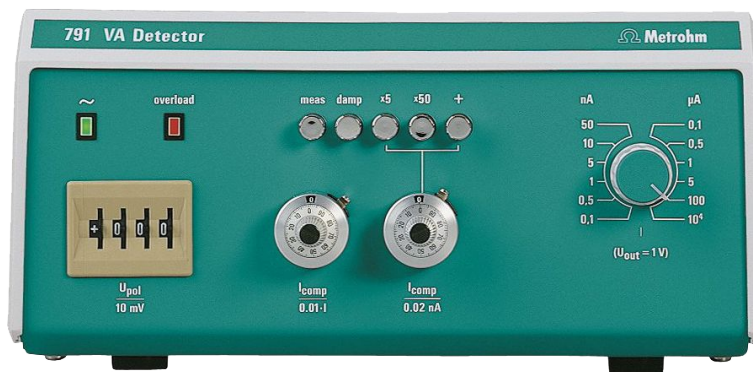


# 791 VA Detector



Manual  
8.791.1013





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# **791 VA Detector**

## **Manual**

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tion**.

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# 1 Introduction

## 1.1 Instrument description

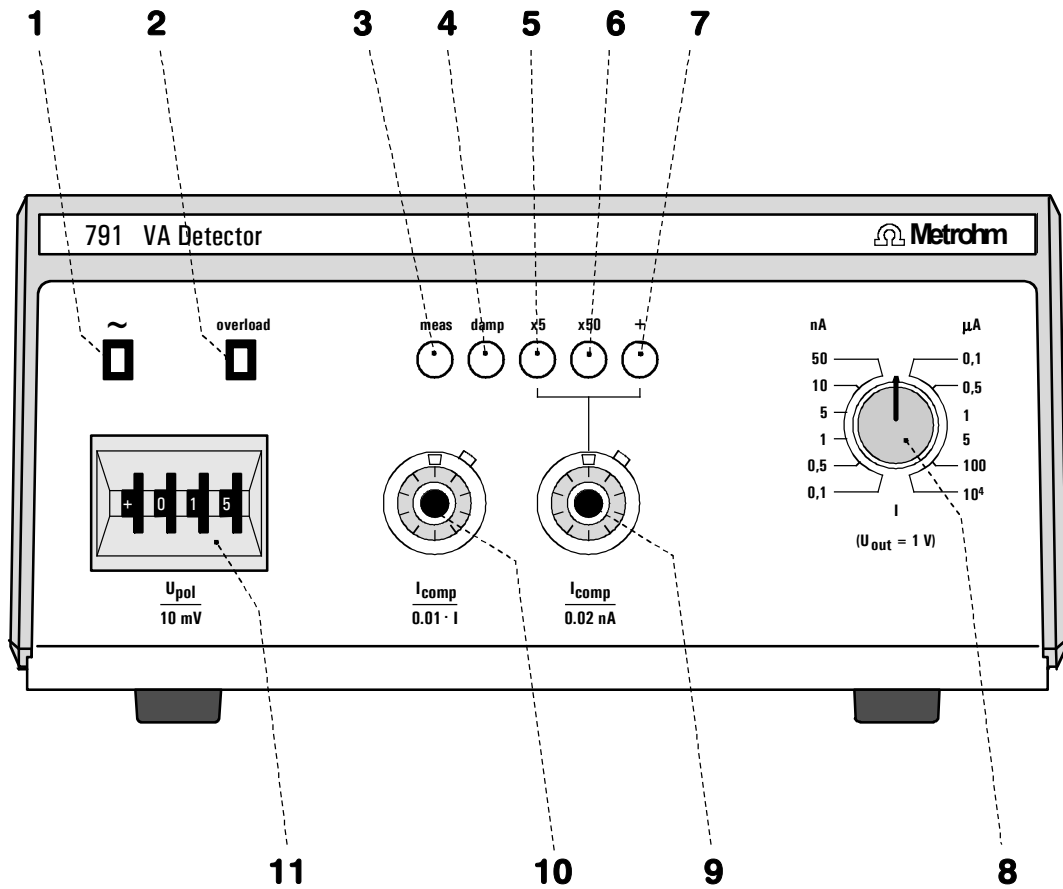
The **791 VA Detector** allows the use of electrochemical/amperometric detection in IC and HPLC; various working electrodes are available depending on the application. It can be used in series with another detector (e.g. 732 Conductivity detector) or simply as a «stand alone» detector.

The self-ventilating measuring cell functions according to the time-improved wall-jet principle with three-electrode technology. A silver/silver chloride system is used as the reference electrode and a solid gold pin as the auxiliary electrode. The carbon paste electrode and the silver electrode have proved to be the best working electrodes; but other electrode materials such as glassy carbon, gold, platinum and impregnated graphite are available for special applications.

The 791 VA Detector is available in the two following versions:

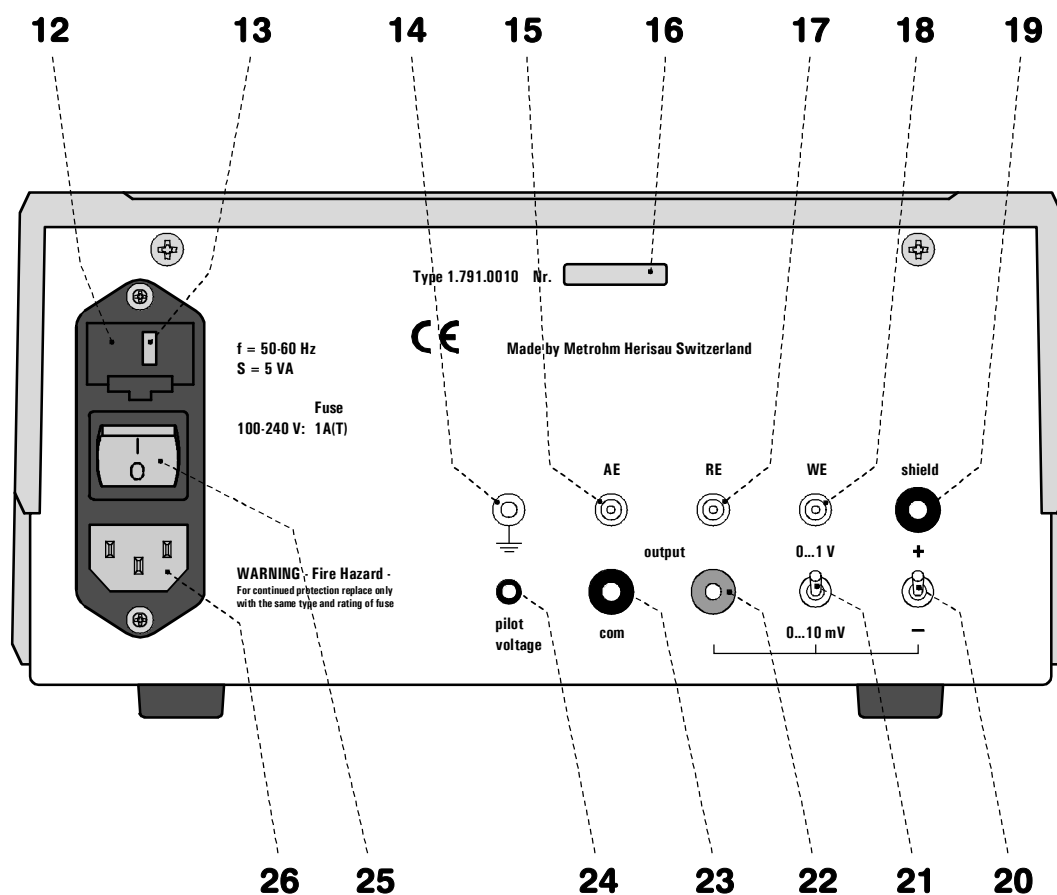
- **2.791.0010 VA Detector for HPLC**  
The measuring cell is built into the 656 Electrochemical detector.
- **2.791.0020 VA Detector for IC**  
The measuring cell belongs to the accessories and is built into the 733 IC Separation Center.

## 1.2 Parts and controls



**Fig. 1:** Front of the 791 VA Detector

<p><b>1 Mains pilot lamp</b> Lit up when instrument switched on</p>	<p><b>7 Commutator</b> Changing the polarity of <math>I_{comp}</math></p>
<p><b>2 Overload display</b></p>	<p><b>8 Adjusting knob</b> Setting the current measuring sensitivity</p>
<p><b>3 Commutator</b> Changeover Stand-by/Measure</p>	<p><b>9 Potentiometer</b> Setting the absolute compensation current <math>I_{comp}</math></p>
<p><b>4 Commutator</b> Switching on/off damping</p>	<p><b>10 Potentiometer</b> Setting the relative compensation current <math>I_{comp}</math></p>
<p><b>5 Commutator</b> Switching on/off multiplication of <math>I_{comp}</math> by a factor of 5</p>	<p><b>11 Digital switch</b> Setting the polarization voltage</p>
<p><b>6 Commutator</b> Switching on/off multiplication of <math>I_{comp}</math> by a factor of 50</p>	



**Fig. 2:** Rear of the 791 VA Detector

<b>12 Fuse holder</b> changing the fuses, see section 2.2	<b>20 Selector</b> Selection of polarity for analog output
<b>13 Mains voltage indicator</b>	<b>21 Selector</b> Selection of full-scale deflection for analog output
<b>14 Earthing socket</b>	<b>22 Analog output (live)</b>
<b>15 Connection AE</b> Connection of auxiliary electrode	<b>23 Analog output (common)</b>
<b>16 Serial number</b>	<b>24 Pilot voltage</b> Input for external voltage input for potentiostat control
<b>17 Connection RE</b> Connection of reference electrode	<b>25 Mains switch</b> To switch instrument on/off: I = ON 0 = OFF
<b>18 Connection WE</b> Connection of working electrode	<b>26 Mains connection plug</b> Mains connection, see section 2.2
<b>19 Connection for protective screen</b>	

## 1.3 Information on the Instructions for Use



*Please read through these Instructions for Use carefully before you put the 791 VA Detector into operation. The Instructions for Use contain information and warnings to which the user must pay attention in order to assure safe operation of the instrument.*

### 1.3.1 Organization





These **8.791.1013 Instructions for Use** for the 791 VA Detector provide a comprehensive overview of the installation, startup procedure, operation, fault rectification and technical specifications of this instrument. The Instructions for Use are organized as follows:

- Section 1 Introduction**  
General description of instrument, parts and controls and safety notes
- Section 2 Installation**  
Installation of accessories, connection to IC system
- Section 3 Operation**  
Electrodes, operating element functions, startup
- Section 4 Maintenance – Faults**  
Maintenance, fault rectification
- Section 6 Appendix**  
Technical data, standard equipment, options, warranty, declarations of conformity, index

To find the required information on the instruments you will find it an advantage to use either the **Table of contents** or the **Index** at the back.

