USB Sample Processor
- 815 Robotic USB Sample Processor XL

For the system configurations shown in the following figures, you will need the following options in addition to the 884 Professional VA semiautomated for CVS (2.884.1210) and a CVS electrode kit (6.5339.0x0):

- 2 800 Dosinos (2.800.0020) and the equipment with 2 dosing units
- 858 Professional Sample Processor (2.858.0110)
- 843 Pump Station (2.843.0240)
- Remote cable (6.2141.300)

### 5.2.3.1 Connecting instruments electrically

The following figure shows an exemplary electrical connection of the devices in automated operating mode:



### NOTICE

We recommend connecting all controller cables (6.2151.000) directly to the PC and not cascading them through the USB ports on the rear of the instrument.





# 5.2.3.2 Establishing the tubing connections

The following figures provide an overview of the tubing setup options in automated operating mode:

Figure 104	Automated operation – Tubing setup – Overview
Figure 105	Automated operating mode – Tubing setup – 884 Professional VA details
Figure 106	Automated operation – Tubing setup – 800 Dosino details
Figure 107	Automated operation – Tubing setup – 858 Profes- sional Sample Processor details – Guide chain
Figure 108	Automated operation – Tubing setup – Adding rins- ing solution and pumping out the waste solution
Figure 109	Automated operating mode – Tubing setup – Rins- ing and waste solutions – 884 Professional VA details



Figure 104 Automated operation – Tubing setup – Overview



*Figure 105* Automated operating mode – Tubing setup – 884 Professional VA details

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a FEP tubing (6.1829.070 tubing set) Use the tubing with the long kink protection.







*Figure 107* Automated operation – Tubing setup – 858 Professional Sample Processor details – Guide chain


Figure 108 Automated operation – Tubing setup – Adding rinsing solution and pumping out the waste solution



*Figure 109* Automated operating mode – Tubing setup – Rinsing and waste solutions – 884 Professional VA details

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- a FEP tubing (6.1829.070 tubing set) Use the tubing with the long kink protection.
- **b FEP tubing (6.1829.070 tubing set)** Use the tubing with the short kink protection.

## 6 Operation and maintenance

### 6.1 Care



### 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.

The instrument requires appropriate care. Excess contamination of the instrument results in malfunctions and a reduction in the service life of the sturdy mechanical and electronic components.

Clean the housing and the measuring head with a damp cloth. A mild detergent can be used to remove stubborn contaminations.



### CAUTION

The instrument has been designed so that aggressive media are largely prevented from entering the instrument. However, if liquids should get into the instrument, unplug the power plug immediately to avoid serious damage to the instrument electronics. In such a case, contact Metrohm Service.

Spilled chemicals and solvents should be removed immediately. In particular, the plug connections (particularly the power plug) should be protected from contamination.

## 6.2 Maintenance by Metrohm Service

Maintenance of the instrument is best carried out as part of annual service, which is performed by specialist personnel from Metrohm. A shorter maintenance interval is recommended 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.

## 6.3 **Replacing the measuring head**

Make sure that no determinations are being carried out when you replace the measuring head.



#### WARNING

### Leaking of chemical substances

Chemical substances can cause chemical burns and skin damage.

- Empty the tubing before you remove the measuring head.
- Tilt the measuring head arm up before you remove the measuring head.



### CAUTION

### LED blinking pattern indicates standby potential

The electrodes may be damaged.

Check the blinking pattern of the "Status" LED. The measuring head may not be removed while standby potential is being applied to the electrodes (blinking pattern: on for a long time - off for a short time).



### CAUTION

#### Stirrer motor is in operation

The stirrer motor may be damaged.

Switch off the stirrer motor before you remove or insert the measuring head.

Proceed as follows:

- **1** Tilt the measuring head arm up.
- **2** Hold the measuring head with both hands, push the metal clip on the bottom of the measuring head arm upwards and, at the same time, pull the measuring head away.



Figure 110 Removing the measuring head

**3** Place the measuring head with the electrodes and tubings in the park station.



### NOTICE

If the measuring head is not used for an extended period, then the reference electrode must be removed from the measuring head and stored separately. Observe the notes on maintenance and care in the electrode leaflet.



*Figure 111 Rear WASTE connector* 

When using a Multi-Mode Electrode pro along with additional automated aspiration of the measuring solution, watch for traces of mercury in the indicated connector (WASTE) when removing the MME measuring head. Remove any traces using a mercury drop catcher (6.2406.000).



### CAUTION

Do not apply pressure to the drive disk on the connector plate of the measuring head arm, as this could damage the stirrer motor.



Figure 112 Do not touch the drive disk

Use one hand to hold the rear of the measuring head arm and use the other hand to insert the new measuring head into the connector plate of the measuring head arm.

The measuring head must snap into place with an audible click.



### WARNING

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.

Lower the measuring head arm again.

## 6.4 Servicing the Multi-Mode Electrode pro (MME pro)

Multi-Mode Electrode pro has to receive regular maintenance to ensure its functionality. In terms of maintenance, a distinction is made between short and full maintenance. Maintenance of the Multi-Mode Electrode pro encompasses the following tasks:



NOTICE

For more detailed information about the individual tasks, observe the *Multi-Mode Electrode pro* document (8.110.8018XX) and the *Elec-*

trodes in Voltammetry multimedia guide (A.717.0003).

### 1 Short maintenance

- Topping up the mercury
- Replacing the capillaries
- Replacing the needle
- Adjusting the needle valve

### 2 Full maintenance

- Emptying the mercury reservoir
- Removing the capillaries
- Removing the needle
- Cleaning the electrode body
- Mounting the new capillaries
- Inserting a new needle
- Top up the mercury
- Adjusting the needle valve

## 6.5 Servicing the screen-printed electrode (SPE)



### NOTICE

Screen-printed electrodes (SPEs) have a limited service life. The electrode must be replaced at regular intervals. The service life of the electrode depends on the electrode type and the performed applications. Additional information can be found in the corresponding application documentation.

## 6.6 Performing a Dummy cell test

Should you note any problems or irregularities during or after determinations (e.g. noisy curves), you can perform a dummy cell test. In this test, the hardware and the electrode cables up to the measuring head are checked; this allows potential problems to be localized or excluded. The electrodes are not tested with the **Dummy cell test**.

Proceed as follows:

1 Load the **Dummy cell test** method template in **viva**.

**2** Plug the 3 electrode cables onto the contacts of the dummy cell (6.2813.050).

Observe the correct assignment of the electrode cables (WE, RE and AE).

**3** Place the measuring head cover and the stopper (3-5) onto the measuring head.

**4** Start the determination; at the prompt, select the name of the 884 Professional VA being used from the list and wait until the measurement is completed.

If the curve meets the following conditions, then defects in the hardware or the electrode cables can be ruled out:

- The curve must be linear.
- At -2.0 V, the current should be -1.67 mA  $\pm 0.33$  mA.
- At +2.0 V, the current should be +1.67 mA  $\pm$  0.33 mA.



Figure 113 Dummy cell test – Ideal curve progression

**5** If the resulting curve is not consistent with this representation and the problems with the determinations persist, contact the local Metrohm Service.

### 6.7 Performing a Test Pb in standard solution

The Test Pb in standard solution is a standard method for testing the electrode function. This method involves measuring the lead concentration in a provided ionic standard solution using a DME.

