IC Professional Detector



IC Amperometric Detector

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IC Professional Detector IC Amperometric Detector

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Manual

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This documentation has been prepared with great care. However, errors can never be entirely ruled out. Please send comments regarding possible errors to the address above.

Table of contents

Table of contents

1	Introduction	on	1
	1.1	Instrument description	1
	1.2	Intended use	2
	1.3 1.3.1 1.3.2 1.3.3 1.3.4 1.3.5	Safety instructions General notes on safety Electrical safety Working with liquids Flammable solvents and chemicals Recycling and disposal	2 2 3
	1.4 1.4.1 1.4.2	About the documentation Content and scope Symbols and conventions	4
2	Overview o	of the instrument	6
	2.1	Front	6
	2.2	Rear	7
3	Installation	1	8
	3.1 3.1.1 3.1.2 3.1.3	Setting up the instrument Packaging Check Location	8 8
	3.2	Inserting the amperometric detector	8
	3.3 3.3.1 3.3.2 3.3.3 3.3.4 3.3.5 3.3.6	Putting the amperometric detector into operation Instrument test with dummy cell Testing the leak sensor Testing the preheating capillary Testing the detector outlet capillary Testing the measuring cell Deaerating the measuring cell	11 13 14 15
	3.4	Connecting the electrode cables	19
	3.5	Attaching the front cover	20
4	Operation	and maintenance	21
	4.1	Operation	21
	4.2	Care	21
	4.3	Maintenance by Metrohm Service	22
	4.4	Maintenance	22

Table of contents

	4.4.2	Preheating capillary maintenance	22
	4.5	Shutting down	23
5	Troublesho	oting	24
	5.1	Problems with the hardware	24
	5.2	Problems with the baseline	24
	5.3	General remarks regarding sensitivity fluctuations	27
	5.4	Problems with sensitivity	27
	5.5	Problems with the pressure	28
	5.6	Problems with the measuring signal	28
	5.7	Problems with the chromatogram	29
	5.8	Other problems	30
	5.9	Systematic error diagnostics	31
6	Technical s	pecifications	33
	6.1	Amperometric detector	33
7	Accessories	5	35
	Index		36

Table of figures

Table of figures

Figure 1	Front	6
Figure 2	Rear	7
Figure 3	Inserting the detector	9

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1 Introduction

1 Introduction

1.1 Instrument description

The **IC Amperometric Detector** is an intelligent amperometric detector that is used in the detector chamber of the instruments of the 850 Professional IC, the 881 Compact IC pro and the 882 Compact IC plus line of instruments.

With the IC Amperometric Detector, electroactive substances can be determined in the mobile phase of an IC system. Amperometric methods are used for the determination which combine an outstanding sensitivity with a high degree of selectivity.

The installed potentiostat generates the voltages for the direct current amperometry (DC), for the pulse amperometry (PAD) and the flexible integrated pulse amperometry (flexIPAD) as well as for the recording of cyclovoltammograms.

The installed preheating capillary ensures a constant eluent temperature on the cell.

On the one hand, the built-in leak sensor detects fluid that has escaped in the interior of the detector, while on the other it also responds when too much fluid has backed up in the tray on the front side of the detector. As a result of this, the leaking instrument is shut down and the ongoing determinations are canceled.

The IC Amperometric Detector can only be used together with the IC instrument. It is utilized in the detector chamber of the IC instrument.

The IC system is operated with **MagIC Net™** software. When the IC instrument is switched on, MagIC Net™ recognizes the IC Amperometric Detector automatically and checks its functional readiness. MagIC Net™ controls and monitors all of the instruments combined to form an IC system, evaluates the measured data and manages it in a database.

Additional information for the operation of MagIC Net^{TM} can be found in the online help and in the tutorial for MagIC Net^{TM} .

1.2 Intended use

1.2 Intended use

The IC Amperometric Detector is used for the amperometric detection in an IC instrument. Its function is to make precise determinations of electroactive substances in the mobile phase of an IC system or of a general liquid chromatography system.

As is the case with the associated IC instrument, the IC Amperometric Detector is also used for working with chemicals and flammable samples. The usage of the IC Amperometric Detector requires that the user have basic knowledge and experience in the handling of toxic and caustic substances for this purpose. Knowledge with respect to the application of the fire prevention measures prescribed for laboratories is also mandatory.

1.3 Safety instructions

1.3.1 General notes on safety



WARNING

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

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

1.3.2 Electrical safety



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.

1 Introduction

Protection against electrostatic charges



WARNING

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

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

1.3.3 Working with liquids



CAUTION

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

1.3.4 Flammable solvents and chemicals



WARNING

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

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

1.3.5 Recycling and disposal



This product is covered by European Directive 2012/19/EU, WEEE – Waste Electrical and Electronic Equipment.

The correct disposal of your old instrument will help to prevent negative effects on the environment and public health.

More details about the disposal of your old instrument can be obtained from your local authorities, from waste disposal companies or from your local dealer.

1.4 About the documentation

1.4 About the documentation



CAUTION

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

1.4.1 Content and scope

Content of this manual

This manual describes:

- The installation of the IC Amperometric Detector in an IC instrument and the insertion and connection of the measuring cell in the detector.
- The start-up of the IC Amperometric Detector together with the IC instrument.
- All maintenance work that can be carried out by the user.
- The technical specifications of the IC Amperometric Detector.
- Possible problems and their solutions.
- The supplied and the optional accessories.