### 1.3.2 Notation and pictograms

The following notations and pictograms (symbols) are used in these Instructions for Use:

15	Part or control of 791
	<p><b>Hazard</b> This symbol draws attention to a possible danger to life or of injury if the associated directions are not followed correctly.</p>
	<p><b>Warning</b> This symbol draws attention to possible damage to instruments or instrument parts if the associated directions are not followed correctly.</p>
	<p><b>Caution</b> This symbol marks important information. First read the associated directions before you continue.</p>
	<p><b>Comment</b> This symbol marks additional information and tips.</p>

## 1.4 Safety notes

### 1.4.1 Electrical safety

While electrical safety in the handling of the 791 VA Detector is assured in the context of the specifications IEC 1010-1 (protection class 1, degree of protection IP40), the following points should be noted:

- **Mains connection**



*Setting of the **mains voltage**, checking the **mains fuse** and the **mains connection** must be effected in accordance with the instructions in section 2.2.*

- **Opening the 791 VA Detector**



*If the 791 VA Detector is connected to the power supply, the instrument must not be opened nor must parts be removed from it, otherwise there is a danger of coming into contact with components which are live. Hence, always disconnect the instrument from all voltage sources before you open it and ensure that the **mains cable is disconnected from mains connection 26** !*

- **Protection against static charges**



*Electronic components are sensitive to static charging and can be destroyed by discharges. Before you touch any of the components inside the 791 VA Detector, you should earth yourself and any tools you are using by touching an earthed object (e.g. housing of the instrument or a radiator) to eliminate any static charges which exist.*

### 1.4.2 General precautionary rules

- **Handling of solvents**



*Check all lines periodically for possible leaks. Follow the relevant instructions regarding the handling of flammable and/or toxic solvents and their disposal.*

## 2 Installation

### 2.1 Setting up the instrument

#### 2.1.1 Packaging

The 791 VA Detector is supplied together with the separately packed accessories in special packagings containing shock-absorbing foam linings designed to provide excellent protection. The instrument itself is packed in an evacuated polyethylene bag to prevent the ingress of dust. Please store all these special packagings as only they assure transport of the instrument free from damage.

#### 2.1.2 Check

After receipt, immediately check whether the shipment is complete and has arrived without damage (compare with delivery note and list of accessories in *section 5.2*). In the case of transport damage, see instructions in *section 5.4.1 "Warranty"*.

#### 2.1.3 Location

Position the instrument in the laboratory at a location convenient for operation, free from vibrations and protected against a corrosive atmosphere and contamination by chemicals.

#### 2.1.4 Arrangement of the instruments

The 791 VA Detector can be placed on the 732 IC Detector or the 733 IC Separation Center.

## 2.2 Mains connection



*Follow the instructions below for connecting to the power supply. If the instrument is operated with a mains voltage set wrongly and/or wrong mains fuse, there is a danger of fire!*

### 2.2.1 Setting the mains voltage

Before switching on the 791 VA Detector for the first time, check that the mains voltage set on the instrument (see *Fig. 3*) matches the local mains voltage. If this is not the case, you must reset the mains voltage on the instrument as follows:

---

#### 1 Disconnect mains cable

Disconnect mains cable from mains connection plug **26** of the 791 VA Detector.

---

#### 2 Remove fuse holder

Using a screwdriver, loosen fuse holder **12** and take out completely.

---

#### 3 Change mains voltage

Completely remove voltage selection insert **13** by hand, rotate it through 180° and reinsert it. The required mains voltage (115 or 230 V) must now be visible from the front.

---

#### 4 Check fuses

Carefully take both fuses out of fuse holder **12** and check their specifications:

**100...240 V 1 A (slow-blow)** Metrohm-No. U.600.0016

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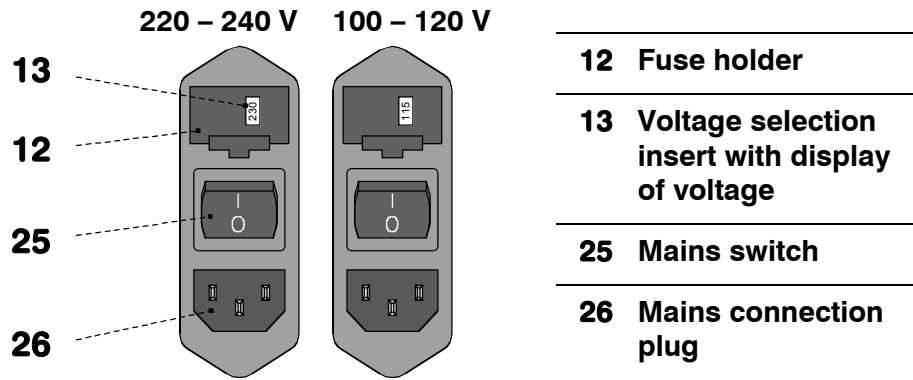
#### 5 Insert fuses

Change both fuses if necessary and reinsert in fuse holder **12**.

---

#### 6 Install fuse holder

Push fuse holder **12** back into the opening of 791 VA Detector by hand until it clicks into place properly.



**Fig. 3: Setting the mains voltage**

**2.2.2 Fuses**

Two fuses 1 A/slow-blow for 100...240 V are installed in the fuse holder **12** of the 791 VA Detector as standard.



*Ensure that the instrument is never put into operation with fuses of another type, otherwise there is danger of fire!*

For checking or changing fuses, proceed as described in section 2.2.1.

**2.2.3 Mains cable and mains connection**

**Mains cable**

The instrument is supplied with one of three mains cables

- 6.2122.020 with plug SEV 12 (Switzerland, ...)
- 6.2122.040 with plug CEE(7), VII (Germany, ...)
- 6.2133.070 with plug NEMA 5-15 (USA, ...)

which are three-cored and fitted with a plug with an earthing pin. If a different plug has to be fitted, the yellow/green lead (IEC standard) must be connected to protective earth (protection class 1).



*Any break in the earthing inside or outside the instrument can make it a hazard!*

**Mains connection**

Plug the mains cable into mains connection plug **26** of the 791 VA Detector (see Fig. 3).

**2.2.4 Switching the instrument on/off**

The 791 VA Detector is switched on and off using mains switch **25**. When the instrument is switched on lamp **1** lights up.

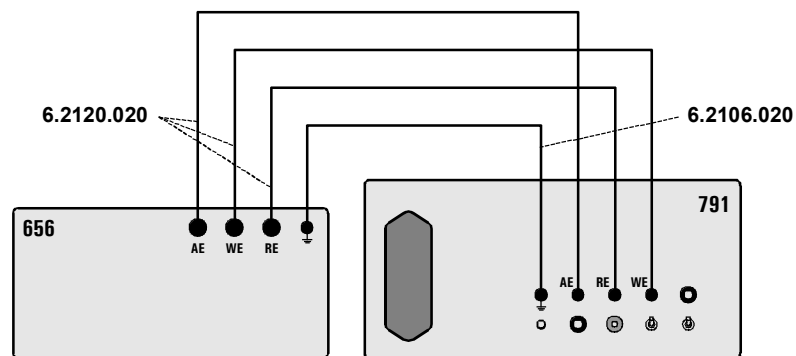
## 2.3 656 Electrochemical detector

### 2.3.1 Installation and startup

Inserting the detector cell in shield housing, fitting the electrodes and startup of the 656 Electrochemical detector (order number 2.656.0020) are described in detail in the *656 Instructions for use*.

### 2.3.2 Connection to 791 VA Detector

The electrodes used in the detector cell are each connected to the corresponding connection of the 791 VA Detector with a 6.2120.020 Electrode cable as shown in *Fig. 4*. In addition the two earthing sockets are connected with a 6.2106.020 Cable.



**Fig. 4:** Connection 656 – 791

## 2.4 6.5303.030 ELCD cell for IC

### 2.4.1 Assembling the ELCD cell

The reference electrode and working electrode must be inserted in the 6.5303.030 ELCD Cell supplied with the 2.791.0020 VA Detector. The cell is then screwed into the detector block built into the 733 IC Separation Center. Proceed as follows (see *Fig. 5* and *Fig. 6*):

---

#### 1 Insert reference electrode

Reference electrode **39** (6.0727.000, details see *section 3.2.2*) included in the supply is already filled with  $c(\text{KCl}) = 3 \text{ mol/L}$  and protected with a cap. It is assembled as follows (see *Fig. 5*):

- Screw off the cap of electrolyte storage vessel **41** of reference electrode **39**. Normally PTFE gasket **31** remains in the cap and can be left there.
- Insert one of the supplied PTFE gaskets **31** into the corresponding opening of cell body **32** and screw reference electrode **39** into this opening.

---

#### 2 Insert working electrode

Working electrode **28** is not supplied with the 791 VA Detector and must be ordered separately. Details about the working electrodes and information about pretreatment (e.g. polishing) can be found in *section 3.2.1*. A working electrode is assembled as follows (see *Fig. 5*):

- Insert working electrode **28** in screw nipple **29**.
- Push one of the supplied PTFE gaskets **31** sufficiently far along the working electrode from below.
- Carefully insert working electrode **28** into the corresponding opening of cell body **32** from above and press it in until it clearly clicks into position.
- Push screw nipple **29** downwards and screw it in.

---

#### 3 Connect ELCD cell to the detector block

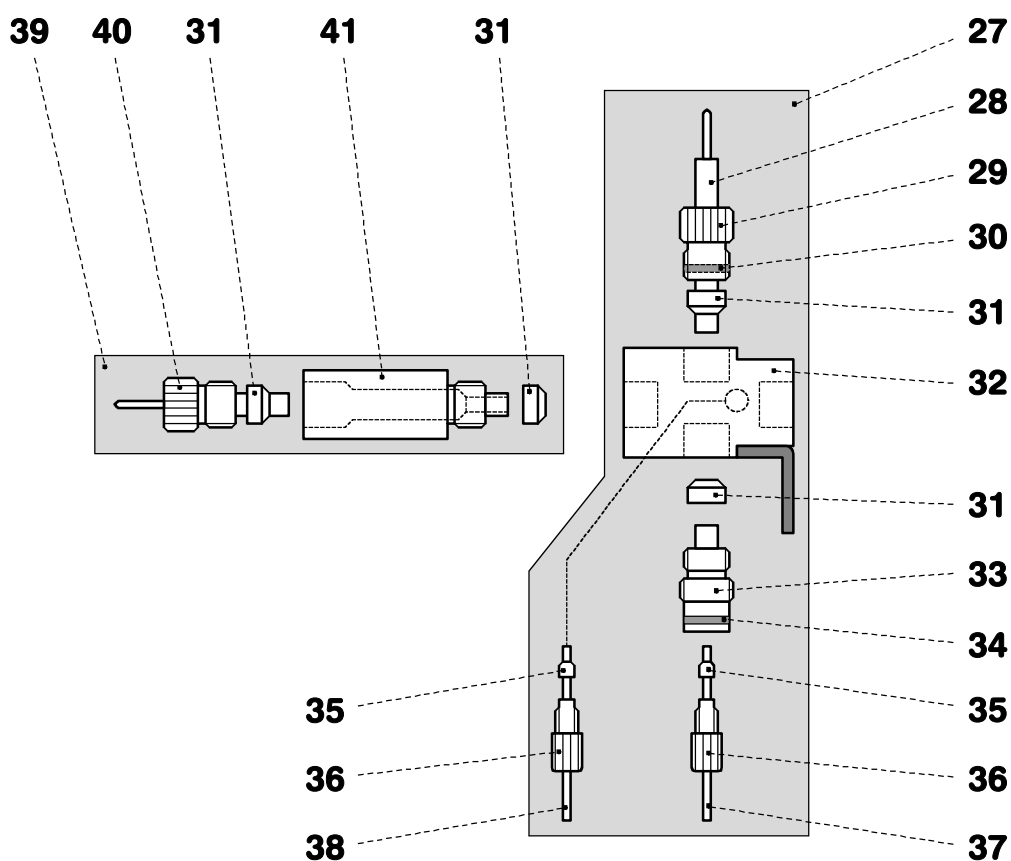
The ELCD cell is screwed onto the 1.733.0X10 Detector block mounted inside the 733 Separation Center. Proceed as follows (see *Fig. 5* and *Fig. 6*):

- Screw cell body **32** onto detector block **42** with the help of the two red knurled screws **43**.
- Push one of the supplied PTFE gaskets **31** over screw nipple **33** and screw this into the corresponding opening of cell body **32**.
- Place a screw nipple **36** and a PTFE gasket **35** on the outlet end of the outlet capillary **37** permanently connected to detector block **42**.
- Insert outlet capillary **37** with screw nipple **36** in place into screw nipple **33** and press in outlet capillary **37** until it reaches the stop.