The following reagents are required for the test:

- 20 mL of ultrapure water
- 0.5 mL of potassium chloride c(KCl) = 3 mol/L (Metrohm no.
   6.2308.020)
- 0.3 mL of lead ionic standard solution  $\beta(Pb) = 1 \text{ g/L}$  (Metrohm no. 6.2301.100)

Proceed as follows to carry out the test:



1 In **viva**, click on the icon for the **Method** program part.

2 Open the **New method** dialog window using the **File** ► **New...** menu.

New method	×
New method Templates Blank method Dummy cell test CVS/CPVS PUTrace analysis Test Pb in standard solution ASV determination (DP, standard addition), manual ASV determination (DP, standard addition), manual DV determination (DP, standard addition), and addition of the standard addition of the stan	Description      Test Pb in standard solution      Standard method for testing the electrode function by manual determination of lead with polarography and the calibration method 'standard addition'
ASV determination (DP, standard addition), automated ASV determination (DP, external calbration), manual ASV determination (SQW, standard addition), manual ASV determination (SQW, standard addition), manual	
	OK Cancel

3 Select **Templates** ► **Trace analysis** ► **Test Pb in standard solution** and click **[OK]**.

The method template opens.

- **4** Select the name of the 884 Professional VA in use from the list in the DP voltammetry command "POL" on the **General/Hardware** tab.
- 5 Using the File ► Save as... menu, save the method under the name Test Pb in standard solution.
- 6 Click on the icon of the **Workplace** program part.
- 7 In the **Run** window, select the **Test Pb in standard solution** method created from the method template on the **Single determination** tab in the **Method** field.
- 8 Select the **Sample** option in the **Sample type** field.
- 9 Enter "0.1 mL" in the Sample amount field.
- **10** Press **[Start]** to start the analysis.



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- **11** Add 20 mL of ultrapure water to the empty measuring vessel of the 884 Professional VA and confirm with **[OK]**.
- **12** Add 0.5 mL of potassium chloride c(KCl) = 3 mol/L to the measuring vessel and confirm with **[OK]**.
- **13** Using a pipette, add 100  $\mu$ L of lead ionic standard solution  $\beta$ (Pb) = 1 g/L to the measuring vessel as a sample and confirm with **[OK]**.
- **14** The measuring solution is measured twice.
- **15** Using a pipette, add 100  $\mu$ L of lead ionic standard solution  $\beta$ (Pb) = 1 g/L to the measuring vessel through the pipetting opening (5-22) and confirm with **[OK]**.
- **16** The spiked sample solution is measured twice.
- **17** Using a pipette, add 100  $\mu$ L of lead ionic standard solution  $\beta$ (Pb) = 1 g/L to the measuring vessel through the pipetting opening (5-22) and confirm with **[OK]**.
- **18** The double-spiked sample solution is measured twice. Finally, the test is completed.
- **19** The measurement can be evaluated using the results displayed in the database under **Results overview**. The following criteria should be met:
  - Result (POL.Pb.Concentration):  $\beta$ (Pb) = 0.9–1.1 g/L
  - Trueness (Recovery): 90–110%
  - Relative error (POL.Pb.Concentration.RSD): ≤ 3%

## 6.8 **Replacing electrode cables**

It may be the case that one or more of the 3 electrode cables are damaged (e.g. accidental pinching in the measuring head cover). If the electrode cables are damaged, then the proper functioning of the measuring instrument can no longer be ensured. In this case, the entire cable set (6.2112.100) needs to be replaced. Proceed as follows:



*Figure 114 Replacing electrode cables* 

- 1 Remove the measuring head from the measuring head arm.
- **2** Remove the stopper and the measuring head cover from the measuring head (*see Figure 35, page 52* and *Figure 36, page 53*).
- **3** Unscrew the screw on the right-hand side of the measuring head connector plate using a screwdriver.
- **4** Push the triple plug contact forward from the rear through the measuring head connector plate.
- **5** Remove the cable set.
- 6 Insert the new cable set.



### NOTICE

Make sure that the ellipsis-shaped recess on the cylinder is facing up when you insert the new cable set. This is important to ensure that the 3 electrodes are connected to the correct connectors inside the measuring instrument. 7 Carefully tighten the screw on the right-hand side of the measuring head connector plate.

## 6.9 Adjusting the sample needle in the Sample Processor

If the entire sample volume is to be transferred from the Sample Processor, then the work position on the tower of the Sample Processor has to be set in such a way that the sample needle is located no more than 0.5 mm above the bottom of the sample vial. The work position settings must be defined in **viva**.

If required, adjust the sample needle in viva as shown in the figure:



### 6.10 Calibrator

The 884 Professional VA is equipped with a built-in calibrator with precisely specified reference values that are defined in the corresponding calibration certificate. This innovative component permits the performance of voltammetric measurements with increased reliability and precision. The measuring input of the 884 Professional VA is automatically adjusted to the reference values of the calibrator before each measurement and thus yields stable, precise and reproducible measured values over a long period, even under suboptimal measuring conditions. In this way, the 884 Professional VA automatically recalibrates itself before each measurement.

When the 884 Professional VA is started up, a prompt to activate the calibrator appears (see "Activating the calibrator", page 106). The automatic adjustment is active starting with this activation time. The certified reference values of the built-in calibrator are valid for 2 years. For this period, we guarantee that the adjustment is undertaken correctly and that the automatic calibration is accurate. Just before 2 years have elapsed, a prompt appears in the **viva** computer software stating that the calibrator should be replaced in order to ensure correct adjustment of the measuring input.

We therefore recommend having the calibrator replaced every 2 years. We can thus guarantee correct automatic self-adjustment of the 884 Professional VA over the entire service life of the instrument. In addition, you are well prepared for your audits. Please contact your local Metrohm representative to replace the calibrator. One of our trained Metrohm service engineers will then replace the calibrator as part of a preventive maintenance procedure.

## 6.11 Servicing the stirrer

### **Removing the stirrer**



*Figure 116 Loosening and removing the stirrer* 

 Loosen the clamping screw for the flexible shaft on the drive shaft (4-9) using 2 wrenches (6.2739.000).

- Pull the end of the flexible shaft out of the drive shaft.
- Remove the stirrer from the opening (5-15).

### 2 Removing the flexible shaft



*Figure 117 Removing the flexible shaft* 

- Loosen the flexible shaft. To do so, loosen the clamping screw for the flexible shaft using 2 wrenches (6.2739.000). In the process, ensure that the flexible shaft is not kinked.
- Pull the flexible shaft out of the stirrer shaft.

### **Disassembling the stirrer**



*Figure 118 Disassembling the stirrer* 

- **1** Fix the shaft with a wrench (6.2739.000). Unscrew the stirring bar counterclockwise.
- **2** Remove the stirring sleeve.

### **Cleaning the stirrer**



*Figure 119 Cleaning the stirrer* 

Clean the inner part of the stirring sleeve with a dry cloth.
 Remove heavy contamination with a damp cloth. Allow the stirring sleeve to dry properly.

2 Clean the shaft of the stirrer from outside with a cloth.

Remove heavy contamination with a damp cloth. Allow the shaft to dry properly.

### Screwing the stirrer together



*Figure 120 Screwing the stirrer together* 

**1** Attach the stirring sleeve to the shaft.

**2** Screw the stirring bar to the shaft clockwise.

### **Reinserting the stirrer**

### **1** Screwing in the flexible shaft



*Figure 121 Screwing in the flexible shaft* 

- Screw one end of the flexible shaft to the stirrer using the clamping screw. While fastening the flexible shaft, push it through as far as it will go.
- Tighten the flexible shaft. To do so, screw the clamping screw for the flexible shaft into the stirrer using 2 wrenches (6.2739.000). In the process, ensure that the flexible shaft is not kinked.