Further information

Detailed information regarding the preparation of the measuring cell and its maintenance can be found in the manuals for the Wall-Jet cell IC equipment.

Information on the utilization, care and maintenance of the working electrodes and reference electrodes can be found in the leaflets which are enclosed with the electrodes.

1.4.2 Symbols and conventions

The following symbols and styles are used in this documentation:

(5- 12)	Cross-reference to figure legend	
	The first number refers to the figure number, the second to the instrument part in the figure.	
1	Instruction step	
Carry out these steps in the sequence shown.		

4 -----

1 Introduction

	Warning
7:	This symbol draws attention to a possible life hazard or risk of injury.
	Warning
7	This symbol draws attention to a possible hazard due to electrical current.
	Warning
<u></u>	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 a possible damage of instruments or instrument parts.
4	Note
	This symbol marks additional information and tips.

2.1 Front

2 Overview of the instrument

2.1 Front

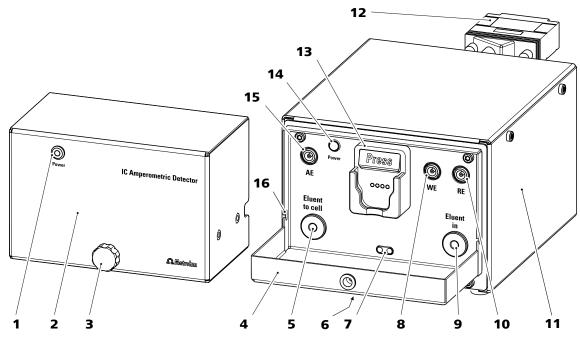


Figure 1 Front

1 Standby indicator

On front cover.

3 Knurled screw

For fastening the front cover.

5 Eluent output

Output of the preheating capillary inside the instrument, labeled with **Eluent to cell**.

7 Leak sensor

9 Eluent input

Input of the preheating capillary inside the instrument, labeled with **Eluent in**.

11 Detector housing

2 Front cover

Removable.

4 Tray

6 Drain nozzle

At the bottom of the tray, sealed with a stopper.

8 Connection socket

For the working electrode, labeled with **WE** (working electrode).

10 Connection socket

For the reference electrode, labeled with **RE** (reference electrode).

12 Connector plug

For connecting the detector to the detector connection socket of the IC instrument.

6 -----

2 Overview of the instrument

13 Measuring cell holder

For measuring cells with chip.

15 Connection socket

For the auxiliary electrode, labeled with **AE** (auxiliary electrode).

14 Standby indicator

16 Capillary feed-through

For leading capillaries into the cell chamber and out of the cell chamber.

2.2 Rear

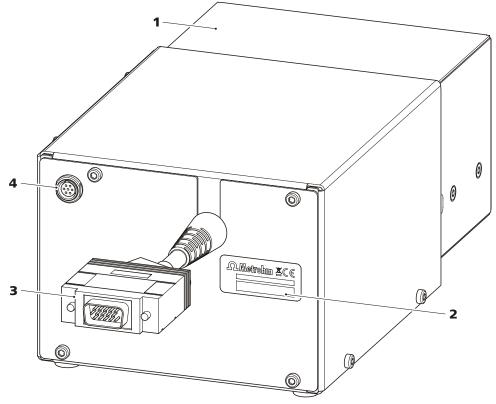


Figure 2 Rear

1 Front cover

Removable.

3 Connector plug

For connecting the detector to the detector connection socket of the IC instrument.

2 Type plate

With serial number.

4 Connection socket

For a testing instrument (for service only).

3 Installation

3.1 Setting up the instrument

3.1.1 Packaging

The IC Amperometric Detector is supplied in highly protective special packaging. Keep this packaging, as only this ensures safe transportation of the instrument.

3.1.2 Check

Immediately upon receiving the shipment, check it against the delivery note to ensure that it is complete and undamaged.

3.1.3 Location

The IC Amperometric Detector is designed for usage in the detector chamber of the IC instruments. For the location, the same conditions apply as for the IC instrument.

3.2 Inserting the amperometric detector

The IC Amperometric Detector can be used with the following IC instruments, insofar as they have an unoccupied detector connection:

- All 850 Professional ICs
- All 881 Compact IC pros
- All 882 Compact IC pluses

The IC Amperometric Detector is placed in the detector chamber of the IC instruments. The detector chamber can be opened on the rear of the instrument.

Figure 3 shows how the detector is inserted into an 850 Professional IC instrument within 6 simple steps. Inserting the detector into an 881 Compact IC pro or 882 Compact IC plus instrument is carried out analogously.