- Screw nipple **36** onto screw nipple **33** to fix outlet capillary **37** in position.

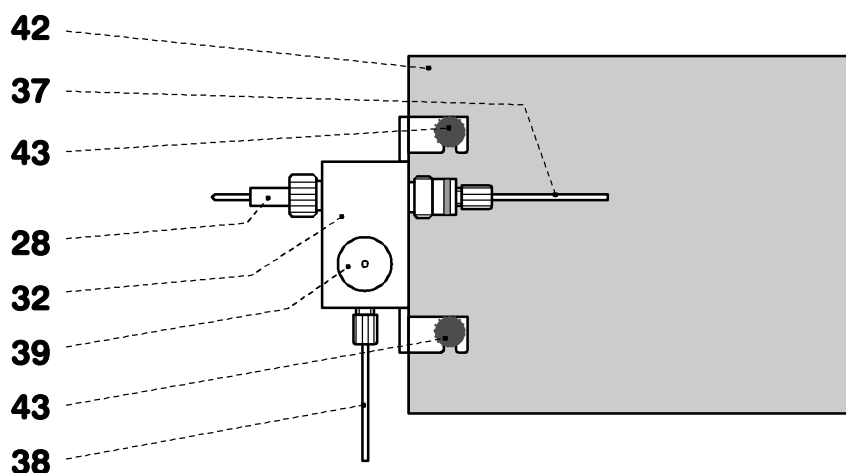
#### 4 Discharge

- Pull outlet capillary **38** which is already mounted on cell body **32** through one of the openings in the rear panel of the 733 IC Separation Center so that it emerges outside the instrument.
- Insert outlet capillary **38** in a sufficiently large waste container and fix it in position.



**Fig. 5:** ELCD cell assembly

<b>27</b>	<b>ELCD cell (6.5303.030)</b>	<b>35</b>	<b>PTFE gasket (6.2704.020)</b>
<b>28</b>	<b>Working electrode</b>	<b>36</b>	<b>Screw nipple (4.420.2580)</b>
<b>29</b>	<b>Screw nipple (6.2730.000)</b>	<b>37</b>	<b>PEEK outlet capillary from 1.733.0X10 Detector block</b>
<b>30</b>	<b>Sealing ring (E.301.0028)</b>	<b>38</b>	<b>PTFE outlet capillary (6.1803.000)</b>
<b>31</b>	<b>PTFE gasket (6.2704.010)</b>	<b>39</b>	<b>Reference electrode (6.0727.000)</b>
<b>32</b>	<b>Cell body</b>	<b>40</b>	<b>Ag/AgCl Reference system</b>
<b>33</b>	<b>Screw nipple (6.2731.030)</b>	<b>41</b>	<b>Electrolyte vessel</b>
<b>34</b>	<b>Sealing ring (E.301.0004)</b>		

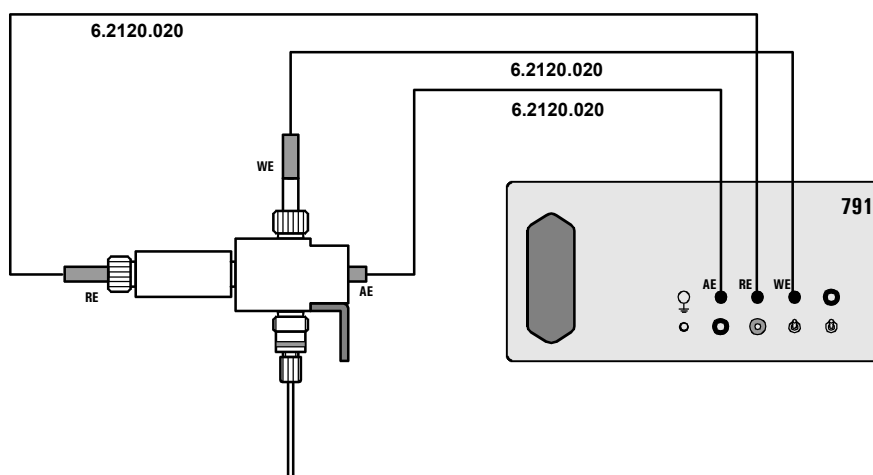


**Fig. 6:** Connection of ELCD cell at detector block

<b>28</b>	<b>Working electrode</b>	<b>39</b>	<b>Reference electrode (6.0727.000)</b>
<b>32</b>	<b>Cell body</b>	<b>42</b>	<b>Detector block (1.733.0X10)</b>
<b>37</b>	<b>PEEK outlet capillary from 1.733.0X10 Detector block</b>	<b>43</b>	<b>Knurled screw (V.900.4006)</b>
<b>38</b>	<b>PTFE outlet capillary (6.1803.000)</b>		

### 2.4.2 Connection to 791 VA Detector

The ELCD cell electrodes are each connected to the corresponding connection of the 791 VA Detector with a 6.2120.020 Electrode cable as shown in Fig. 7. The 791 VA Detector must be switched off and in the "stand-by" position when this is taking place.



**Fig. 7:** Connection of ELCD cell at 791 VA Detector

## 2.5 Analog output connections

The 791 VA Detector has two analog output sockets: **22** (live) and **23** (common) for connecting a chart recorder or a different data recording instrument. The full-scale deflection range for the analog output (1 V or 10 mV) can be set with switch **21**; the polarity can be changed with switch **20** (see *section 3.3*).

The 6.2115.010 Cable can be used for connecting recorders with banana plug connections; the 6.2115.060 Cable can be used for connecting data recording instruments with terminals.

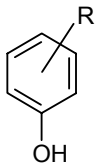
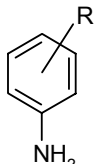
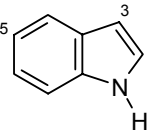
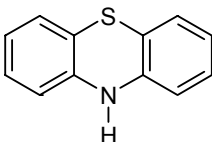
# 3 Operation

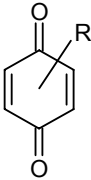
## 3.1 Electrochemical detection

### 3.1.1 Classes of substances which can be determined

A precondition for the use of electrochemical detection is that the substances to be determined are electrochemically active on the particular working electrode used, i.e. they can easily be oxidized or reduced. The electrical current produced by this reaction is proportional to the concentration of the substance throughout a wide range. It is measured, amplified and recorded as a function of time by the 791 VA Detector in the form of a chromatogram.

The following table provides an overview of the classes of substances and ions which can be detected by oxidation or reduction. The approximate polarization voltage obtained when an  $\text{Ag}/\text{AgCl}/c(\text{KCl}) = 3 \text{ mol/L}$  reference electrode is used is given; this depends to a large extent on the working electrode and eluents used (see section 3.2). The limits of detection which can be achieved are in the lower ppb range.

Structural formula	Classes/substances	Polarization voltage
	<b>Aromatic hydroxy compounds</b> Antioxidants Catecholes Flavones Halogenated phenols Hydroxybiphenyls Hydroxycumarines Methoxyphenols Oestrogens Phenols Tocopherols	+800 ... +1200 mV + 800 mV + 1000 mV + 1200 mV + 800 mV + 1000 mV + 800 mV + 1000 mV + 1200 mV + 800 mV
	<b>Aromatic amines</b> Anilines Benzidines Sulfonamides	+ 1000 mV + 600 mV + 1200 mV
	<b>Indols</b> Indolyl-3-compounds 5-Hydroxy-indols	+ 1000 mV + 800 mV
	<b>Phenothiazines</b>	+ 1000 mV

Structural formula	Classes/substances	Polarization voltage
R-SH	<b>Mercaptans</b>	+ 800 mV
	<b>Miscellaneous</b> Ascorbic acid Carotenes Purine derivatives Vitamin A	+ 800 mV + 800 mV + 1000 mV + 1000 mV
	<b>Quinones</b> Vitamin K <sub>1</sub>	- 700 mV
R-NO <sub>2</sub>	<b>Nitro compounds</b> Nitrophenol Nitroglycerin Isosorbidedinitrate	- 700 mV - 800 mV - 800 mV
R <sup>-</sup>	<b>Inorganic anions</b> Nitrite Sulfide Thiosulfate Chlorite Bromide Iodide Cyanide Thiosulfate Thiocyanate	+ 1100 mV + 0 mV + 1100 mV + 1100 mV + 150 mV + 150 mV + 0 mV + 1100 mV + 1200 mV

### 3.1.2 Influence on detector signal

The sensitivity of detection, i.e. the height of the detector signal and the background current in the electrochemical detection depend on the following parameters:

- polarization voltage
- temperature (approx. 1.5 %/°C)
- flow rate of eluent
- surface of electrodes and interior of cell
- Eluent composition  
(pH value, type and concentration of conductive salt,  
type and concentration of organic components, etc.)

Each change of one of these parameters will change the background current and the detector signal. Therefore, every parameter change needs a stabilization time to elapse; this is normally a few minutes for

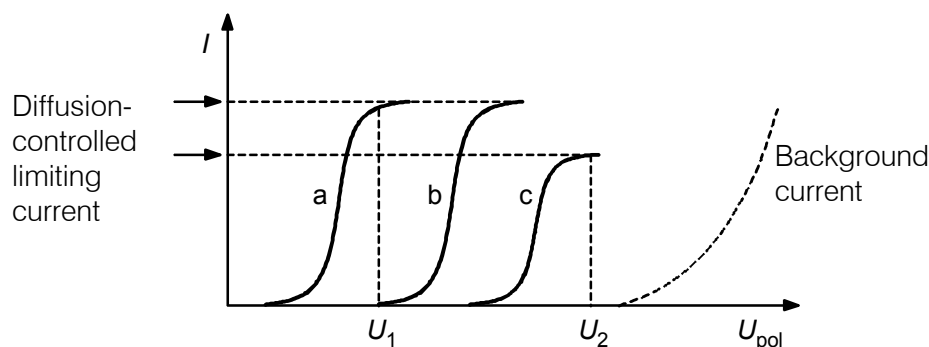
current ranges > 5 nA. Please pay particular consideration to this on changing the eluent or the sample solution: the "same" eluent may have a different background current because eluents are never exactly the same (ionic strength, pH value, etc.).