### 2 Inserting and connecting the stirrer

*Figure 122* Inserting and connecting the stirrer

- Insert the stirrer into the opening (5-15) and press it down as far as it will go.
- Fasten the other end of the flexible shaft to the drive shaft (4-9) using the second clamping screw. While doing so, push the flexible shaft through far enough so that the course is as straight as possible.

 Screw the clamping screw for the flexible shaft to the drive shaft using 2 wrenches (6.2739.000). In the process, ensure that the flexible shaft is not kinked.

## 6.12 Relocating the 884 Professional VA

If you wish to relocate the 884 Professional VA within the laboratory, proceed as follows:

Make sure that all electrical and tubing connections are disconnected before carrying the instrument to a new location.



#### CAUTION

### Instrument components breaking off

Lifting the instrument by the measuring vessel holder or the measuring head arm may result in injuries if the instrument is dropped.

Hold the instrument by the drip pan holder and at the rear of the measuring head arm.

**1** Remove the drip pan from the holder.

2 If the measuring vessel is filled, tilt the measuring head arm up.

**3** Remove the measuring vessel.

4	

### WARNING

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.

Lower the measuring head arm again.

**5** Hold the instrument with one hand on the drip pan holder and with the other hand at the rear of the measuring head arm and relocate the instrument.

# 7 Troubleshooting

# 7.1 List of faults

### 7.1.1 General problems

Problem	Cause	Remedy
An electrode cable has broken.	The cable has accidentally been jammed in the mea- suring head cover or a cable contact has been kinked.	Replace the electrode cable set .
Solution is leaking between the mea- suring instrument	The measuring head is not correctly locked in place on the measuring head arm.	Let the measuring head snap into place. The click must be audible.
and the measuring head.	The sealing rings have aged or are defective.	Contact Metrohm Service.
The electrode test fails.	One or several electrodes are not connected.	Check the electrode cable connections.
	One or several electrodes are defective.	Perform maintenance in accordance with the electrode leaflet. You can find more detailed information at <i>http://va-elec-trodes.metrohm.com</i> .
	The measuring vessel con- tains too little solution or is empty.	Check the fill level in the measuring vessel and add solution if necessary.
The measured values are widely scattered.The solutions are piper manually.tered.The solutions are not added via the pipettin opening.The solutions are meter automatically: Tubing nections are not seale the 4-way micro dosint for adding standard sec tions is not immersed the measuring solution	The solutions are pipetted manually.	Use an 800 Dosino with dosing unit.
	The solutions are not added via the pipetting opening.	Add solutions only via the pipetting opening .
	The solutions are metered automatically: Tubing con- nections are not sealed or the 4-way micro dosing tip for adding standard solu- tions is not immersed in the measuring solution.	Check the tubing connections and the 4-way micro dosing tip and replace if necessary.

Problem	Cause	Remedy
	One of the electrodes (nor- mally the working or refer- ence electrode) is not operational.	Service the electrode or replace the electrode in the case of a solid-state electrode (e.g. plati- num RDE).
	The stirrer or RDE is not rotating properly.	Inspect the drive. Service or replace the drive if necessary.
		For other application-specific problems, observe ( <i>see chapter 7.1.2, page 155</i> ) and ( <i>see chapter 7.1.3, page 165</i> ).
The measuring ves- sel overflows.	Incorrect pump times have been defined in <b>viva</b> .	Adjust the pump times.
	The volumes defined in the dosing commands in <b>viva</b> are too large.	Reduce the volumes.
The peak is no lon- ger recognized.	The reference potential has shifted.	Perform the maintenance procedure for the reference electrode in accordance with the electrode leaflet. You can find more detailed information at http://va-elec-trodes.metrohm.com.
	One or more of the solu- tions in use (water, electro- lyte, VMS, etc.) has become contaminated.	Apply fresh solution; use water from a differ- ent source.

## 7.1.2 884 Professional VA for VA trace analysis

Problem	Cause	Remedy
A peak is not found in either the sample or for the standard	The peak is not within the sweep range.	Check <b>Start potential</b> and <b>End potential</b> of the sweep in <b>viva</b> .
addition.	The electrolyte solution is too old.	Produce new electrolyte solution. Organic additives may only last 1 day or less.
	Organic constituents inter- fere with analysis.	Carry out UV digestion or equivalent sample preparation.
	The complexing agent has been omitted (adsorptive stripping voltammetry).	Use complexing agent.
	No drops form at the capil- lary.	<ul><li>Check the MME pro.</li><li>Adjust the needle and capillary.</li></ul>

Problem	Cause	Remedy
		<ul> <li>If necessary, replace the capillary or needle (see the Multi-Mode Electrode pro docu- ment (8.110.8018XX)).</li> </ul>
	The pH value of the mea- suring solution is not cor- rect.	Check the pH value and adjust it by adding a buffer, acid or base if necessary.
A peak was not found when measur- ing the sample but the standard addi- tion is being evalu-	The peak in the sample has shifted.	Ensure that the peak actually originates from the analyte. If it does, set the characteristic potential and have the results recalculated. If it does not, locate the cause based on "Double peak present" and resolve the problem.
	The concentration of the ion to be determined is too small.	<ul> <li>Use more sample.</li> <li>Use stripping voltammetry (HMDE) instead of polarography (DME, SMDE) if the application allows.</li> <li>Extend the deposition time (only HMDE).</li> </ul>
	The deposition time for stripping voltammetry is too short.	In the voltammetry command (DP, SQW), increase the deposition time under <b>Potentio-</b> static pretreatment.
A slight hissing noise can be heard at the measuring head.	The measuring head is not correctly locked in place on the instrument. This can allow nitrogen to escape at the connection.	Remove the measuring head and put it back in place until it clearly clicks in place.
	The seals at the transitions are too old.	Contact Metrohm Service for how to proceed.
Contacts for the SPE are corroded.	Repeated penetration of liquids into the electrode shaft.	Replace the electrode holder.
Curves have a large amount of noise (all electrode types).	The needle is not adjusted.	<ul> <li>Adjust the needle (see the <i>Multi-Mode</i> <i>Electrode pro document (8.110.8018XX)</i>).</li> <li>In the case of an SMDE, alternatively use a DME if the application allows.</li> </ul>
	The capillary and/or needle is defective.	Replace the capillary and needle. If necessary, clean the MME pro (see the <i>Multi-Mode Elec-trode pro document (8.110.8018XX)</i> )

Problem	Cause	Remedy
	The tapping mechanism on the VA stand base is not functioning correctly.	<ul> <li>Check the positioning of the capillary in the MME pro. The capillary has to be centered in the retaining screw.</li> <li>The retaining screw is too loose or too tight. Use the wrench for MME (6.1247.220) to tighten the retaining screw to the right torque.</li> </ul>
	The electrolyte solution is too old.	Produce new electrolyte solution. Organic additives may only last 1 day or less.
	The mercury in the MME pro displays the black streaks of mercury oxide.	Completely remove and clean the electrode (see the <i>Multi-Mode Electrode pro document</i> (8.110.8018XX)).
	The reference electrode is not filled correctly (inside and outside).	Top up the reference electrode.
	The reference electrode's diaphragm is partially or completely blocked (e.g. from precipitation caused by incompatible solutions like KCl as a bridge electrolyte and $HClO_4$ as an electrolyte in the measuring solution).	Ensure the compatibility of the chemistry. Replace the bridge electrolyte if necessary (such as using a NaCl or KNO <sub>3</sub> solution in place of KCl).
Curves have a large amount of noise (with DME/SMDE).	The electrode drips at irreg- ular intervals.	<ul> <li>Check the tapping of the dripping on the MME pro. If necessary, center the capillary in the MME pro or readjust the anchoring of the capillary using the wrench for the MME (6.1247.220).</li> <li>Adjust the needle and capillary.</li> <li>If necessary, replace the capillary or needle (see the <i>Multi-Mode Electrode pro document (8.110.8018XX)</i>).</li> </ul>
	The electrode drips too quickly (with DME).	Increase the voltage step time in the voltam- metry command on the <b>Sweep</b> tab.
Curves have a large amount of noise (with HMDE).	The electrode surface is overloaded.	Check the deposition potential and time and reduce them if necessary.