3 Installation

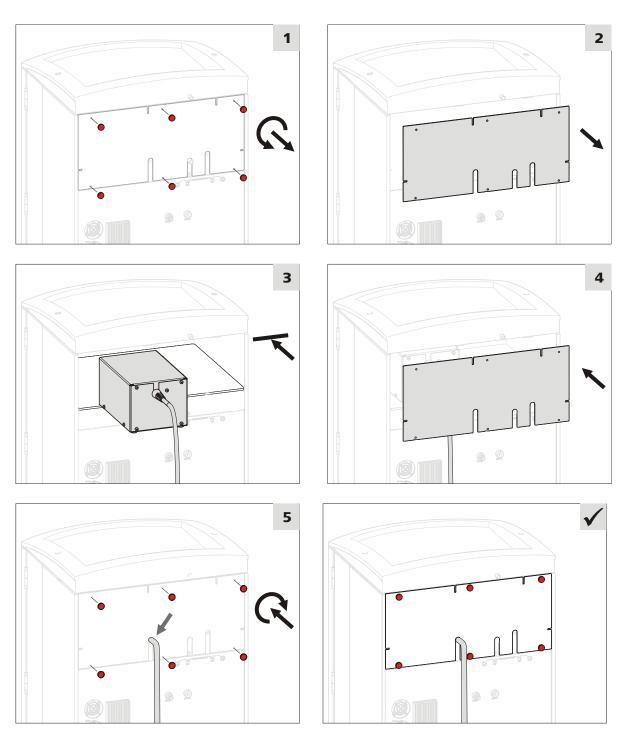


Figure 3 Inserting the detector



NOTICE

With the instruments of the 850 Professional line of instruments, up to two detectors can be placed and connected.

Placing the detector in the instrument

1 Remove the knurled screws

- Unscrew the knurled screws on the rear panel.
- Optional with 850 instruments: Remove the handle if it is still fastened to the instrument.

2 Remove the rear panel

• Remove the rear panel.

3 Position the detector

 Put the detector through the opening and position it on the support surface intended for this purpose and slide it as far as the limit stop to the front.

4 Replace the rear panel

- Insert the detector cable in a cable feed-through on the rear panel.
- Replace the rear panel.

The detector cable must hang freely from the cable feed-through.

5 Tighten the knurled screws

- Optional with 850 instruments: Reattach the handle higher up and use it as a holder for MPaks.
- Tighten the knurled screws.

Connecting the detector connection cable



CAUTION

The IC instrument **must** be switched off when the detector is being connected.

Before connecting the detector, make sure that the IC instrument is switched off.

3 Installation



NOTICE

The detector connection cable cannot be extended!

Connect the detector connection cable to the detector connection socket *Detector 1* or *Detector 2*.

3.3 Putting the amperometric detector into operation

The IC Amperometric Detector is put into operation together with the IC instrument in which it has been inserted to.

The following measures must be carried out on the IC Amperometric Detector during the initial start-up of the IC system (see the chapter *Initial start-up* in the manual for the IC instrument), even before the separation column is used:

- Testing the instrument electronics with the dummy cell (see chapter 3.3.1, page 11)
- Testing the leak sensor (see chapter 3.3.2, page 13)
- Testing the preheating capillary (see chapter 3.3.3, page 14)
- Testing the detector output capillary (see chapter 3.3.4, page 15)
- Testing the measuring cell (see chapter 3.3.5, page 16)
- Deaerating the measuring cell (see chapter 3.3.6, page 18)

3.3.1 Instrument test with dummy cell

When you are putting the IC Amperometric Detector into operation for the first time, or when problems occur which may be caused by signal recording or signal transfer, we recommend testing the electronics and the connection to the PC using the dummy cell (6.2813.040).

Proceed as follows:

Testing with the dummy cell

Prerequisites:

• In order to achieve accurate results, we recommend that the front cover be closed during the instrument test with the dummy cell. Since the space under the front cover is rather limited, we further recommend removing the measuring cell from the cell holder for the instrument test with the dummy cell.

For the instrument test you require:

• The dummy cell (6.2813.040)

• The three electrode connection cables (6.2165.000)

1 Connecting the electrode connection cables to the dummy cell

 Plug the angled plug of the working electrode connection cable (labeled WE) into the WE socket.

- Plug the angled plug of the reference electrode connection cable (labeled RE) into the RE socket.
- Plug the angled plug of the auxiliary electrode connection cable (labeled AE) into the AE socket.

2 Connecting the electrode connection cables to the detector

(unless they are already connected)

- Plug the straight plug of the working electrode connection cable (red sleeve) into the WE socket of the detector.
- Plug the straight plug of the reference electrode connection cable (black sleeve) into the RE socket of the detector.
- Plug the straight plug of the auxiliary electrode connection cable (blue sleeve) into the **AE** socket of the detector.

3 Inserting the dummy cell

- Place the dummy cell into the tray of the detector.
- Attach the front cover.



NOTICE

The metal parts of the cable plugs must not touch the front cover.

4 Adjusting settings in MagIC Net

In the **Method** program part, create a new method for the instrument test with the dummy cell.

- Add the detector and the instrument and select them.
- Select the **DC** mode.
- Set the following parameters for the DC mode:

DC potential: 0.8 V

Range: AutoDamping: off

- Add an analysis for the detector channel **Current**.
- Add the entry **Current** ► **Start data acquisition** in the Time program subwindow.
- Save the method.

3 Installation

In the Workplace program part:

- Load the method.
- In the **Watch window**, display the **Current** channel with at least three decimal places.

5 Carrying out the test

In the **Manual** program part:

- Set all components of the IC instrument to inactive, particularly the high-pressure pump(s).
- On the tab of the detector, switch on the dummy cell with **[Apply**].

After no more than one minute, the detector signal should level off at $2.667 \text{ nA} \pm 7\%$. Noise should not exceed 0.005 nA.