### 3.1.3 Selection of polarization voltage

The hydrodynamic voltammograms show the voltage limit from the point where a substance is oxidized or reduced. This voltage limit is generally higher than the voltage predicted by thermodynamics because of inhibitions of the electrode reaction. Increasing voltage values give rise to increasing currents up to a maximum value, the diffusion-controlled limiting current.

In Fig. 8 for example, analytes a, b, and c are detected at voltage  $U_2$ ; whereas at voltage  $U_1$ , component a is detected selectively. Therefore the polarization voltage to be set has to be optimized for sensitivity on one hand and selectivity on the other.

The polarization voltage to be set also depends on the type and condition of the working electrode (see section 3.5) as well as on the eluent. The latter could be illustrated by Fig. 8, saying that the curves are from one single analyte taken in 3 eluents with different pH values.



**Fig. 8: Selection of polarization voltage**

### 3.1.4 Procedure for unknown substances

The hydrodynamic current/voltage curve of an analyte with unknown electrochemical characteristics is recorded as follows:

- Short circuit the system, i.e. connect the injector directly (without separating column) to the electrochemical detector.
- Set current range of 791 VA Detector to 100 ... 500 nA.
- Set polarization voltage of 791 VA Detector to +1200 mV.
- Inject analyte (20 ... 100 ng).

If no signal is observed, change the pH value of the eluent (a protonated or deprotonated substance may be oxidized more easily). Generally, a higher polarization voltage has to be applied with lower pH values (approx. 50 ... 100 mV per pH unit).

- 1<sup>st</sup> experiment with eluent of pH  $\approx$  6
- 2<sup>nd</sup> experiment with eluent of pH  $\approx$  3

If there is still no signal, the analyte is "inactive", i.e. not suitable for electrochemical detection.

As soon as a signal is observed, decrease the polarization voltage in steps of 100 mV: a hydrodynamic current/voltage curve results.

For detection, select the lowest polarization voltage  $U_{\text{pol}}$  at which a good signal is still observed.

### 3.1.5 Practical information about ELCD

In principle the electrochemical detector can be connected to all HPLC and IC systems. In order to carry out work successfully, particularly in trace-level quantities, all components such as pump, dampening system, injector, separating column, capillaries, couplings, etc. must be in perfect condition. In electrochemical detection it is possible that interferences may occur which, for example, are not observed with UV or conductivity detection. These can often be traced back to contaminants from the water supply system (e.g. Fe(II) ions), contaminants from the separating column (e.g. organic residues) or variations in pressure caused by leaking connections.

Particular attention must be given to the following points:

#### **Cleanliness**

The general demands placed on trace analysis work also apply here. Each possible source of corrosion of steel components must be avoided as Fe(II) ions may interfere. The eluent should also not contain any complexing agents.

#### **Pulsation**

The noise of the detector signal depends on the pressure variation inside the cell and on the back-pressure (max. 1 bar), which is determined by the length of outlet capillary **38** (i.d. 0.3 mm). The use of a pulsation dampener (e.g. Metrohm 6.2620.150 Pulsation dampener) for dampening such pulsations has proved itself in practice and is also adequate if only single-piston pumps are used.

#### **Eluent**

Most of the solvents normally used in HPLC and IC can be used to prepare eluents. The conductivity which is necessary for detection is achieved by the addition of a conductive salt. Concentrations of 1...10 g/L are normally adequate for this purpose, this results in a conductivity of approx. 1...10 mS/cm. Sulfates, nitrates, phosphates, acetic acid,

sulfuric acid, perchloric acid, lithium perchlorate, etc. can be used for this. Chlorides and hydroxycarboxylic acids should not be used; some reagents which form ion pairs can be used.

Solvent gradients can be used in less sensitive ranges (current range > 100 nA). The eluent is degassed as usual by vacuum or passing a stream of helium through it.

### **Stationary phase**

In principle all reversed-phase and ion exchanger materials can be used. New columns must be purged well and for a sufficiently long time for any contaminants which may be present to be washed out. This "equilibration phase" of a column may take more than 24 hours in the most sensitive areas.

### **Sample solution**

Whenever possible, the eluent used should also be used as the solvent for the sample in order to minimize interference owing to changing surroundings (as small a front peak as possible). Very small amounts of the active substance should be used, in general < 100 ng.

## 3.2 Electrodes

### 3.2.1 Working electrodes

The quality of a working electrode WE can be assessed in terms of the following criteria:

- **Background current** (after adequate running-in time):  
this should be small and constant
- **Interference level** ("noise"):  
this should be low
- **Response characteristics**:  
after switching on, the low background current should be quickly reached

It should nevertheless be borne in mind that increases in both background current and noise levels are often due to other causes than a poor-quality working electrode (for example, contamination of the HPLC system, malfunction of the pulsation dampener, etc.). Generally speaking, both background current and noise levels tend to increase with higher polarization voltages, and are usually greater in eluents based on mixed solvents than in those composed of purely aqueous solutions.

#### **Glassy carbon electrode GCE**

*Ordering designation* 6.0805.010

*Sensor material* Glassy carbon, GC

*Chemical resistance* good resistance to acetonitrile, methanol, water, etc.

*Voltage range* (–800) ... 0 ... +1200 mV (pH < 7)

#### *Regeneration*

Sprinkle a little aluminium oxide powder ( $\text{Al}_2\text{O}_3$ , grain size 0.3  $\mu\text{m}$ ) on the polishing cloth (ordering number of both these accessories: 6.2802.000) and moisten with distilled water. Hold the electrode vertically and polish with a circular motion for about 20...60 s, exerting only light pressure. Rinse with distilled water and clean with dry cloth. A glassy carbon electrode should always be polished for a short while immediately before being fitted to the detector cell.

#### **Ultra-Trace graphite electrode**

*Ordering designations* 6.1204.100 Ultra-Trace graphite tip  
+ 6.2103.110 Contact pin

*Sensor material* Graphite (turnover is three times larger than in GC-electrode; does not need tamping)

*Chemical resistance* good resistance to acetonitrile, methanol, water, etc.

*Voltage range* –800 ... 0 ... +1200 mV (pH < 7)

#### *Regeneration*

With 6.2827.000 Trimming tool (see electrode data sheet).

### Carbon paste electrode CPE

<i>Ordering designation</i>	6.0807.000
<i>Sensor material</i>	paste composed of spectroscopic grade carbon powder and paraffin
<i>Chemical resistance</i>	good resistance to aqueous solutions; limited resistance only to water/acetonitrile and water/methanol mixtures
<i>Voltage range</i>	(-200) ... 0 ... +1200 mV (pH < 7)

#### *Regeneration*

The sensor surface of the CPE is more vulnerable to both mechanical damage and chemical attack than that of the GCE, it is more easily regenerated: often it suffices merely to rub the tip of the electrode backwards and forwards a few times against the surface of a sheet of ordinary, smooth paper, or alternatively the upper layer of carbon paste can be renewed (see below) and then polished in this manner.

#### *Filling*

Carefully remove the old carbon paste from the approximately 2.5 mm deep cavity with a suitable sharp instrument. Take care not to damage the platinum contact wire at the bottom of the cavity. Using a small spatula, fill bit by bit with the fresh 6.2801.020 carbon paste and tamp well down using the 6.2826.000 stopper. The cavity should be filled over the brim with paste. Now rub off the excess paste by continuous circular motion against a sheet of paper with a hard, smooth, non-porous surface; the circular motion plus the light pressure will polish the surface of the paste, which must be smooth and shiny when the operation is finished. There should be no cracks in the peripheral zone where the paste is up against the plastic material of the electrode shaft.

### Metal electrodes

<i>Ordering designations</i>	6.1204.120 Pt tip 6.1204.130 Ag tip 6.1204.140 Au tip + 6.2103.110 Contact pin
<i>Chemical resistance</i>	good resistance to acetonitrile, methanol, water, etc.
<i>Voltage range</i>	Pt: (-1200) ... 0 ... +200 mV (pH < 7) Ag: (-800) ... 0 ... +400 mV (pH > 7) Au: (-400) ... 0 ... +1200 mV (pH < 7)

#### *Regeneration*

Sprinkle a little aluminium oxide powder (Al<sub>2</sub>O<sub>3</sub>, grain size 0.3 μm) on the polishing cloth (ordering number of both these accessories: 6.2802.000) and moisten with distilled water. Hold the electrode vertically and polish with a circular motion for about 20...60 s, exerting only light pressure. Rinse with distilled water and clean with dry cloth. A metal electrode should always be polished for a short while immediately before being fitted to the detector cell.

### Amalgamated electrode

"Home-made" amalgamated electrodes are prepared as follows:

<i>Base material</i>	6.1204.140 Au tip
<i>Preparation</i>	Put a few drops of Hg on the front side of the electrode and leave it for a few minutes. Knock off excess Hg and rinse electrode with distilled water.
<i>Chemical resistance</i>	good resistance to acetonitrile, methanol, water
<i>Voltage range</i>	-1200 ... 0 mV
<i>Regeneration</i>	polish electrode with aluminium oxide and re-amalgamate

### 3.2.2 Reference electrode

The reference system of the 6.0727.000 Reference electrode (RE) is Ag/AgCl/c(KCl) = 3 mol /L (see Fig. 5). The electrolyte vessel **41** can be refilled. The KCl solution has to be renewed every 1...2 months.

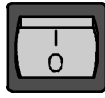
Ordering designations for KCl solutions: 6.2308.020 (250 mL)  
6.2313.000 (1 L)

### 3.2.3 Auxiliary electrode

A gold electrode is built-in in the detector cell as an auxiliary electrode.

### 3.3 Operating element functions

#### Mains switch



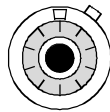
The 791 VA Detector is switched on and off using mains switch **25** on the rear of the instrument (see Fig. 2):

- I Instrument switched on
- 0 Instrument switched off



After the instrument has been switched on, the mains pilot lamp **1** lights up to show that the instrument is ready for use.

#### Relative compensation current

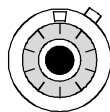


$$\frac{I_{comp}}{0.01 \cdot I}$$

The relative compensation current  $I_{comp} rel.$  is set using the potentiometer **10** (see Fig. 1). The value set is related to the setting of the adjusting knob **8** (current  $I$ ).

- Setting range** -500 (red) ... +500 (white)  $\times 0.01 \cdot I$
- Resolution**  $0.01 \cdot I$  per scale division

#### Absolute compensation current



$$\frac{I_{comp}}{0.02 \text{ nA}}$$

The absolute compensation current  $I_{comp} abs.$  is set using the potentiometer **9** (see Fig. 1). The value set is not related to the setting of the adjusting knob **8** (current  $I$ ).