Problem	Cause	Remedy
	No drops form at the capil- lary.	<ul> <li>Check the nitrogen connection and pressure.</li> <li>Ensure that the measuring head is attached correctly and locked in place.</li> <li>Replace the capillary and needle (see the <i>Multi-Mode Electrode pro document (8.110.8018XX)</i>).</li> </ul>
Curves have an unusual shape (with	The electrode cables were connected to the wrong	Install the electrode cables as follows:
SPE).	electrode contact.	<ul> <li>Connect the cable with the WL marking to the metal contact WE for the working electrode.</li> <li>Connect the cable with the RE marking to the metal contact RE for the reference electrode.</li> <li>Connect the cable with the AE marking to the metal contact AE for the auxiliary electrode.</li> </ul>
	The surface of the elec- trode was touched while installing the electrode.	Replace the electrode.
		During assembly, only hold the electrode by its edges. Avoid contact with the electrode surface.
	Measuring solution has penetrated the electrode holder and caused a short circuit.	Take apart the electrode holder. Clean all parts with ultrapure water to remove corrosive chemicals. Dry all parts thoroughly. If the con- nection socket of the electrode was rinsed as well, allow the electrode holder to dry for sev- eral hours.
Double peak pres- ent.	Organic constituents inter- fere with analysis.	Carry out UV digestion or equivalent sample preparation.
	The electrolyte solution is too old.	Produce new electrolyte solution. Organic additives may only last 1 day or less.
	A second substance is pres- ent at the same potential.	Spike the sample with that substance and run the analysis again. If the second peak became higher, the second element is present.
		<ul><li>Try out different electrolytes (e.g. substances acting as complexing agents).</li><li>Optimize the method parameters.</li></ul>

Problem	Cause	Remedy
	Complexing (e.g. Cu with chloride).	For Cu: Work without chloride in the electro- lyte or massively increase the chloride concen- tration.
	Formation of sparingly soluble compounds (e.g. Fe(III) in a neutral or alka- line solution).	<ul> <li>Optimize the measuring solution, e.g.:</li> <li>Use a different electrolyte for a different pH value.</li> <li>Add a complexing agent (such as citrate).</li> </ul>
No spiking (all elec- trode types).	The wrong standard solu- tion has been used or the concentration is too low.	<ul> <li>Increase the volume of the standard addition.</li> <li>Use a higher concentration.</li> <li>Reduce the sample amount accordingly.</li> </ul>
	The concentration of the analyte is too large.	Dilute the sample.
	The potential of the refer- ence electrode has shifted.	Service the reference electrode.
	Standard solutions that contain metal complexing agents need time to form the complex.	No actions are necessary.
Outliers/signal jumps are present in the voltammogram.	<i>The MME pro or compo- nents of the MME pro have been damaged.</i>	<ul> <li>Check the MME pro.</li> <li>If necessary, replace the capillary and needle (see the <i>Multi-Mode Electrode pro document (8.110.8018XX)</i>).</li> </ul>
	The potentiostat switches during measurement of the current measuring range.	Check the current measuring ranges used dur- ing the measurement and select a higher value for the lowest range if necessary.
	Air bubbles present in the reference electrode.	Check the internal reference system and the bridge electrolyte vessel.
	Gas bubbles present on the working electrode.	Remove the gas bubbles and prevent them from forming again.
SPE cannot be inserted into elec- trode holder.	The nut has been overtight- ened and closes off the slit in the silicone seal.	Loosen the nut to open the slit in the silicone seal.
	The supporting ring has rotated.	Loosen the nut. Reposition the supporting ring. Align the slit in the supporting ring with the slit in the silicone seal.

Problem	Cause	Remedy
Sensor shows no measuring signal (with SPE) even though analyte is present.	<i>The electrode is installed upside down.</i>	Reinstall the electrode. This time, align the electrode with the symbol on the electrode shaft. Only in this position does the electrode have electrical contact to the connection socket.
	The electrode was not pushed into the electrode shaft as far as it would go.	Loosen the nut. Push the electrode into the electrode shaft as far as it will go. Then, tighten the nut again.
	The SPE is defective.	Replace the SPE.
Standard addition curves are not linear or poorly reproduci-	The method parameters are set incorrectly.	Check the method parameters (deposition potential, deposition time, etc.).
or poorly reproduci- ble.	Pipetting has not been car- ried out correctly.	<ul> <li>Always add the standard solution through the pipetting opening.</li> <li>Instead of manual pipetting, automatically add standard solution using an 800 Dosino.</li> <li>Pipetting standard solutions has to be carried out by the same person or with the same instrument and the same pipette</li> <li>Use the pipetting unit correctly.</li> <li>If necessary, have the pipettes calibrated (GLP).</li> </ul>
	Organic constituents inter- fere with analysis.	Carry out UV digestion or equivalent sample preparation.
	The standard solutions are too old.	Replace the standard solutions.
	The additions are outside the linear working range of calibration.	<ul> <li>Decrease the volume or concentration of the standard solution. While doing so, it is important to note that each addition should increase the sample signal by 50– 100%.</li> <li>Shorten the deposition time in the voltam- metry command under <b>Potentiostatic</b> <b>pretreatment</b>.</li> <li>Carry out the determination using polarog- raphy at the DME instead of stripping vol- tammetry.</li> <li>Use less of the sample amount or dilute the sample</li> </ul>

Problem	Cause	Remedy
		<b>INFO</b> : The linear working range of calibration depends heavily on the respective application. As a rule of thumb, the following linear working ranges can be assumed:
		<ul> <li>Polarography: 2-3 orders of magnitude</li> <li>ASV: 1-2 orders of magnitude</li> <li>AdSV, CSV: 1 order of magnitude</li> </ul>
	The stirrer does not work correctly.	<ul> <li>Ensure that the measuring head is attached correctly and locked in place.</li> <li>Ensure that the drive shaft is intact and fastened correctly. Replace the drive shaft if necessary.</li> <li>Ensure that the stirrer can turn easily. If necessary, service or replace the stirrer.</li> </ul>
	The measurement curves are generally not easily reproducible.	See "Unstable or abnormally low base current" and "Curves have a large amount of noise".
The measuring head vibrates when oper- ating the stirrer.	The measuring head is not correctly locked in place on the instrument. This can hinder the connection between the drive disk on the instrument and the coupling on the measuring head.	Remove the measuring head and put it back in place until it clearly clicks in place.
	Only MME and SPE mea- suring head: The flexible shaft was not installed cor- rectly.	Install the flexible shaft according to ( <i>see chapter 3.2.1, page 32</i> ) or ( <i>see chapter 3.4.1, page 66</i> ). Make sure that the course of the flexible shaft is as straight as possible.
The peak is in the uppermost mA range (all electrode types).	The concentration of the ion to be determined is too high.	Reduce the sample volume, run the analysis again.
The peak is in the uppermost mA range (with HMDE).	The deposition time (in the viva method, in the vol- tammetry command under <b>Potentiostatic pretreat-</b> <b>ment</b> ) is too long.	Reduce the deposition time.
	The HMDE is not suitable for the specific use case.	Instead of the HMDE, use a SMDE or DME.