Switch the dummy cell off with [Cell Off].
 With the dummy cell switched off and the detector hardware still running, the signal should drop below an absolute value of 1 nA, and noise should be limited to the third decimal place.
 Exactly even signals may indicate that new detector data is not correctly transmitted.

6 Removing the dummy cell

- Pull out the electrode connection cables from the connectors AE,
 WE and RE of the dummy cell.
- Remove the dummy cell from the tray.

The dummy cell incorporates a resistor (300 M Ω) and a capacitor (100 nF) connected in parallel. If, in DC mode, a potential of 0.8 V is applied, then a current of 2.667 nA (\pm 7%) is measured in the dummy cell. The capacitor simulates a well-working measuring cell.

3.3.2 Testing the leak sensor

The leak sensor should not respond during the start-up. If the leak sensor nevertheless does respond during the start-up, you will find information for eliminating the problem in Chapter (see chapter 5, page 24).

To check whether the leak sensor is functioning, proceed as follows:

Testing the leak sensor

Hold a cloth moistened with eluent or tap water on the two contacts of the leak sensor (1-7).

The leak sensor of the detector responds.

If the leak sensor does not respond, please request Metrohm Service.

3.3.3 Testing the preheating capillary

The amperometric detector is equipped with a preheating capillary in its interior to ensure that the eluent flows through the measuring cell at a constant temperature. The preheating capillary need not, however, always be connected. If the ambient conditions are optimal, then the measuring results can be sufficiently accurate, even without the use of the preheating capillary.



CAUTION

The preheating capillary may not be used when working with highly flammable liquids.

The preheating capillary must be free of both leaks and blockages.

To check whether the preheating capillary is free of both leaks and blockages, proceed as follows:

Testing the preheating capillary

1 Connecting the detector inlet capillary

Use a pressure screw (6.2744.014) to fasten the detector inlet capillary to the **Eluent in** connector on the detector.

2 Adjusting the settings in MagIC Net

- In the program part **Manual** of MagIC Net, set the maximum pressure of the high-pressure pump to 5 MPa.
- Set the flow rate to 0.1 mL/min.
- Start the high-pressure pump.

3 Observe the Eluent to cell connector

After a while, liquid must emerge from the **Eluent to cell** connector (wipe up fluid with paper towel).

If no liquid emerges at the **Eluent to cell** connector, then the preheating capillary is likely to be blocked. To eliminate the problem, *see Chapter Preheating capillary maintenance*, page 22.

4 Observe the pump pressure

Observe the pump pressure display in the program part **Manual** of MagIC Net.

A constant pressure should establish itself after a while.

3 Installation

3.3.4 Testing the detector outlet capillary

The detector outlet capillary must be of a certain length in order to be able to generate sufficient backpressure. The required length is dependent on the flow that has been set. *The table 1* shows the recommended lengths, as determined by the set flow rate.

Table 1 Recommended lengths for the detector outlet capillary

Flow rate	Capillary length (□0.25 mm)
2.0 mL/min	0.5–1.5 m
0.5-1.0 mL/min	1.0-2.5 m
0.25 mL/min	3 m

To check whether the detector outlet capillary is free of blockages, proceed as follows:

Testing the detector outlet capillary

Prerequisites:

- The detector inlet capillary is connected to the **Eluent in** connector.
- The high-pressure pump runs with a flow rate of 0.1 mL/min.

1 Connecting the detector outlet capillary

Use a pressure screw (6.2744.014) to fasten the detector outlet capillary to the **Eluent to cell** connector.

2 Adjusting the settings in MagIC Net

In the program part **Manual** of MagIC Net, increase the flow rate to 1.0 mL/min and wait until the pressure has stabilized.

3 Observe the end of the detector outlet capillary

After a while, liquid must emerge from the end of the detector outlet capillary.

If no liquid emerges at the end of the detector outlet capillary, then the detector outlet capillary is blocked and must either be cut back once again or replaced.

4 Loosening the detector outlet capillary

Loosen the detector outlet capillary from the **Eluent to cell** connector. Wipe up emerging liquid with a cloth.

5 Observe the pump pressure

Observe the pump pressure display in the program part **Manual** of MagIC Net.

The drop in pressure should range from 0.1 MPa to a maximum of 0.3 MPa.

If the pressure differential is greater, then the detector outlet capillary must be cut back once again or replaced.

6 Finish the test

- In the program part **Manual** of MagIC Net, stop the high-pressure pump.
- Remove the detector outlet capillary from the Eluent to cell connector.

3.3.5 Testing the measuring cell

To test the measuring cell, proceed as follows:

Testing the measuring cell

Prerequisites:

- The measuring cell is completely assembled (see measuring cell manual).
- The working electrode and the reference electrode are inserted (see measuring cell manual).

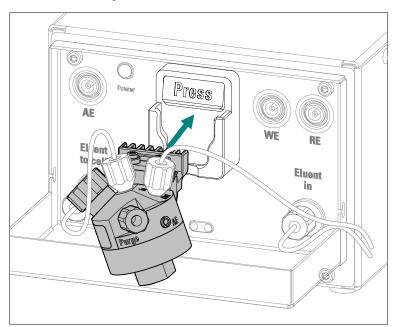
1 Connecting the measuring cell

- Connecting the measuring cell inlet:
 - When the preheating capillary is used: Use a pressure screw (6.2744.014) to fasten a piece of the PEEK capillary (6.1831.010) to the **Eluent to cell** connector on the detector.
 - Use a pressure screw (6.2744.014) to fasten the other end to the **In** connector of the measuring cell.
 - If the preheating capillary is not used: Use a pressure screw (6.2744.014) to fasten the detector inlet capillary directly to the In connector on the measuring cell.
- Connecting the measuring cell outlet:
 Use a pressure screw (6.2744.014) to fasten the tested detector outlet capillary to the **Out** connector on the measuring cell (see "Testing the detector outlet capillary", page 15).