- Setting range**
  - direct  $0 \dots 1000 \times 0.02 \text{ nA} = 20 \text{ nA}$
  - with commutator **5**  $5 \times 20 \text{ nA} = 100 \text{ nA}$
  - with commutator **6**  $50 \times 20 \text{ nA} = 1000 \text{ nA}$
- Resolution**
  - direct  $0.02 \text{ nA}$  per scale division
  - with commutator **5**  $0.10 \text{ nA}$  per scale division
  - with commutator **6**  $1.00 \text{ nA}$  per scale division

The polarity of the absolute compensation current is set separately with commutator **7**.

#### Changeover Stand-by/Measure



For this changeover commutator **3** (see Fig. 1) is used.

- Button depressed**  
Measuring position, i.e. measuring cell connected.
- Button not depressed**  
Stand-by position, i.e. measuring cell disconnected.



### Damping

The damping is switched on and off using commutator **4** (see *Fig. 1*).

#### Button depressed

Signal damped for all ranges set on adjusting knob **8** in order to suppress interference (spurious signals).

#### Button not depressed

No signal damping (exception: the most sensitive range, 0.1 nA, is always damped to some extent).



### Multiplication of $I_{comp}$ abs. by a factor of 5

For this changeover commutator **5** (see *Fig. 1*) is used.

#### Button depressed

Multiplication of the absolute compensation current by a factor of 5.

#### Button not depressed

No multiplication of the absolute compensation current.



### Multiplication of $I_{comp}$ abs. by a factor of 50

For this changeover commutator **6** (see *Fig. 1*) is used.

#### Button depressed

Multiplication of the absolute compensation current by a factor of 50.

#### Button not depressed

No multiplication of the absolute compensation current.



### Polarity of $I_{comp}$ abs.

For this changeover commutator **7** (see *Fig. 1*) is used.

#### Button depressed

All current values set on the potentiometer **9** have a positive sign.

#### Button not depressed

All current values set on the potentiometer **9** have a negative sign.

### Overload display

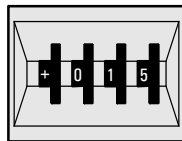


If the electronic current measuring circuit is overloaded, the red light **2** is lit up (see Fig. 1).

#### Remedies

Select a less sensitive current range with adjusting knob **8** or compensate current with potentiometer **9** and/or **10** (for further measures see section 4.3).

### Polarization voltage



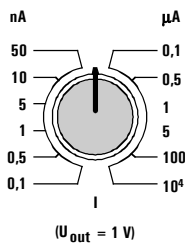
$U_{pol}$   
10 mV

The detection voltage  $U_{pol}$  is set using the digital switch **11** (see Fig. 1).

**Setting range**                    -1990 ... 0 ... +1990 mV

**Resolution**                        10 mV

### Current measuring sensitivity



The sensitivity of current measurement is set using adjusting knob **8** (see Fig. 1). The set range applies to the full-scale deflection of the analog signal at sockets **22** and **23** (1 V or 10 mV depending on the setting of switch **21**).

**Setting ranges**                    0.1, 0.5, 1, 5, 10, 50 nA  
0.1, 0.5, 1, 5, 100, 10'000  $\mu$ A

### Polarity of analog output



The polarity (+ or -) of the analog output signal is set using switch **20** (see Fig. 2).

### Full-scale deflection of analog output



The full-scale deflection (1 V or 10 mV) of the analog output signal is set using switch **21** (see Fig. 2).

## 3.4 Startup

### 3.4.1 Preparing the ELCD cell

The ELCD cell must be correctly connected to the HPLC or IC system before it is filled (see *section 2.3* and *section 2.4*). Then proceed as follows:

---

#### 1 Fill reference electrode (only if necessary)

Reference electrode **39** (6.0727.000) is supplied in a filled condition. However, the KCl electrolyte solution must be replaced every 1 to 2 months. Proceed as follows:

- Screw Ag/AgCl reference system **40** out of electrolyte vessel **41** (see *Fig. 5*).
- Fill electrolyte vessel **41** to the brim with  $c(\text{KCl}) = 3 \text{ mol/L}$ .
- Screw Ag/AgCl reference system **40** back onto electrolyte vessel **41**. Take care that no air bubbles are found in the electrolyte vessel (to check: hold reference electrode against the light).

---

#### 2 Connect reference electrode

- Insert reference electrode **39** into cell body **32** (see *Fig. 5*).
- Connect reference electrode **39** to connection **17** of the 791 VA Detector with 6.2120.020 Cable (see *Fig. 7*).

---

#### 3 Connect auxiliary electrode

- Connect the auxiliary electrode in cell body **32** to connection **15** of the 791 VA Detector 791 with 6.2120.020 Cable (see *Fig. 7*).

---

#### 4 Prepare working electrode

- Prepare and polish working electrode **28** according to *section 3.2.1*.
- Insert working electrode **28** into cell body **32** (see *Fig. 5*).
- Do not connect working electrode **28** to the 791 VA Detector at this point.

---

#### 5 Start pump

- Switch on delivery drive of HPLC or IC pump.

### 3.4.2 Zero balancing and equilibration phase

Before the first measurement is made the working electrode must be allowed to equilibrate and the chart recorder or data recording instrument must be zero-balanced. Proceed as follows:

---

#### 1 Chart recorder settings

If a chart recorder is used to record the analog signals then proceed as follows (the working electrode must not be connected to the 791 VA Detector):

- Switch on 791 VA Detector.
- Set commutator **3** on the 791 VA Detector to "**Stand-by**".
- Set the required polarity (+ or -) with switch **20**; use switch **21** to set the required voltage range for the analog output on the 791 VA Detector (**1 V** or **10 mV**).
- Set potentiometer **9** on the 791 VA Detector to **0**.
- Use knob **8** on the 791 VA Detector to set the current sensitivity to **0.5  $\mu$ A**.
- Switch on recorder.
- Set the voltage measuring range on the recorder to the same setting as on the 791 VA Detector (**1 V** or **10 mV**).
- Use potentiometer **10** on the 791 VA Detector to adjust the zero point of the recorder.

---

#### 2 Settings with a data recording program

If a PC recording and evaluation program is used for recording the analog signals then proceed as follows (the working electrode must not be connected to the 791 VA Detector):

- Switch on 791 VA Detector.
- Set commutator **3** on the 791 VA Detector to "**Stand-by**".
- Set the required polarity (+ or -) with switch **20**; use switch **21** to set the required voltage range for the analog output on the 791 VA Detector (**1 V** or **10 mV**).
- Set potentiometers **9** and **10** on the 791 VA Detector to **0**.
- Use knob **8** on the 791 VA Detector to set the current sensitivity to **0.5  $\mu$ A**.
- Switch on the data recording program on the PC and start recording data.
- If required, use potentiometer **10** on the 791 VA Detector to adjust the zero point (so that the mV display on the PC shows 0).

---

**3 Overload test**

For this test the working electrode must not be connected to the 791 VA Detector:

- Set the polarization voltage on the 791 VA Detector to the required value for the following determinations with digital switch **11**.
- Set commutator **3** on the 791 VA Detector to "**meas**". Overload display **2** must light up briefly and then go out again.
- If overload display **2** remains lit up for a longer period of time this indicates that there is a faulty electrical contact between the reference electrode and the auxiliary electrode. Eliminate the cause (e.g. air in the system or in the reference electrode, blocked diaphragm, faulty cable connection) and repeat the test.
- If the overload test is OK, set commutator **3** on the 791 VA Detector back to "**Stand-by**".

---

**4 Equilibrating the working electrode**

- Connect working electrode **28** to connection **18** of the 791 VA Detector with the 6.2120.020 Cable.
- Set commutator **3** on the 791 VA Detector to "**meas**".
- Observe equilibration curve: the curve should flatten out noticeably within a relative short time (10...20 min), otherwise the working electrode must be repolished or refilled.
- In current measuring ranges  $> 5$  nA a constant base current is usually achieved with one hour. At higher sensitivities the working electrode should be equilibrated overnight.

---

**5 Zero balancing the base current (only for recorders)**

- Set the current measuring sensitivity on the 791 VA Detector with knob **8** to the required value.
- Use potentiometer **9** on the 791 VA Detector to adjust the base current to the zero point of the recorder. If the setting range of potentiometer **9** is not sufficient then it can be extended by using commutator **5** ( $\times 5$ ) or **6** ( $\times 50$ ).

### 3.4.3 Measuring procedure

In order to be able to carry out measurements it is essential that the working electrode has achieved a stable base current (see *section 3.4.2*). If the value of the base current regains its original value after the determination then as many determinations as required can be carried out. Attention should be given to the following points:

---

#### 1 Carrying out the determination

- Inject the sample solution into the HPLC or IC system.
- Record and evaluate the current curve.

---

#### 2 Allow ELCD system to run overnight

- If the ELCD system is to be used again on the following day then the 791 VA Detector should not be switched off overnight. The working electrode is constantly under the polarization current and is quickly ready for new measurements.
- The HPLC or IC pump is set to a minimal flow rate and runs overnight. This means that the base current is stabilized after only a few minutes.

---

#### 3 Switching off the ELCD system

- Set commutator **3** on the 791 VA Detector to "Stand-by".
- Unplug working electrode **28**.
- Switch off the 791 VA Detector.



## 4 Maintenance – Malfunctions

### 4.1 Maintenance and servicing

#### Care

The 791 VA Detector requires proper care and attention. Excessive contamination of the instrument could possibly lead to malfunctions and a shorter service life of the inherently rugged mechanical and electronic parts.

Spilled chemicals and solvents should be wiped up immediately. It is especially important to protect the plug connections at the rear of the instrument (particular the mains plug) against contamination.



*Although constructional measures have been designed to virtually eliminate such a situation, should corrosive media penetrate the interior of the instrument the mains plug of the 791 VA Detector must be immediately disconnected to prevent extensive damage to the instrument electronics. Inform Metrohm service if your instrument have been damaged in such a way.*



*The instrument must not be opened by untrained personnel. Please comply with the safety notes in section 1.4.1.*

#### Maintenance by Metrohm service

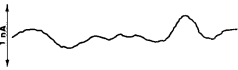
Maintenance of the 791 VA Detector is best done as part of an annual service performed by specialists from the Metrohm company. If work is frequently performed with caustic and corrosive chemicals, it may be necessary to shorten the interval between servicing.


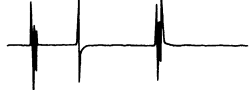

The Metrohm service department is always willing to offer expert advice on the maintenance and servicing of all Metrohm instruments.

### 4.2 Shutdown

If the ELCD cell is shut down for a considerable length of time, the entire HPLC or IC system (**without** column and suppressor) must be **rinsed free from salt** with methanol/water (1:4) to avoid crystallization of eluent salts with the corresponding subsequent damage.