Problem	Cause	Remedy
The peak is shifted.	The pH of the solution is set incorrectly.	Check and set the pH of the solution.
	The electrolyte composition is incorrect.	Check the electrolyte composition and correct it if necessary.
	The incorrect peak has been evaluated.	<ul> <li>Carry out spiking with a standard solution to check whether the correct peak has been evaluated.</li> <li>Enter the characteristic potential in viva again and recalculate the results.</li> </ul>
	Organic constituents inter- fere with analysis.	Carry out UV digestion or equivalent sample preparation.
	The reference electrode is connected incorrectly, filled incorrectly or defective.	Check the reference electrode ( <i>see chapter 3.2.2.2, page 42</i> ).
	The electrolyte solution is too old.	Produce new electrolyte solution. Organic additives may only last 1 day or less.
The peaks of the standard addition have shifted (with HMDE).	When using the HMDE, potential shifts of more than 20–30 mV are often normal and acceptable; this is especially true for adsorptive stripping vol- tammetry.	<ul> <li>No actions are necessary if the peaks are detected.</li> <li>In the event of problems with automated peak detection, increase the value for the <b>Tolerance</b> of the characteristic potential (can be found in the <b>viva</b> method under <b>Evaluation - Substance</b>).</li> </ul>
The peaks of the standard addition	The standard solution is too heavily acidulated.	Reduce the pH value of the standard solution.
have shifted. The wrong peak is evalu- ated in the sample	The buffer capacity of the electrolyte is not sufficient.	Increase the electrolyte volume.
(all of the electrode types).	The electrolyte solution is too old.	Produce new electrolyte solution. Organic additives may only last 1 day or less.
There is liquid close to the connection socket for the SPE.	The nut has not been tight- ened enough.	Dry the electrode holder with a cloth. Tighten the nut until the silicone seal seals the SPE completely.
	<i>The silicone seal is defec- tive.</i>	Silicone seals are consumables. The sharp edges of SPEs can damage silicone seals. Metrohm recommends replacing the silicone seal after using 25 electrodes.

Problem	Cause	Remedy
		Dry the electrode holder with a cloth.
	The supporting ring is deformed.	The supporting ring deforms with time. A deformed supporting ring does not distribute the pressure evenly. Replace the supporting ring as soon as it is deformed.
		Dry the electrode holder with a cloth.
Unstable or abnor- mally low base cur- rent in the pA range (all electrode types).	The concentration of the electrolyte and the pH of the solution are insuffi- cient.	Check the concentration of the electrolyte and the pH of the solution.
	The measuring parameters are not correct.	Check the parameters on the <b>Pretreatment</b> and <b>Sweep</b> tabs of the voltammetry com- mand in <b>viva</b> .
	The ion concentration in the solution is too high.	Dilute the electrolyte.
	The ion concentration in the solution is too low.	Use more concentrated electrolyte.
	Oxygen is interfering with the measurement. The sample has not been purged sufficiently.	We recommend purging with nitrogen for at least 5 min or approximately 10 min for alka- line solutions.
	The reference electrode is not filled sufficiently (inside and outside).	Top up the reference electrode ( <i>see chapter 3.2.2.2, page 42</i> ).
	The reference electrode's diaphragm is partially or completely blocked (e.g. from precipitation caused by incompatible solutions like KCl as a bridge electrolyte and HClO <sub>4</sub> as an electrolyte in the measuring solution).	Ensure the compatibility of the chemistry. Replace the bridge electrolyte if necessary (such as using a NaCl or KNO <sub>3</sub> solution in place of KCl).
	The electrolyte solution is too old.	Produce new electrolyte solution. Organic additives may only last 1 day or less.

Problem	Cause	Remedy
Unstable or abnormally low base current in the pA range (with MME pro).	The electrode drips contin- uously.	<ul> <li>Check the MME pro.</li> <li>Adjust the needle and capillary.</li> <li>If necessary, replace the capillary or needle (see the <i>Multi-Mode Electrode pro docu- ment (8.110.8018XX)</i>).</li> </ul>
	The tapping mechanism on the VA stand base is not functioning correctly.	<ul> <li>Check the positioning of the capillary in the MME pro. The capillary has to be centered in the retaining screw.</li> <li>The retaining screw is too loose or too tight: Use the wrench for the MME (6.1247.220) to tighten the retaining screw to the right torque.</li> </ul>
	The gas pressure is set incorrectly.	Set the gas pressure (1–1.2 bar; 14.5–17.4 psi; 0.1–0.12 MPa).
	The gas pressure is unsta- ble.	The gas pressure must hardly change during the measurement. This can only be ensured if a high-quality pressure relief valve is used and the tubing connection between the pressure relief and the instrument is not extended.
	The concentration to be determined is substantially higher than assumed.	<ul> <li>Reduce the deposition time.</li> <li>If the application permits, change the electrode type (e.g. HMDE to SMDE or DME).</li> <li>Reduce the sample volume.</li> </ul>
	Dripping stops prematurely (only for HMDE).	<ul> <li>The stirrer hits the capillary: Check the position of the MME pro and the capillary.</li> <li>Nitrogen bubbles rise up when the Hg drips: Check the position of the gas inlet tubing and correct if necessary.</li> <li>Capillary defective: Replace the capillary (see the <i>Multi-Mode Electrode pro document (8.110.8018XX)</i>).</li> </ul>
	The drop size is not repro- ducible.	• The capillary and/or needle is not adjusted correctly: Adjust the needle and capillary (see the <i>Multi-Mode Electrode pro document (8.110.8018XX)</i> ).