3 Installation

2 Inserting the measuring cell

Insert the chip of the measuring cell into the cell holder so that you can hear it locking in.





NOTICE

Do not move the measuring cell for at least 5 seconds after having inserted it.

During this time, data is read from the chip of the measuring cell and written into the database. This process must not be interrupted, because otherwise the data may be transferred incorrectly or incompletely.

3 Testing at low flow

- In the program part Manual of MagIC Net, set the flow rate of the high-pressure pump to 0.2 mL/min and start the high-pressure pump.
- Watch the detector outlet capillary: Liquid must emerge from the end of the detector outlet capillary.

If no liquid emerges from the end of the detector outlet capillary:

- Detach the capillary from the **Out** connection on the measuring cell and check whether the end has been pinched by the pressure screw.
- Shorten the capillary and fasten once again to the **Out** connector on the measuring cell.

 Observe the measuring cell: No liquid should emerge from the body of the measuring cell.

If the measuring cell is leaking:

- Remove the measuring cell from the measuring cell holder.

- Remove all capillaries and cables.
- Check whether the pressure screw of the working electrode is properly connected and retighten it.
- Restore the capillary connections.
- Reinsert the electrode cables.
- Reinsert the measuring cell.
- Repeat the test.

4 Testing at normal flow

- In the program part **Manual** of MagIC Net, raise the flow rate of the high-pressure pump to 1.0 mL/min.
- Observe the measuring cell: No liquid should emerge from the body of the measuring cell.

3.3.6 Deaerating the measuring cell

The cell must be deaerated in order to ensure that it contains no air bubbles.

The measuring cell must be deaerated after the installation and after each subsequent opening of the cell.

Proceed as follows:

Deaerating the measuring cell

Prerequisites:

- The high-pressure pump is switched on and pumps the eluent through the IC system to the measuring cell.
- The measuring cell is switched off.

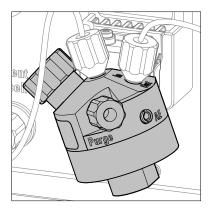
1 Deaerating the reference electrode chamber

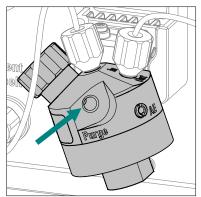
- Unscrew the nut on the RE connector and remove it.
- Lift out the reference electrode.
- Wait until the reference electrode chamber has filled with eluent.
- Reinsert the reference electrode. Wipe up any emerging eluent with a cloth.
- Screw the nut on the reference electrode connector back on tightly.

2 Removing the purge stopper

Remove the stopper from the **Purge** connector.

3 Installation





3 Deaerating the measuring cell

Observe the eluent that emerges through the deaeration opening. Wipe up liquid with a cloth.

Once no more air bubbles are visible, screw the stopper back on the **Purge** connector and tighten it by hand.

4 Switch off the high-pressure pump in MagIC Net.

3.4 Connecting the electrode cables



CAUTION

The electrode cables may not be plugged or unplugged unless the measuring cell is switched off in the software.



NOTICE

The sockets and the plugs of the cables must be clean and dry.

Connecting the electrode cables to the detector

Prerequisites:

- The measuring cell is switched off.
- 1 Plug the straight plug of the working electrode cable (red sleeve) into the **WE** socket of the detector.

2 Plug the straight plug of the reference electrode cable (black sleeve) into the **RE** socket of the detector.

Plug the straight plug of the auxiliary electrode cable (blue sleeve) into the **AE** socket of the detector.

Connecting the electrode cables to the measuring cell

Prerequisites:

- The working electrode and the reference electrode are inserted into the measuring cell.
- 1 Plug the angled plug of the working electrode cable (labeled **WE**) into the working electrode socket.
- Plug the angled plug of the reference electrode cable (labeled **RE**) into the reference electrode socket.
- Plug the angled plug of the auxiliary electrode cable (labeled **AE**) into the socket (labeled **AE**).

3.5 Attaching the front cover

In order to obtain good measuring results, we recommend that the front cover be put back in place.

When you are attaching the front cover, observe the following:

- Do not pinch any capillaries!
 Guide the capillaries through the capillary feed-throughs (1-16).
- Do not pinch any cables!

4 Operation and maintenance

4.1 Operation

The IC Amperometric Detector is operated with the MagIC Net[™] software, as is the IC instrument and the entire IC system. Additional information regarding operation can be found in the software documentation and in the online help.

4.2 Care



WARNING

The instrument housing must not be opened by untrained personnel.

The instrument requires appropriate care. Excess contamination of the instrument may result in functional disruptions and a reduction in the lifetime of the sturdy mechanics and electronics.



CAUTION

Even though design measures ensure that this will largely be prevented, the detector should be switched off without delay in the event that aggressive media have found their way into the interior of the detector. It is only thus that massive damage to the instrument electronics can be prevented. In such cases, the Metrohm Service must be informed.