## 4.3 Malfunctions and their rectification

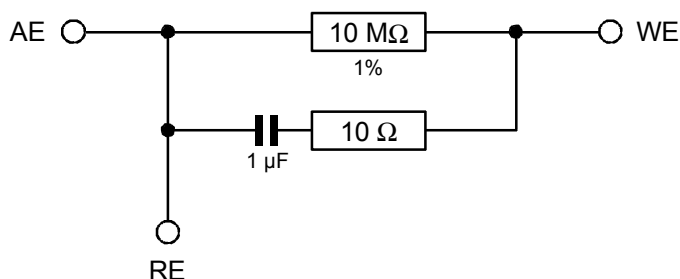
<b>Malfunction</b>	<b>Cause</b>	<b>Rectification</b>
<b>No signal</b>	<ul style="list-style-type: none"> <li>• VA Detector is in "stand-by" mode.</li> <li>• VA Detector switched off or disconnected from mains voltage.</li> <li>• Recorder switched off or disconnected from mains voltage.</li> <li>• Recorder disconnected from VA Detector.</li> <li>• Electrodes disconnected.</li> <li>• Broken interconnecting cables.</li> </ul>	<ul style="list-style-type: none"> <li>• Press "meas" button on VA Detector.</li> <li>• Connect VA Detector and switch on.</li> <li>• Connect recorder and switch on.</li> <li>• Connect recorder to the VA Detector.</li> <li>• Connect electrodes.</li> <li>• Use new interconnecting cables.</li> </ul>
<b>VA Detector shows "overload"</b>	<ul style="list-style-type: none"> <li>• Current range set exceeded by too high a concentration of substance.</li> <li>• The VA Detector is either incorrectly balanced or not balanced at all.</li> <li>• Reference electrode disconnected.</li> <li>• Auxiliary electrode disconnected.</li> <li>• Polarization voltage badly adjusted.</li> </ul>	<ul style="list-style-type: none"> <li>• Wait or set a higher current range at the VA Detector.</li> <li>• Balance the recording instrument and VA Detector correctly.</li> <li>• Connect reference electrode.</li> <li>• Connect auxiliary electrode.</li> <li>• Adjust polarization voltage correctly.</li> </ul>
<b>Excessively high background current</b>	<ul style="list-style-type: none"> <li>• Polarization voltage too high.</li> <li>• Eluent contaminated.</li> <li>• Eluent contaminated by Fe(II) ions from rusty steel parts (traces only suffice!)</li> </ul>	<ul style="list-style-type: none"> <li>• Polarization voltage should generally not be set higher than 1200 mV.</li> <li>• Prepare fresh eluent using only the purest (AR grad) chemicals.</li> <li>• Check entire HPLC or IC system for rust; check resistance of all materials to the reagents in use.</li> </ul>
<b>Baseline unstable</b> 	<ul style="list-style-type: none"> <li>• Highly retarded or enriched components from the separating column.</li> <li>• Pressure fluctuations due to poor seals, blocked filters or capillaries, defective pump, etc.</li> <li>• Eluent contaminated by Fe(II) ions from rusty steel parts (traces only suffice!).</li> <li>• Defective reference electrode.</li> </ul>	<ul style="list-style-type: none"> <li>• Rinse the column well with eluent or regenerate it, use new column if necessary.</li> <li>• Check HPLC or IC system for these faults.</li> <li>• Check entire HPLC or IC system for rust; check resistance of all materials to the reagents in use.</li> <li>• Replace reference electrode.</li> </ul>

<b>Malfunction</b>	<b>Cause</b>	<b>Rectification</b>
<p><b>Noise level too high</b></p> 	<ul style="list-style-type: none"> <li>• Inadequate pulsation damping.</li> <li>• Electrostatic screening defective.</li> <li>• Background current too high.</li> <li>• Eluent outlet tubing under electrostatic influence.</li> <li>• Defective working electrode.</li> <li>• Defective 791 VA Detector.</li> </ul>	<ul style="list-style-type: none"> <li>• Use pulsation dampener.</li> <li>• Check screening, if necessary connect steel column and/or its outlet to socket <b>14</b> of the VA Detector by means of a cable.</li> <li>• Check screening, if necessary connect steel column and/or its outlet to socket <b>14</b> of the VA Detector by means of a cable.</li> <li>• Immerse eluent outlet tubing completely in spent solution in waste recipient or renew PTFE tubing if necessary.</li> <li>• Replace working electrode.</li> <li>• Check 791 VA Detector using the dummy cell (see section 4.1.4)</li> </ul>
<p><b>Spikes on baseline</b></p> 	<ul style="list-style-type: none"> <li>• Air in detector cell.</li> <li>• Air entering detector cell.</li> <li>• If a CPE working electrode is in use, the carbon paste is not in good condition.</li> </ul>	<ul style="list-style-type: none"> <li>• Unscrew working electrode and deaerate detector cell.</li> <li>• Degas eluent or subject it to appropriate treatment.</li> <li>• Refill CPE and ensure paste is well tamped down.</li> </ul>
<p><b>Isolated spikes on baseline</b></p> 	<ul style="list-style-type: none"> <li>• Electrostatic discharges from operating personnel on the apparatus.</li> <li>• Mains interference.</li> <li>• Electromagnetic interference from environment.</li> </ul>	<ul style="list-style-type: none"> <li>• Such discharges cannot be completely avoided, but are not usually too troublesome at normal (i.e. not too dry) atmospheric humidity.</li> <li>• Connect all instruments to the power supply via a suitable filter (e.g. Metrohm 615 Mains Distributor).</li> <li>• Change "environment".</li> </ul>
<p><b>Substance turnover too low or non-existent</b></p>	<ul style="list-style-type: none"> <li>• Polarization voltage too low.</li> <li>• Active surface of working electrode blocked by substances.</li> </ul>	<ul style="list-style-type: none"> <li>• Increase polarization voltage.</li> <li>• Polish working electrode or renew paste in CPE.</li> </ul>
<p><b>Substance turnover falls off</b></p>	<ul style="list-style-type: none"> <li>• Active surface of working electrode blocked by substances.</li> <li>• Quantity of substance too great.</li> </ul>	<ul style="list-style-type: none"> <li>• Polish working electrode or renew paste in CPE.</li> <li>• The sample quantity should be less than 100 ng per injection to avoid blocking of the electrode surface.</li> </ul>
<p><b>Front peak too high</b></p>	<ul style="list-style-type: none"> <li>• Environment altered by sample solution.</li> <li>• Eluent contaminated by Fe(II) ions from rusty steel parts (traces only suffice!).</li> </ul>	<ul style="list-style-type: none"> <li>• Whenever possible, use eluent as solvent for the sample solution.</li> <li>• Check entire HPLC or IC system for rust; check resistance of all materials to the reagents in use.</li> </ul>

## 4.4 Instrument test with the dummy cell

The correct functioning of the 791 VA Detector can be checked with the included 6.2813.020 Dummy cell. Several simple checks can be carried out with this extremely simplified electrical simulation of a detector cell.

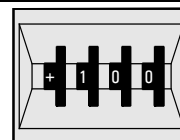
Fig. 9 shows the construction of the dummy cell. The checks described below can be carried out with the recording instrument which is connected (chart recorder, integrator, PC data recording program) or, for precise measurements, with a voltmeter which is connected to analog outputs **22** and **23**. The dummy cell is connected to the corresponding cable instead of the electrode. Each test can be carried out separately. The settings of any operating elements which are not mentioned are irrelevant.



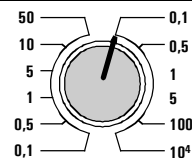
**Fig. 9:** Circuit of 6.2813.020 Dummy Cell

### 4.4.1 Check of "meas" and "damp" commutators and of current amplifier offset

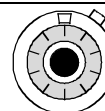
- 1** Set digital switch **11**  $U_{pol}$  to **+1000 mV**.






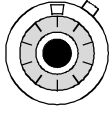



- 2** Set adjusting knob **8** to **0.1 μA**.



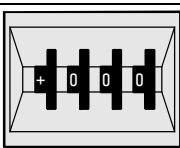
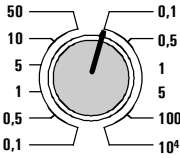
- 3** Set potentiometer **9**  $I_{comp}$  *abs.* to **0**.




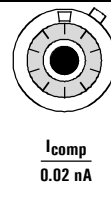
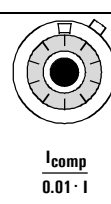
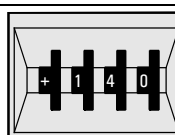
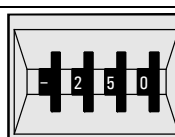


$I_{comp}$   
0.02 nA

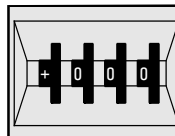
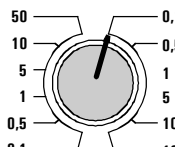
<b>4</b>	Set all commutators <b>3 ... 7</b> to " <b>off</b> " position (non-depressed position).	
<b>5</b>	Set selector <b>20</b> on the rear panel to <b>+</b> .	
<b>6</b>	Set selector <b>21</b> on the rear panel to <b>0...10 mV</b> .	
<b>7</b>	Bring recorder of voltmeter to <b>0</b> using potentiometer <b>10</b> $I_{comp}$ <i>rel.</i> . The value shown on the knob scale must not vary from 0 by more than 120 scale divisions.	 $\frac{I_{comp}}{0.01 \cdot I}$
<b>8</b>	Depress commutator <b>3</b> "meas": The recorder or voltmeter should read <b>-10 mV</b> .	
<b>9</b>	Depress commutator <b>4</b> "damp" and wait until recorder of voltmeter have attained a stable position.	
<b>10</b>	Set commutator <b>3</b> "meas" to "off" position: The time constant $\tau$ to be measured, i.e. the time which has to elapse until 63.2 % of full-scale deflection is reached, should have a value of app. 3 s (consider $\tau$ of recorder or voltmeter).	




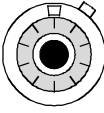
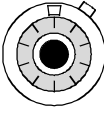
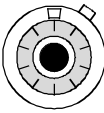
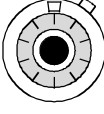

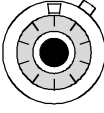
### 4.4.2 Check output voltage

<b>1</b>	Set digital switch <b>11</b> $U_{pol}$ to <b>0 mV</b> .	
<b>2</b>	Set adjusting knob <b>8</b> to <b>0.1 <math>\mu</math>A</b> .	
<b>3</b>	Set commutators <b>4 ... 7</b> to " <b>off</b> " position (non-depressed position).	