Problem	Cause	Remedy
		<ul> <li>The capillary and/or needle is defective:</li> </ul>
		Replace the capillary and needle. If neces-
		sary, clean the MME pro (see the Multi-
		Mode Electrode pro document
		(8.110.8018XX).
		<ul> <li>The tapping mechanism is not functioning correctly: Check the tapping of the drops.</li> </ul>

Problem	Cause	Remedy
Conditioning takes a long time.	The reference electrode has not yet reached equili- brium.	After maintenance of the reference electrode, wait for at least 20 min before starting a mea- surement.
	The working electrode is contaminated.	Condition the working electrode in pure VMS.
	The electrode tip of the working electrode has been in use for a long period.	<ul> <li>Immerse the electrode tip in c(NaOH) = 0.5 mol/L for approx. 10 min and then thoroughly rinse with distilled water.</li> <li>Replace the electrode tip if necessary.</li> </ul>
	VMS is contaminated.	Check the purity of the reagents (only reagents of sufficient purity, i.e. > 99%, should be used).
	The water quality is poor.	Use distilled water, type-II grade (ASTM D1193-91) or higher.
	The measuring solution has been contaminated as a result of diffusion.	No infeed tubing other than the 4-way micro dosing tip (6.1824.000) may be immersed in the measuring solution.
The electrode tip of the working elec- trode and/or the auxiliary electrode shows copper deposits.	An incorrect potential has been applied because the reference electrode is defective.	<ul> <li>Check the reference electrode; perform maintenance procedure in accordance with the electrode leaflet, if necessary.</li> <li>Dip the electrode tip of the working electrode and/or the auxiliary electrode into concentrated nitric acid for 1 to 2 seconds and then thoroughly rinse with distilled water.</li> </ul>

### 7.1.3 884 Professional VA for CVS

Problem	Cause	Remedy
	An incorrect potential was applied because the refer- ence electrode is not con- nected.	<ul> <li>Check the electrode connectors.</li> <li>Dip the electrode tip of the working electrode and/or the auxiliary electrode into concentrated nitric acid for 1 to 2 seconds and then thoroughly rinse with distilled water.</li> </ul>
	An incorrect potential was applied because the mea- suring vessel contains too little measuring solution.	<ul> <li>All 3 electrodes must be immersed in the measuring solution.</li> <li>Dip the electrode tip of the working electrode and/or the auxiliary electrode into concentrated nitric acid for 1 to 2 seconds and then thoroughly rinse with distilled water.</li> </ul>
The measurement curves are noisy.	Contact problem at the driving axle (6.1204.510 or 6.1204.520).	<ul><li>Remove the abrasion residue from the driving axle.</li><li>Replace the driving axle.</li></ul>
The measuring sig- nal for Cu VMS fluc- tuates.	<i>The ambient and/or solu- tion temperature is not constant.</i>	Keep the ambient and solution temperature stable during measurements (±4 °C).
	The reference potential drifts off.	Perform the maintenance procedure for the reference electrode in accordance with the electrode leaflet. However, for CVS analyses, replace the reference electrolyte every other day and the bridge electrolyte every day. After replacing the reference electrolyte, wait at least 20 minutes (preferably 1–2 h) until the potential has reached equilibrium.
	The measuring vessel and the electrodes have been contaminated with organic additives.	Thoroughly rinse the measuring vessel and the electrodes and use fresh VMS.
The signal does not decrease in spite of suppressor addition.	No chloride is contained in the Cu VMS.	Check the preparation of the Cu VMS and modify it if required.
	The wrong dosing unit is being used for the addition of standard solution or the dosing unit contains the wrong solution.	Check the automatic solution addition.

Problem	Cause	Remedy
	The concentration of the	Add a larger volume or use the RC calibration
	standard solution or the	technique instead of DT.
	sample is too low.	

## 7.1.4 Peripheral devices

Problem	Cause	Remedy
The 800 Dosino can- not be actuated by the 884 Professional VA.	The connection between the 800 Dosino and the 884 Professional VA is either interrupted or an error has occurred on the 800 Dosino.	<ul> <li>Check the cable connections.</li> <li>Disconnect the 884 Professional VA from the power grid and connect it again.</li> <li>Check the dosing and filling rate.</li> <li>Contact Metrohm Service if necessary.</li> </ul>
The data of the dos- ing unit cannot be read.	The data chip of the dosing unit is mechanically dam- aged or impaired by chem- icals.	<ul> <li>Remove the dosing drive and attach it again.</li> <li>Clean the data chip and the contact surfaces.</li> <li>Have the data chip replaced by Metrohm Service.</li> </ul>
The dosing unit is blocked and/or leak- ing.	Crystals have formed (in the dosing cylinder, on the valve disk or in the capil- lary).	<ul> <li>Check the flow path.</li> <li>Rinse the dosing unit and the connected tubing and capillaries (<b>Prepare</b> function) when the measuring system is not in use.</li> <li>Clean the dosing unit at least every 2 weeks.</li> </ul>
The dosing unit is recognized either not at all or incor- rectly.	The dosing drive was not attached correctly.	<ul> <li>Remove the dosing drive and attach it again.</li> <li>Check whether the dosing drive is correctly seated.</li> <li>Disconnect the 884 Professional VA from the power grid and connect it again.</li> <li>Contact Metrohm Service if necessary.</li> </ul>
The membrane pumps of the 843 Pump Station do not	The cables are not or not correctly connected.	Connect the cables as described in <i>Chapter</i> 3.6.5, page 102.
operate at full pump capacity.	The tubing connections are leaking.	Check the tubing connections and tighten, if necessary.
	The rinse and/or waste canister are sealed air- tight.	Loosen the caps on the canisters a little or remove them.

Problem	Cause	Remedy
The pump time of the peristaltic pump increases.	The pump tubing of the peristaltic pump has aged or is defective.	Replace the pump tubing.
The sample is not completely transfer- red from the Sample Processor to the measuring vessel via the peristaltic pump.	The sample needle on the Sample Processor is posi- tioned more than 0.5 mm from the bottom of the sample vial.	Position the sample needle as described in .
	The contact pressure set for the tubing cartridge is insufficient.	Set the contact pressure of the tubing car- tridge in accordance with the information in the Sample Processor manual.
	The selected pump times are too short.	Prolong the pump times.
	The pump tubing has been in use too long.	Replace the pump tubing.

## 7.2 Detailed troubleshooting for VA trace analysis

### Oxygen in the measuring solution

Oxygen can be reduced electrochemically and provides 2 levels in the voltammogram where one is marked by the occurrence of a distinct maximum. Oxygen reduction can cause interference for 2 reasons:

- The signals for the substances to be determined are covered by the oxygen levels. This is primarily noticeable in trace analysis because the oxygen in air-saturated solutions is present in relatively high concentration (approx. 8 mg/L at room temperature).
- The hydrogen peroxide formed during the 1st step in the oxygen reduction can lead to other reactions with certain substances.

For this reason, the oxygen has to be removed from the measuring solution through saturation with an inert gas (usually nitrogen) before polarographic analysis. For the inert gas throughput of approximately 20 L/h set on the 884 Professional VA ex works, a purge time of 3-5 minutes is generally sufficient for accomplishing this.

Compare the curves ( $c(KNO_3) = 0.1 \text{ mol/L}$ ) before and after purging:

### Before purging (still contains oxygen in the solution):


After purging (oxygen removed from the solution):



### Unsuitable bridge electrolyte solution in the reference electrode

When choosing the bridge electrolyte solution in the reference electrode, potential complications with the substances present in the measuring solution have to be taken into consideration.

For the frequently used bridge electrolyte solution **c(KCl) = 3 mol/L**, for instance, the following faults can occur:

Precipitation of KClO<sub>4</sub> in the ceramic diaphragm for base electrolytes containing HClO<sub>4</sub>

In the event of partial blockage, unexplainable side peaks can occur. To avoid this kind of precipitation, a potassium-free bridge electrolyte solution (such as c(NaCl) = 3 mol/L) has to be used with measuring solutions containing  $HClO_4$ .

• Introduction of chloride due to KCI discharge from the reference electrode

The discharge of bridge electrolyte from the ceramic diaphragm of the 6.1245.010 electrolyte vessel (part of the reference electrode) amounts to 2-5  $\mu$ L/h. The chloride that ends up flowing into the measuring solution this way can cause interference during Cu determinations. As a countermeasure, we recommend the use of chloride-free bridge electrolyte solutions (e.g. c(KNO<sub>3</sub>) = 1 mol/L).