Spillages of chemicals and solvents should be cleaned up immediately. In particular, the plug connections should be protected from contamination.

Do not use scouring agents for cleaning the tray.

4.3 Maintenance by Metrohm Service

Maintenance of the instrument is best carried out as part of an annual service, which is performed by specialist personnel from Metrohm. If working frequently with caustic and corrosive chemicals, a shorter maintenance interval is recommended. The Metrohm service department offers every form of technical advice for maintenance and service of all Metrohm instruments.

4.4 Maintenance

4.4.1 Maintenance



WARNING

When **rinsing the detector without column**, the pressure must not exceed **5 MPa**.

In order to ensure this, set the maximum pressure of the high pressure pump to **5 MPa** in MagIC Net.

4.4.2 Preheating capillary maintenance

The preheating capillary can become blocked, e.g. if the IC system has inadvertently been run dry.

To dissolve this blockage, proceed as follows:

Rinsing the preheating capillary

1 Removing the separation column

Remove the separation column from the IC system and replace with a coupling (6.2744.040).

2 Adjusting the settings in MagIC Net

In MagIC Net, adjust the following settings:

- Maximum pressure of the high-pressure pump: 5 MPa
- Flow rate: < 0.1 mL/min
- **3** Rinse the system with the same eluent as before the blockage or with ultrapure water.

The eluent requires sufficient time to trickle through and dissolve the crystals.

4 Do not increase the flow rate until the pressure has stabilized.

If the preheating capillary remains blocked, then you can attempt to rinse the capillary in the opposite direction. To accomplish this, connect the detector inlet capillary to the connector **Eluent to cell** and repeat the procedure (see "Rinsing the preheating capillary", page 22).

If the blockage can also not be dissolved by rinsing in the opposite direction, then the preheating capillary must be replaced by a Metrohm Service employee.

4.5 Shutting down

If the instrument is not used for a longer period, the whole IC system (without separation column) must be rinsed salt free with methanol/ultrapure water (1:4), in order to prevent eluent salts from forming crystals which may cause subsequent damage.

Rinsing salt free the IC system

To rinse the system, proceed as follows:

- 1 Remove the guard column and the separation column from the eluent path. Connect the connection capillaries directly with each other using a coupling (6.2744.040).
- 2 Rinse the IC system for 15 minutes with methanol/ultrapure water (1:4).

Rinse with eluent for at least 15 minutes at starting up again and before connecting the guard column and separation column.

5 Troubleshooting

5.1 Problems with the hardware

Problem	Cause	Remedy
Leak sensor responds.	Leaking capillary connection.	Find any leaking capillary connections and seal them.
	Measuring cell leaking.	Screw apart the measuring cell and then reassemble it.
The amperometric detector is not recognized in the software.	IC system – No connection.	 Check the cable connection. Switch the IC instrument off and then on again after 15 seconds.

5.2 Problems with the baseline

Problem	Cause	Remedy
Pulsing baseline.	High-pressure pump – Con- taminated valves.	Clean valves (see <i>Chapter Operation and maintenance</i> in the manual for the IC instrument).
	High-pressure pump – Defective piston seal.	Replace the piston seals (see <i>Chapter Operation and maintenance</i> in the manual for the IC instrument).
	High-pressure pump – Quality of the pump is not sufficient for the selected sensitivity.	 Use a pulsation absorber. Use a higher-performance high-pressure pump. Reduce the sensitivity.
	Measuring cell – Air bubble in the measuring cell.	Deaerate the measuring cell.Degas the eluent continuously.
	IC system – temperature fluctuations.	 Switch on the column thermostat or the column oven. Amperometric detector – Connect the preheating capillary . Amperometric detector – Attach and close the front cover (see chapter 3.5, page 20).

5 Troubleshooting

Problem	Cause	Remedy
	Measuring cell – Working electrode contaminated.	Clean the working electrode (see the leaflet for the working electrode).
	Measuring cell – Measur- ing cell leaking.	Check the capillary connections on the measuring cell.
	IC system – Eluent contam- inated.	Prepare a new eluent.
Smooth baseline (no noise).	Communications problem between the amperometric detector and MagIC Net.	 Check whether the electrode cables are properly connected. Check the electrode cable with dummy cell (see chapter 3.3.1, page 11). Switch off the instrument, close and restart MagIC Net, switch the instrument back on.
	All of the data lies outside of the measuring range.	 Adjust the measuring range. Deaerate the measuring cell (see "Deaerating the measuring cell", page 18).
	Short-circuit bridge between the electrodes.	 Examine the working electrode for prominent deposits. Polish the working electrode (see the leaflet for the working electrode). Replace the working electrode. Clean the measuring cell. Check the spacer.
	The reference electrode is worn out.	Replace the reference electrode.
	The cause is not clear.	Perform a systematic error diagnostics (see chapter 5.9, page 31).
The baseline has a large amount of noise.	Disruptive influences from outside.	 In the DC mode: Switch on the damping. In the other measuring modes: Set a suitable smaller measuring range. Attach the front cover.
	The Ag/AgCl reference electrode is worn out.	Replace the reference electrode.
	The auxiliary electrode is contaminated.	Clean the auxiliary electrode of the measuring cell.
	The working electrode is contaminated.	 Clean and polish the working electrode (see the leaflet for the working electrode).