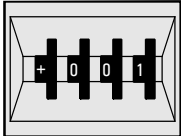
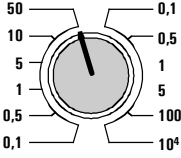


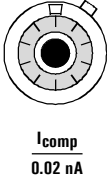
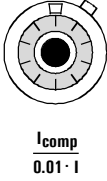

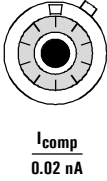
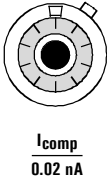
- |  |   |
|--|---|
| <p><b>4</b> Set selector <b>20</b> on the rear panel to <b>-</b>.</p>  |    |
| <p><b>5</b> Set selector <b>21</b> on the rear panel to <b>0...1 V</b>.</p>  |    |
| <p><b>6</b> Set recorder sensitivity to <b>1000 mV</b>.</p>  |   |
| <p><b>7</b> Set commutator <b>3</b> "meas" to <b>"on" position</b> (depressed position).</p>   |    |
| <p><b>8</b> Set potentiometer <b>9</b> <math>I_{comp}</math> <i>abs.</i> to <b>0</b>.</p>  |    |
| <p><b>9</b> Bring recorder of voltmeter to <b>0</b> using potentiometer <b>10</b> <math>I_{comp}</math> <i>rel.</i>.</p>   |   |
| <p><b>10</b> Alter the settings of digital switch <b>11</b> <math>U_{pol}</math> in steps of <b>100 mV</b> and <b>10 mV</b>: The settings shown on the digital switch should be reproduced on the recorder or voltmeter within a tolerance of &lt;12.5 mV and &lt;1.25 mV respectively.</p>                  |  |
| <p><b>11</b> Set digital switch <b>11</b> <math>U_{pol}</math> to <b>-</b> and alter the settings in steps of <b>100 mV</b> and <b>10 mV</b>: The settings shown on the digital switch should be reproduced on the recorder or voltmeter within a tolerance of &lt;12.5 mV and &lt;1.25 mV respectively.</p> |  |

**4.4.3 Check of current compensation**

- |   |   |
|---|---|
| <p><b>1</b> Set digital switch <b>11</b> <math>U_{pol}</math> to <b>0 mV</b>.</p> |  |
| <p><b>2</b> Set adjusting knob <b>8</b> to <b>0.1 μA</b>.</p>                     |  |

- |           |  |   |
|-----------|--|---|
| <b>3</b>  | Set selector <b>20</b> on the rear panel to <b>-</b> .   |    |
| <b>4</b>  | Set selector <b>21</b> on the rear panel to <b>0...1 V</b> .   |    |
| <b>5</b>  | Set commutator <b>3</b> "meas" to " <b>on</b> " position (depressed position).   |    |
| <b>6</b>  | Set potentiometer <b>9</b> $I_{comp}$ <i>abs.</i> to <b>0</b> .  | <br>$\frac{I_{comp}}{0.02 \text{ nA}}$   |
| <b>7</b>  | Bring recorder of voltmeter to <b>0</b> using potentiometer <b>10</b> $I_{comp}$ <i>rel.</i> .   | <br>$\frac{I_{comp}}{0.01 \cdot I}$      |
| <b>8</b>  | If potentiometer <b>10</b> $I_{comp}$ <i>rel.</i> is now moved counterclockwise by <b>100 divisions</b> (= 1 turn) then this should correspond to <b>1000 mV</b> on the recording instrument or voltmeter.   | <br>$\frac{I_{comp}}{0.01 \cdot I}$     |
| <b>9</b>  | If potentiometer <b>9</b> $I_{comp}$ <i>abs.</i> is now moved clockwise by <b>100 divisions</b> (= 1 turn) then this should correspond to the following values on the recording instrument or voltmeter:<br><br>direct: +20 mV<br>x5 button <b>5</b> depressed: +100 mV<br>x50 button <b>6</b> depressed: +1000 mV | <br>$\frac{I_{comp}}{0.02 \text{ nA}}$ |
| <b>10</b> | Set commutator <b>7</b> (+) to " <b>on</b> " position (depressed position).  |                                        |
| <b>11</b> | Similarly check the functioning of potentiometer <b>10</b> $I_{comp}$ <i>rel.</i> according to items <b>7...9</b> .  | <br>$\frac{I_{comp}}{0.01 \cdot I}$    |

4.4.4 Check of current and voltage overload

- |  |   |
|--|---|
| <p><b>1</b> Set digital switch <b>11</b> <math>U_{pol}</math> to <b>+10 mV</b> or <b>-10 mV</b>.</p>   |    |
| <p><b>2</b> Set adjusting knob <b>8</b> to <b>50 nA</b>.</p>   |    |
| <p><b>3</b> Set selector <b>20</b> on the rear panel to <b>-</b>.</p>  |    |
| <p><b>4</b> Set selector <b>21</b> on the rear panel to <b>0...1 V</b>.</p>  |    |
| <p><b>5</b> Set potentiometer <b>9</b> <math>I_{comp}</math> <i>abs.</i> to <b>0</b>.</p>  |   |
| <p><b>6</b> Set commutators <b>3 ... 7</b> to <b>"off" position</b> (non-depressed position).</p>  |   |
| <p><b>7</b> Bring recorder of voltmeter to <b>0</b> using potentiometer <b>10</b> <math>I_{comp}</math> <i>rel.</i>.</p>   |  |
| <p><b>8</b> Set commutator <b>6</b> "x50" to <b>"on" position</b> (depressed position).</p>  |  |
| <p><b>9</b> Turn potentiometer <b>9</b> <math>I_{comp}</math> <i>abs.</i> clockwise until overload display <b>2</b> lights up: The voltage at the recorder output should be app. 10.5...12 V. Without digital voltmeter, read the value on the scale of potentiometer <b>9</b>: The value should be between 500 and 620 scale divisions.</p> |  |
| <p><b>10</b> Reset potentiometer <b>9</b> <math>I_{comp}</math> <i>abs.</i> to <b>0</b>: The overload display <b>2</b> extinguishes.</p>   |  |

- 11** Set commutator **3** "meas" to "on" position (depressed position).

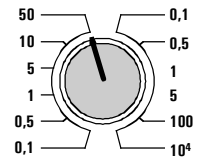


- 12** Unplug dummy cell at "AE": the overload display **2** lights up.

**4.4.5 Check sensitivity knob 8**

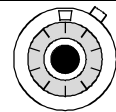
- 1** Unplug dummy cell.

- 2** Set adjusting knob **8** to **50 nA**.



- 3** Set commutators **3 ... 7** to "off" position (non-depressed position).

- 4** Set potentiometer **9**  $I_{comp\ abs.}$  to **0**.

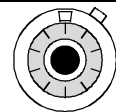


$I_{comp}$   
0.02 nA

- 5** Set selector **21** on the rear panel to **0...1 V**.

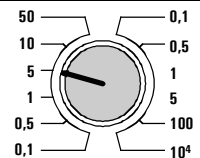


- 6** Bring recorder of voltmeter to **0** using potentiometer **10**  $I_{comp\ rel.}$  (set line recorder first to mid-scale).



$I_{comp}$   
0.01 · I

- 7** Turn adjusting knob **8** through all positions and check the deviation from zero after a waiting time of app. 5 s. Deviation should be  $< \pm 1$  V.



### 4.4.6 Check of noise from current amplifier

- 
- 1** Connect dummy cell.
- 
- 2** Set digital switch **11**  $U_{pol}$  to **0 V**.
- 
- 
- 3** Set adjusting knob **8** to **1 nA**.
- 
- 
- 4** Set commutator **3** "meas" to "**on**" position (depressed position).
- 
- 
- 5** Set commutators **4 ... 7** to "**odd**" position (non-depressed position).
- 
- 6** Set selector **21** on the rear panel to **0...1 V**.
- 
- 
- 7** Allow the recording instrument to record for a longer period of time (first adjust chart recorder to middle of the scale). Typical deflections caused by noise are about 10 mVpp or less (the 656 housing must be earthed).

# 5 Appendix

## 5.1 Technical data

### Measuring instrument

<i>Measuring technique</i>	Direct current amperometry
<i>Polarization voltage <math>U_{pol}</math></i>	Range: -1990 ... 0 ... +1990 mV Resolution: 10 mV Deviation: $\pm 1\% \pm 1$ mV
<i>Current measurement</i>	Measurement: direct, not integrated Sensitivity: 0.1, 0.5, 1, 5, 10, 50 nA 0.1, 0.5, 1, 5, 100, 10'000 $\mu$ A Deviation: $\pm 15\%$ for 0.1 nA ... 5 nA $\pm 1.5\%$ for 10 nA ... 0.1 $\mu$ A $\pm 1.0\%$ for 0.5 ... 100 $\mu$ A $\pm 4.0\%$ for 10'000 $\mu$ A Time constants: $\approx 5.0$ s for $< 0.1$ nA $\approx 0.6$ s for 0.1 ... 0.5 nA $\approx 0.3$ s for 0.5 ... 1 nA $\approx 0.1$ s for 1 ... 5 nA
<i>Damping</i>	Switchable Time constant: 1.7 s
<i>Compensation current <math>I_{comp}</math></i>	
<i>absolute</i>	Basic range: -20 ... 0 ... +20 nA Resolution: 0.02 nA Multiplication: 5 $\times$ , 50 $\times$
<i>relative</i>	Range: -5.00 ... 0 ... +5.00 $\cdot I$ Resolution: 0.01 $\cdot I$
<i>Potentiostat</i>	Input resistance: $10^{12} \Omega$ (typ. value) Input current: $< 5$ pA (typ. value) Zero-point drift: 20 $\mu$ V/K (typ. value) Output voltage: $\pm 10$ V Output current: $\leq 10$ mA

### Analog output

<i>Signal</i>	$U = f(I)$ 1 V or 10 mV DC for the full current range $I$
<i>Polarity</i>	Switchable
<i>Max. load</i>	$< 5$ mA
<i>Input resistance at connected instrument</i>	$> 2$ k $\Omega$

### Mains connection

<i>Voltage</i>	115 V: 100...120 V $\pm$ 10 % 230 V: 220...240 V $\pm$ 10 % Switchable with voltage selector insert in the fuse holder (see <i>section 2.2.1</i> )
<i>Frequency</i>	50...60 Hz
<i>Power consumption</i>	5 VA
<i>Fuse</i>	5 mm dia., 20 mm length 100...240 V: 1 A (slow-blow)

### Safety specifications

<i>Construction/testing</i>	According to IEC 1010 / EN 61010 / UL 3101-1, protection class 1, degree of protection IP40
<i>Safety directions</i>	The Instructions for Use include information and warnings which must be heeded by the user to assure safe operation of the instrument.