### **Overloading the working electrode**

Deposition of species on the polarizable electrodes leads to overload phenomena like non-linear spiking or splitting into multiple peaks under unfavorable conditions (large concentrations and/or long deposition times). These phenomena are caused by saturation and various deposit forms.

A shorter deposition time usually solves the problem. As a rule of thumb, generally only solutions with a mass concentration  $\beta < 0.5 \text{ mg/L}$  (= 0.5 ppm) are to have any deposition at all. In various instances, work can even begin without deposition starting from concentrations  $\beta > 100 \text{ µg/L}$  (e.g. DP voltammetry on a HMDE or even on a DME).

The 2 following examples show the effects of a deposition time that is too long:

 Nickel and cobalt determination in the trace range using adsorptive stripping voltammetry (with dimethylglyoxime complexes)

Extending the deposition time from 30 s to 120 s (at measuring parameters that remain otherwise identical) leads to non-linear spiking and, in the case of the nickel, also lead to shifts in the peak maximum:



### Interference on the HMDE due to the formation of gas

The formation of gases on the HMDE during the deposition time can lead to the drop falling or a loss of contact in the Hg capillary. The following example shows such a case:

### Determination of zinc in deionized water

If the zinc sample is acidulated with  $HCIO_4$  to a pH = 2, hydrogen also forms at the voltage selected for deposition. In this example, this leads to the Hg drops falling off prematurely at the 2nd spiking, meaning it is not possible to carry out an evaluation. If an acetate buffer (pH = 4.64) is used, none of these difficulties occur. This requires, however, that chemicals of the highest purity level are used to maintain the lowest possible zinc blank value (we recommend producing the acetate buffer from ultrapure ammonia and ultrapure acetic acid).



### Complexing

Substances that can be determined using polarography can be present in a variety of complexed forms depending on the composition of the measuring solution. Because complexing is always associated with a shift in the half-wave potential and limiting current, difficulties can arise during peak evaluation. These difficulties have to be resolved through corresponding changes to the composition of the base electrolyte.

If this is not possible, the interfering complexing agent has to be removed from the measuring solution or masked using suitable substances; a

change in base electrolyte pH often helps. Another technique that is often used is to add a strong complexing ligand (such as EDTA) to completely put the substance to be determined in a defined form. The latter option is also used in the following example:

Copper determination in solutions containing chloride

Copper can be present as both a  $CuCl_4^{2-}$  complex as well as a  $CuCl_2^{-}$  complex in solutions containing chloride. The two associated current peaks are close together. In the worst case, determination of the copper is not even possible. The difficulties vanish after adding EDTA as a complexing agent, because all of the copper is then present as a Cu-EDTA complex. (Increasing the chloride concentration [such as by add-ing 1 mL c(KCl) = 1.5 mol/L of ultrahigh purity per 10 mL of measuring solution] would also result in a clearly defined current peak for  $CuCl_2^{-}$ .)



Peak on heavily distorted baseline

If peaks are on a heavily distorted baseline, the first step should be to use chemical or measuring-related countermeasures to eliminate the impairment to peak evaluation from the heavily distorted baseline. These kinds of measures include using longer purge times (*see "Oxygen in the measuring solution", page 168*), changing the pH value, changing the base electrolyte concentration, changing or swapping out the base electrolyte, using complexing agents (*see "Complexing", page 172*), applying longer deposition times or changing the measuring technique.

If the distortion to the baseline cannot be resolved or is only partially fixed by the measures listed above, the 884 Professional VA provides the option of evaluating the distorted baseline by choosing **Polynomial** or **Exponential** for the baseline type.

### **Peak overlap**

If the peak overlap has reached a critical level at which the calculated peak height or peak area is being distorted by the neighboring peak, we recommend taking the overlap into consideration by changing the calculation for the baseline. To change the calculation, select the **Horizontal start** or **Horizontal end** baseline type.

If the overlap is too large, the peak can no longer be evaluated. In this case, you have to attempt to use chemical or measuring-related countermeasures to better separate such peaks. Potential measures include changing the pH value, changing the base electrolyte concentration, changing or swapping out the base electrolyte, using complexing agents *(see "Complexing", page 172)*, applying longer deposition times or changing the measuring technique.

### Determination of lead and thallium

For a base electrolyte with a pH = 1, the Pb and Tl peaks have heavy overlap. Changing the pH to a pH = 13 separates both peaks. (The separation of lead and thallium can also be implemented by subsequent electrolysis or in an acetate buffer with EDTA.)



Measuring Solution: Spiking: Electrode

0.5 mg/L Pb; 1 mg/L Tl each with 10  $\mu g$  Pb and 10  $\mu g$  Tl SMDE

### Calibration with chemically non-isoform standards

For all potential calibration techniques, ensure that the standards used for calibration are chemically isoform with the substances to be determined. In other words, the standard substances have to have the same oxidation state (e.g. for AsIII/V or CrIII/VI) or complexing form (e.g. for As, Cr, Se) as the substances already present in the measuring solution. If this is not the case, calibrations may return entirely incorrect results due to different peak voltages and sensitivities.

### **Results are not reproducible**

Voltammetric measurements (including CVS and CPVS) are heavily temperature-dependent. This is can have a particularly negative and noticeable effect during determinations with a separate calibration curve, such as external calibration, DT or RC.

Observe the following points so that measuring results are reproducible:

- Avoid severe temperature fluctuations in the lab.
- Do not set up the instrument directly under the outlet of an air-conditioning system.
- If necessary, use a thermostable measuring cell with a connection to a thermostat/cryostat.

### 7.3 General rules for VA trace analysis

### **Chemicals and equipment**

- The purity of reagents plays a key role in determining results. Only ultrapure chemicals should be used to determine low concentrations.
- The specifications of the reagents in use should be checked against the analytes to be determined. For concentrations in the mg/kg range, significant blank values are to be expected in trace analysis in the ng/L and lower µg/L range.
- Even the ultrapure water used to produce electrolytes, buffers, standards and other solutions is to be treated as a reagent. It is important to strive for the highest quality here for this reason. The ultrapure water in use should meet the specifications from ASTM D1193 for Reagent Water Type 1 (resistivity > 18 M $\Omega$ ·cm (25 °C); TOC < 50 µg/L).
- Measuring vessels, electrodes and all other parts that come in contact with the sample solution, as well as containers (bottles, graduated flasks, etc.) used to store solutions (buffers, standards, etc.) have to be clean and free of interfering substances.

### Electrolytes

- The pH value plays an important role in many determinations (e.g. it should be approximately 4.5 for Zn). Acetate, ammonium acetate and PIPES buffer solutions are frequently used. Refer to the Application Bulletins for more information.
- The electrolyte has to be sufficiently conductive and concentrated.
- The purity of the electrolyte and the cleanliness of the reagent bottles play an important role.
- Electrolytes, especially those with organic additives (buffer substance (e.g. PIPES, HEPS, etc.), complexing agents (e.g. Dimethylglyoxime (DMG)), have very limited shelf lives. The solution may have to be freshly applied every day.