Problem	Cause	Remedy
		 Replace the GC working electrode if it has been used with oxidative potentials at the upper limit and polishing no longer helps.
	Air bubble in the measur-ing cell.	Deaerate the measuring cell (see chapter 3.3.6, page 18).
	The background current is too high, e.g. caused by contaminated eluent.	Check the background current, e.g. use fresh eluent.
The baseline is drift- ng.	IC system – Thermal equili- brium not yet attained.	Condition the system with the heater switched on.
	IC system – Leak in the system.	Check the capillary connections and seal them.
	IC system – Eluent is old (too much CO₂).	Prepare a new eluent.
Unexpectedly high or low baseline.	Pd reference electrode – Working conditions not yet achieved.	Equilibrate until the electrode has adjusted to the new elution conditions (over night).
	DC method – Working conditions not yet achieved.	An excessively high baseline is normal at the start of the equilibration. Equilibrate until the baseline corresponds to the one in the Application Works.
	Detector parameters – Potentials set incorrectly.	Set the potentials to correspond to the specifications in the leaflet and in the Application Works.
	Incorrect eluent in the reference chamber.	Remove the purge stopper on the measuring cell, wait until approx. 1 mL of eluent has emerged, screw the purge stopper back in tightly.
	Electrodes contaminated.	 Clean and polish the working electrode. Possibly clean the auxiliary electrode. Replace the reference electrode with a well-conditioned new reference electrode.

5 Troubleshooting

5.3 General remarks regarding sensitivity fluctuations

Sensitivity fluctuations of up to 20% per week are normal for an unchanged system in constant operation.

The sensitivity can increase to approximately twice as much for a short time when new working electrodes are inserted or when the conditions change.

5.4 Problems with sensitivity

Problem	Cause	Remedy
Declining sensitivity.	Measuring cell – Auxiliary electrode contaminated.	Clean the auxiliary electrode (see measuring cell manual).
	Incorrect eluent in the reference chamber.	Remove the purge stopper on the measuring cell, wait until approx. 1 mL of eluent has emerged, screw the purge stopper back in tightly.
	Sample concentration is no longer correct.	Replace the sample and/or the standard solution.
	Temperature fluctuations.	 Amperometric detector – Use preheating capillary. IC instruments – Use column oven.
	Replace the measuring cell.	 Use a measuring cell of the same type. Use the same spacer. Use the same electrodes.
	Software – Measurement potential incorrect.	Optimize the measurement potential.
	Measuring cell – Working electrode contaminated.	Clean the working electrode (see the leaflet for the working electrode).
	IC system – Eluent contam- inated.	Prepare a new eluent.
	IC system – pH of the elu- ent has changed.	Check the pH value of the eluent and optimize it if necessary.

5.5 Problems with the pressure

Problem	Cause	Remedy
Marked drop in pressure.	IC system – Leak in the sys- tem.	Check the capillary connections and seal them.
The pressure in the system markedly increases.	IC system – Inline filter blocked.	Replace the filter pad (see <i>Chapter Operation</i> and maintenance in the manual for the IC instrument).
	IC system – Separation col- umn contaminated.	 Regenerate the separation column (see Chapter Operation and maintenance in the manual for the IC instrument). Replace the separation column (see Chapter Operation and maintenance in the manual for the IC instrument).
		Note: Samples should always be micro-filtered (see Chapter <i>Operation and maintenance – Inline sample preparation</i> in the manual for the IC instrument).
	Amperometric detector – Preheating capillary blocked.	Perform a maintenance procedure for the preheating capillary (see chapter 4.4.2, page 22).
	Amperometric detector – Detector outlet capillary not free of blockage.	Test the detector outlet capillary (see chapter 3.3.4, page 15).

5.6 **Problems with the measuring signal**

Problem	Cause	Remedy
Measuring signal "overload".	Air bubble in the measur-ing cell.	Deaerate the measuring cell (see chapter 3.3.6, page 18).
	Measuring cell – Working electrode damaged.	Replace the working electrode.
	Measuring cell – Measur- ing cell not correctly con- nected.	Check the cable connections (see "Connecting the electrode cables to the measuring cell", page 20).
	Software – Measurement potential incorrect.	Optimize the measurement potential.

5 Troubleshooting

Problem	Cause	Remedy
No measuring sig- nal.	IC system – No power sup- ply.	Check the power connection and the supply voltage.
Peaks cut off at the top.	Measuring range too small.	 Set a less sensitive measuring range. Reduce the peak height, e.g. by means of sample dilution.

5.7 Problems with the chromatogram

Problem	Cause	Remedy
Peak drift with sugar analysis.	Carbonate absorption in the eluent.	Use the Metrosep CO3 Trap 1 (6.1015.300) trap column.
Peaks have poor resolution.	IC system – Diminished separating efficiency of the separation column.	 Regenerate the separation column (see Chapter Operation and maintenance in the manual for the IC instrument). Replace the separation column (see Chapter Operation and maintenance in the manual for the IC instrument).
	IC system – Eluent is old.	Prepare a new eluent.
	The ionic strength of the sample or the pH value of the sample deviates greatly from the eluent.	Dilute the sample or optimize the pH value of the sample.
	Absorption of analyte at the electrodes.	Use a suitable combination of electrodes and eluent.
The retention times in the chromatogram have changed unexpectedly.	IC system – Diminished separating efficiency of the separation column.	 Regenerate the separation column (see Chapter Operation and maintenance in the manual for the IC instrument). Replace the separation column (see Chapter Operation and maintenance in the manual for the IC instrument).
	IC system – Eluent is old.	Prepare a new eluent.
	The ionic strength of the sample or the pH value of the sample deviates greatly from the eluent.	Dilute the sample or optimize the pH value of the sample.