### Electromagnetic compatibility (EMC)

<i>Emitted interference</i>	Standards met: EN55011 (class B), EN55022 (class B), EN 50081-1/2
<i>Immunity to interference</i>	Standards met: IEC801-2/IEC1000-4-2 (class 3), IEC801-4/ IEC1000-4-4 (class 3), IEC1000-3/EN61000-3, IEC1000-4-11/EN61000-4-11

### Ambient temperature

<i>Nominal operating range</i>	+5...+40°C (at 20...80 % atmospheric humidity)
<i>Storage, transport</i>	-40...+70°C

### Housing

<i>Material of cover</i>	Polyurethane rigid foam (PUR) with fire protection for fire class UL94VO, CFC-free
<i>Material of base</i>	Steel, enameled

### Dimensions

<i>Width</i>	260 mm
<i>Height</i>	129 mm
<i>Depth</i>	366 mm
<i>Weight</i>	3.6 kg (with accessories)

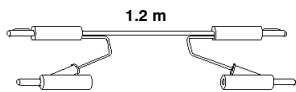
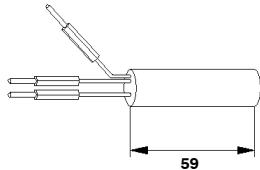
## 5.2 Standard equipment



*Subject to changes !  
All dimensions are given in mm.*

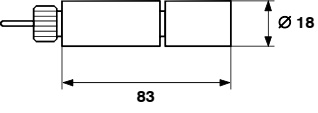
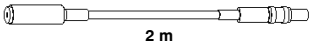
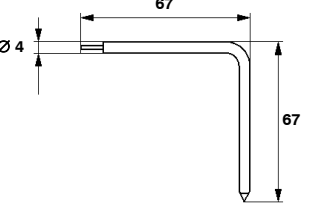
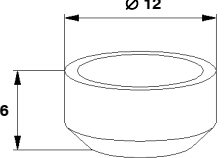
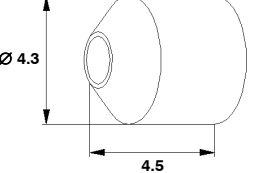
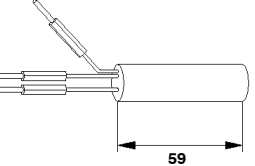
### 5.2.1 2.791.0010 VA Detector for HPLC

The 2.791.0010 VA Detector includes the following parts:

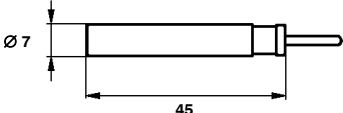
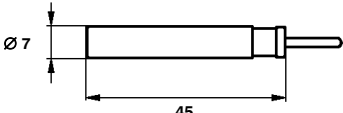
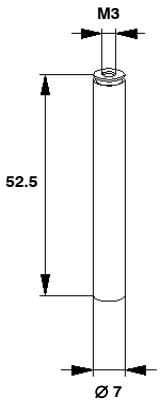

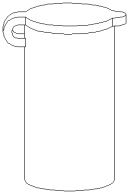
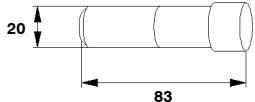
Quant.	Order No.	Description												
1	6.2105.030	<p><b>Cable</b> Connection cable 791 VA Detector (analog output) – recorder</p> 												
1	6.2122.0X0	<p><b>Mains cable</b> to customer's specifications:</p> <table style="width: 100%; border-collapse: collapse;"> <tr> <td style="text-align: center;"><u>Cable socket</u></td> <td style="text-align: center;"><u>Cable connector</u></td> <td></td> </tr> <tr> <td>Type IEC 320/C 13</td> <td>Type SEV 12 (CH...)</td> <td style="text-align: right;">6.2122.020</td> </tr> <tr> <td>Type IEC 320/C 13</td> <td>Type CEE (7), VII (D...)</td> <td style="text-align: right;">6.2122.040</td> </tr> <tr> <td>Type CEE (22), V</td> <td>Type NEMA 5-15 (USA...)</td> <td style="text-align: right;">6.2122.070</td> </tr> </table>	<u>Cable socket</u>	<u>Cable connector</u>		Type IEC 320/C 13	Type SEV 12 (CH...)	6.2122.020	Type IEC 320/C 13	Type CEE (7), VII (D...)	6.2122.040	Type CEE (22), V	Type NEMA 5-15 (USA...)	6.2122.070
<u>Cable socket</u>	<u>Cable connector</u>													
Type IEC 320/C 13	Type SEV 12 (CH...)	6.2122.020												
Type IEC 320/C 13	Type CEE (7), VII (D...)	6.2122.040												
Type CEE (22), V	Type NEMA 5-15 (USA...)	6.2122.070												
1	6.2813.020	<p><b>Dummy Cell</b> Electrical simulation of the ELCD cell for testing and checking functions</p> 												
1	8.791.1013	<p><b>Instruction for Use (English)</b> for 791 VA Detector</p>												

## 5.2.2 2.791.0020 VA Detector for IC

The 2.791.0020 VA Detector includes the following parts:

Quant.	Order No.	Description	
1	6.0727.000	<b>Reference electrode</b> Mini reference electrode for ELCD cell with Ag/AgCl/c(KCl) = 3 mol/L reference system; including cap	
3	6.2120.020	<b>Electrode cable</b> Electrode cable with mini socket for connection pins 2 mm and mini plug type F	
1	6.2122.0X0	<b>Mains cable</b> to customer's specifications: <u>Cable socket</u> <u>Cable connector</u> Type IEC 320/C 13                  Type SEV 12 (CH...) .....6.2122.020 Type IEC 320/C 13                  Type CEE (7), VII (D...) .....6.2122.040 Type CEE (22), V                    Type NEMA 5-15 (USA...).....6.2122.070	
1	6.2617.000	<b>Tool for PTFE gasket</b> To remove 6.2704.010 and 6.2704.020 PTFE gaskets	
4	6.2704.010	<b>PTFE gasket</b> Gasket for reference electrode, working electrode and eluent inlet	
3	6.2704.020	<b>PTFE gasket</b> Gasket for eluent inlet	
1	6.2813.020	<b>Dummy Cell</b> Electrical simulation of the ELCD cell for testing and checking functions	
1	6.5303.030	<b>ELCD cell</b>	
1	8.791.1013	<b>Instruction for Use (English)</b> for 791 VA Detector	

### 5.3 Optional accessories

Order No.	Description																
6.0805.010	<b>Mini Glassy Carbon electrode</b> Diameter of active zone: 2.8 mm.																
6.0807.000	<b>Mini Carbon paste electrode</b> Diameter of active zone: 3 mm.																
6.1204.XXX	<b>Electrode tips for working electrode</b> Together with 6.2103.110 Contact pin forms the working electrode. The following electrode tips are available:  <table border="0"> <thead> <tr> <th>Order No.</th> <th>Disk material</th> <th>Shaft material</th> </tr> </thead> <tbody> <tr> <td>6.1204.100</td> <td>Ultra Trace Graphite</td> <td>PVC</td> </tr> <tr> <td>6.1204.120</td> <td>Pt</td> <td>PEEK</td> </tr> <tr> <td>6.1204.130</td> <td>Ag</td> <td>PEEK</td> </tr> <tr> <td>6.1204.140</td> <td>Au</td> <td>PEEK</td> </tr> </tbody> </table> Disk diameter: 2.0 +0 / -0.05 mm	Order No.	Disk material	Shaft material	6.1204.100	Ultra Trace Graphite	PVC	6.1204.120	Pt	PEEK	6.1204.130	Ag	PEEK	6.1204.140	Au	PEEK	
Order No.	Disk material	Shaft material															
6.1204.100	Ultra Trace Graphite	PVC															
6.1204.120	Pt	PEEK															
6.1204.130	Ag	PEEK															
6.1204.140	Au	PEEK															
6.2103.110	<b>Contact pin</b> Together with 6.1204.1X0 Electrode tip forms the working electrode.																
6.2801.020	<b>Carbon paste</b> Carbon paste for 6.0807.000 Mini carbon paste electrode																
6.2826.000	<b>Filling tool</b> Filling tool for 6.0807.000 Mini carbon paste electrode																

## 5.4 Warranty and conformity

### 5.4.1 Warranty

The warranty on our products is limited to defects that are traceable to material, construction or manufacturing error which occur within 12 months from the day of delivery. In this case, the defects will be rectified in our workshops free of charge. Transport costs are to be paid by the customer.

For day and night operation, the warranty is limited to 6 months.

Glass breakage in the case of electrodes or other parts is not covered by the warranty. Checks which are not a result of material or manufacturing faults are also charged during the warranty period. For parts of outside manufacture insofar as these constitute an appreciable part of our instrument, the warranty stipulations of the manufacturer in question apply.

With the regard to the guarantee of accuracy, the technical specifications in the instruction manual are authoritative.

Concerning defects in material, construction or design as well as the absence of guaranteed features, the orderer has no rights or claims except those mentioned above.

If damage of the packaging is evident on receipt of a consignment or if the goods show signs of transport damage after unpacking, the carrier must be informed immediately and a written damage report demanded. Lack of an official damage report releases Metrohm from any liability to pay compensation.

If any instruments and parts have to be returned, the original packaging should be used if at all possible. This applies above all to instruments, electrodes, burette cylinders and PTFE pistons. Before embedment in wood shavings or similar material, the parts must be packed in a dust-proof package (for instruments, use of a plastic bag is imperative). If open assemblies are enclosed in the scope of delivery that are sensitive to electromagnetic voltages (e.g. data interfaces etc.) these must be returned in the associated original protective packaging (e.g. conductive protective bag). (Exception: assemblies with built-in voltage source belong in a non-conductive protective packaging). For damage which arises as a result of non-compliance with these instructions, no warranty responsibility whatsoever will be accepted by Metrohm.

**5.4.2 EU Declaration of conformity**



**EU Declaration of Conformity**

The METROHM AG company, Herisau, Switzerland hereby certifies, that the instrument:

**791 VA Detector**

meets the requirements of EC Directives 89/336/EEC and 73/23/EEC.

**Source of the specifications:**

- |              |   |
|--------------|---|
| EN 50081-1/2 | Electromagnetic compatibility, basic specification; Emitted Interference        |
| EN 61010     | Safety requirements for electrical laboratory measurement and control equipment |

**Description of the instrument:**

High-sensitivity current measuring instrument for electrochemical detection in HPLC and IC.

Herisau, September 11, 1998

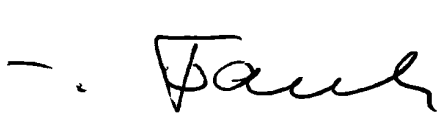

Dr. J. Frank

Ch. Buchmann

Development Manager

Production and Quality Assurance Manager

### 5.4.3 Certificate of conformity and system validation

<b>Certificate of Conformity and System Validation</b>	
This is to certify the conformity to the standard specifications for electrical appliances and accessories, as well as to the standard specifications for security and to system validation issued by the manufacturing company.	
Name of commodity:	<b>791 VA Detector</b>
Name of manufacturer:	Metrohm Ltd., Herisau, Switzerland
Principal technical information:	Voltages: 100...120, 220...240 V Frequency: 50...60 Hz
This Metrohm instrument has been built and has undergone final type testing according to the standards:  IEC801-2/IEC1000-4-2 (class 3), IEC801-4/IEC1000-4-4 (class 3), IEC1000-3/EN 61000-3, IEC1000-4-11/EN 61000-4-11, EN55011 (class B), EN55022 (class B), EN50081-1/2 <div style="text-align: right;"><i>— Electromagnetic compatibility</i></div> IEC1010, EN61010, UL3101-1 <div style="text-align: right;"><i>— Security specifications</i></div> The technical specifications are documented in the instruction manual.	
Metrohm Ltd. is holder of the SQS-certificate of the quality system ISO 9001 for quality assurance in design/development, production, installation and servicing.	
Herisau, September 11, 1998	
 	
Dr. J. Frank	Ch. Buchmann
Development Manager	Production and Quality Assurance Manager

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