### **Standard solutions**

- Metal standard solutions should be acidulated (approx. pH = 1-2) and stored in plastic bottles. Stabilizing the standard through acidulation is often not possible if the oxidation state of the metal is important to its use or in the case of anions or organic compounds. In cases such as this, the standard always has to be produced fresh.
- Diluted standard solutions (ppb range) are very unstable and have to be prepared fresh. They have to be acidulated accordingly. Ensuring the cleanliness of all of the vessels used during production is very important.
- The concentration of the standard solutions has to be tuned so that a volume between 20 and 500 µL is dosed per standard addition in each case.
- We recommend the standard addition calibration method to eliminate any influence of the sample matrix on the result. The peak height after the last spiking should be 2-5 times as high as the sample peak.
- 1000 ppm solutions are commercially available for many elements and are frequently used as stock solutions. They are stable over prolonged periods. Dilutions are added to diluted acid.

### Samples

- The sample amount depends on the substance concentration to be determined and the linear working range of the method.
- If you are familiar with the sample, you can better judge the analysis (organic constituents?).
- In the event of a contaminated sample or suspicion of such, digestion has to be carried out (refer to the "Sample preparation techniques in voltammetric trace analysis" Metrohm monograph).
- Many errors are committed during sampling and storage of samples. You should be careful and critical at all times.
- The sample is to be easily soluble in and miscible with the electrolyte.

### **Blank values and contamination**

If the results are too high, you should check the following points:

- Were the dilutions made correctly?
- Have the risks of contamination been eliminated?
- The risk of contamination is very high at low concentrations: Measuring vessels should be conditioned with a diluted HNO<sub>3</sub> solution.
- Are the chemicals pure enough? At low concentrations, reagents declared specifically for trace analysis should be used (e.g. Merck Suprapur(R), Sigma-Aldrich TraceSELECT(R) or similar quality from another manufacturer).
- Very high concentrations were measured in the previous analysis: Carefully clean and condition the electrodes and measuring vessel (memory effects).
- Has the standard addition been carried out correctly? Has the volume been set correctly on the pipetting unit?
- Are the sample signal and the additions in the linear working range of the method?

If the results **are too low**, you should check the following points:

- Does the sample contain organic material that has to be removed before the determination, such as by using UV digestion?
- Is the concentration of the standard in use correct and are the volumes in use in the method specified correctly?
- Is the correct sample amount specified in the determination?
- Is the concentration too high? HMDE overloaded, using a DME/SMDE?
- Is the buffer in good condition? If necessary, make a new one.
- Is the spiking ratio too small?
- Is the spiking ratio too high?

### Selecting the VA measuring mode

The following points should be taken into account when choosing the VA measuring mode:

- DP (differential pulse measuring mode) should always be selected first. It is the most comprehensive and most frequently used voltammetric measuring mode and is equally well-suited for reversible and irreversible systems. It offers high sensitivity down to 10<sup>-8</sup> mol/L.
- SQW (square-wave measuring mode) is especially well-suited for reversible electrode processes and kinetic experiments. It is specifically used for sensitive, inverse voltammetric determinations on a HMDE or solid-state electrode such as a scTRACE Gold.

## 8 Appendix

### 8.1 **Tubing lengths in the measuring head arm**

The following list provides information about the lengths and diameters of the pieces of tubing used in the measuring head arm between the connector plate and the tubing connector. You will need this information for setting the parameters of the dosing units in **viva** (e.g. for the function **Prepare**).

Connection between con- nector plate (1-4)	and tubing connector of the measuring head arm (see chapter 2.3.3, page 17)	Article number	Tubing length in mm	Diameter in mm
Connector <b>1</b>	Connector <b>1</b>	6.1805.550	250	2.00
Connector <b>2</b>	Connector <b>2</b>	6.1805.550	250	2.00
Connector <b>3</b>	Connector <b>3</b>	6.1805.550	250	2.00
Connector <b>SMP</b>	Connector <b>SMP</b>	6.1831.020	220	0.75
Connector	Connector	6.1805.050 and	210 and	2.00
WASTE	WASTE	6.1805.540	80	
Connector <b>OUT</b>	Connector <b>OUT</b>	6.1805.550	250	2.00

### 8.2 "Status" LED – Various instrument statuses

The "Status" LED on the front of the instrument indicates 3 different statuses:

The instrument is operating.

Continuously on

Blinking regularly

Blinking pattern "LED on a long time - off a short time - on a long time - off a short time..."

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A standby potential is being applied to the electrodes. Do not remove the measuring head or the electrode cables in this instrument status.

The instrument is ready for operation.

## **9** Technical specifications

## 9.1 Operating modes

Potentiostat	yes
Galvanostat	yes
Temperature mea-	yes (Pt1000)
surement	

## 9.2 Potentiostat

Maximum output voltage (AE)	
(maximum potential applied)	±25 V
Maximum output current (AE)	
(maximum cur- rent applied)	±224 mA
Sweep potential range	±5.0 V
Current measuring ranges	200 pA–224 mA (Not all ranges are available in all measuring modes.)
Bandwidth	> 800 kHz
Rise time / fall time	300 ns
Noise	5 pA typical

## 9.3 Galvanostat

Potential measur- 5 mV–5 V ing ranges

## 9.4 Temperature measurement

Measuring range (Pt1000)	0 to +100 °C
Accuracy (Pt1000)	±0.5 °C

## 9.5 Measuring input

Measuring interval	
Sampling rate	100 kHz
Input impedance (RE)	$>$ 15 G $\Omega$ // < 8 pF
Input bias current (RE) at 25 °C	< 2 pA
Bandwidth of the electrometer	25 MHz

### 9.6 Accuracy

Current applied	$\pm$ (0.2% of the current +0.2% of the current measuring range)
Current measured	±(0.2% of the current +0.2% of the current measuring range)
Potential applied	$\pm$ (0.2% of the potential $\pm$ 1 mV)
Potential mea- sured	$\pm$ (0.2% of the potential $\pm$ 1 mV)

## 9.7 Resolution

Potential applied	15 μV
Potential mea- sured	150 μV
Current applied	0.0031% of the current measuring range
Current measured	0.0031% of the current measuring range
Current measured in the smallest current measuring range (200 pA)	6 fA

## 9.8 Calibrator

Reference voltage	2.5 V
R17.8	17.8 Ω
R178	178 Ω
R1.9k	1900 Ω
R18k	18000 Ω
R180k	180000 Ω
R1.8M	1800000 Ω
R18M	18000000 Ω
Validity of the cer- tification	Two years after activation

### 9.9 Stirrer

Stirring rate	100–3,000 min <sup>-1</sup>
Stability	±2%

## 9.10 Hardware

A/D converter	16-bit
D/A converter	16-bit, 4 channels
Integrator	Digital
Interface	USB
MSB	4

## 9.11 Power connection

Supply voltage	100–240 V
Frequency	50–60 Hz
Power consump-	45 W
tion	

## 9.12 Ambient temperature

Nominal function range	0 to +45 °C (at a maximum of 85% relative humidity)
Automatic interior temperature moni- toring	> 70 °C
Storage	–40 to +70 °C
Transport	–40 to +70 °C

## 9.13 Reference conditions

Ambient tempera- ture	+25 °C (±3 °C)
Relative humidity	$\leq$ 60%
Operating temper- ature status	Instrument in operation at least 30 min

## 9.14 Housing data

Dimensions with-	
out accessories	
Width	188 mm
Height	294 mm
Depth	406 mm
Weight	7,400 g
Dimensions with measuring head and drip pan	
Width	188 mm
Height	322 mm
Depth	452 mm
Material of hous- ing	
Cover	PP with flame retardation for fire class UL94 V-0
Base	Steel sheet, coated

## **10 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. **2.884.XX10**) 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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