5.8 Other problems

Problem	Cause	Remedy
Extreme spread of the peaks in the chromatogram. Splitting (dual peaks)	IC system – Dead volume at the ends of the separa- tion column.	Replace the separation column.
	IC system – Dead volume in the IC system.	Check the capillary connections.
	Inhibition of the detection mechanism by the analyte (with PAD).	Dilute the sample.Allow the waveform to run-in better.Adjust the PAD waveform.
	The column is overloaded.	Dilute the sample.

5.8 Other problems

Problem	Cause	Remedy
High background current.	IC system – Eluent contam- inated.	Prepare a new eluent.
	Software – Measurement potential / pulse settings incorrect.	Optimize the parameters.
	Very wide peaks through substances with delayed elution.	Wait for the complete elution of these substances.
Unstable tempera- ture.	The set temperature is too low.	Set the temperature to at least 8 °C higher than the highest ambient temperature to be anticipated.
Current display/ charge display in the software is frozen.	Measuring cell – electrodes are either not connected or not correctly connected.	Connect the electrode connection cables correctly (see chapter 3.4, page 19).
	Measuring cell – Small air bubbles in the measuring cell.	Deaerate the measuring cell (see chapter 3.3.6, page 18).
	Measuring cell – Electrode connection cable defective.	Perform an instrument test with the dummy cell (see chapter 3.3.1, page 11).

5 Troubleshooting

5.9 Systematic error diagnostics

If the causes of a malfunction cannot be found among the problem descriptions in the above chapters, then proceed systematically as follows:

Systematic error diagnostics

1 Restarting the instrument and the software

- Switch off the instrument.
- Close and restart MagIC Net.
- Switch the instrument back on again.

If the problem has not yet been localized, continue with Step 2.

2 Performing an instrument test with dummy cell

(see chapter 3.3.1, page 11)

If the problem has not yet been localized, continue with Step 3.

3 Checking the software settings

- Check the method parameters of the detector and reset them to values that you know will function.
- Check the measuring range and reset it to values that you know will function or select a larger measuring range.
- Check manual changes to the settings and reset them to values that you know will function.
- Check manual settings in the time program and reset them to values that you know will function.

If the problem has not yet been localized, continue with Step 4.

4 Cleaning the measuring cell

- Switch off the measuring cell.
- Remove the measuring cell.
- Clean the measuring cell (see measuring cell manual).
- Polish the working electrode (see the leaflet for the working electrode).
- Reinsert the measuring cell.

If the problem has not yet been localized, continue with Step 5.

5 Replacing the reference electrode

If the problem has not yet been localized, continue with Step 6.

6 Replacing the working electrode

If the problem has not yet been localized, continue with Step 7.

7 Replacing the body of the measuring cell

Replace the body of the measuring cell with another one of the same type.

If the problem has not yet been localized, continue with Step 8.

8 Requesting Metrohm Service

If none of these measures help, please request Metrohm Service.



NOTICE

Please note that, when the electrodes are replaced, the system requires a longer run-in time before the earlier values can be reproduced.

6 Technical specifications

6 Technical specifications

6.1 Amperometric detector

Type Microprocessor-controlled Digital Signal Processing (DSP technology)

Potentiostat

Potential range -5.0 to +5.0 V in steps of 0.001 V

Potential step < 1 ms

response time

Detection DC modes PAD

flexIPAD (flexible IPAD)

CV

Measuring unit

AutoRange yes, (DC only)

Digital signal

range

 DC mode
 0.00012 pA-2 mA

 PAD mode
 0.012 pA-2 mA

 flexIPAD mode
 0.12 pC-200 μC

 CV
 0.12 pA-20 mA

Electronic noise

DC mode < 2 pA
PAD mode < 10 pA
flexIPAD mode < 30 pC

Filter

DC mode Hardware filter, can be selected by the user

All modes Software filter, can be set by the user

Temperature con-

trol

Temperature better than 0.05 °C with ambient temperature +8 °C-80 °C

stability at the

heater

Operation

Direct Via Software MagIC Net

Remote Via Remote Box

6.1 Amperometric detector

Analog output With 891 Professional Analog Out

Output voltage 0–1000 mV

Full scale Can be adjusted within the digital signal range Offset Can be adjusted within the digital signal range

System standby • Automatic function test at start-up

Leak sensor

• Temperature stability monitoring

Output channels • Current rating

Charge

GLP conformity Yes, optional

7 Accessories

7 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. **2.850.9110**) into the search field. The search result is displayed.
- 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.

Index

Index

Α	
Amperometric detector	
Placement	8
Technical specifications	. 33
С	
Care	21
E	
Electrode cable	
Connect	10

Electrostatic charge
R
Rinsing
Detector 22
S
Safety instructions 2
Service 2, 22
Shutting down

T	
Technical specifications	
Amperometric detector	33