945 Professional Detector Vario
IC Amperometric Detector

Amperometric detection for determining
electroactive components

Metrohm

Amperometric detection: the answer for complex analytical tasks

Amperometric detectors are an alternative to conductivity and UV/VIS detectors. They may always be used when electroactive – i.e., oxidizable or reducible – components are to be determined. The IC Amperometric Detector excels by its high selectivity and measurement sensitivity. It can be used for sensitive and accurate determination of concentrations down to the ng/L range.

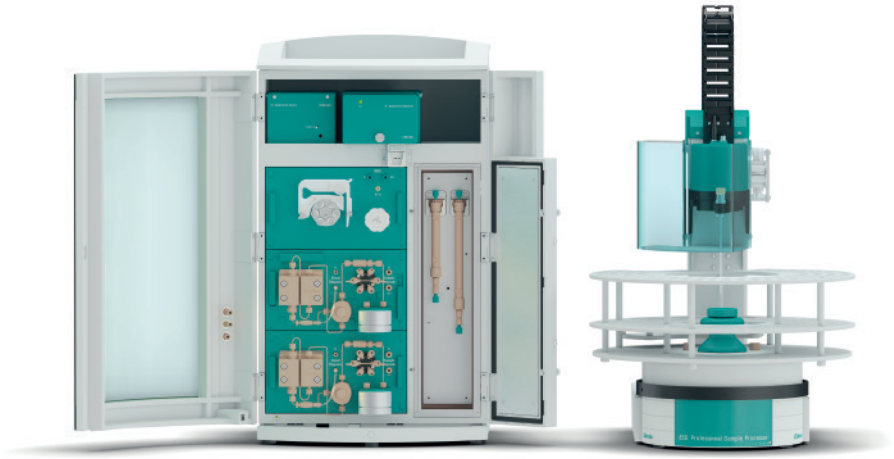
The amperometric detector is available as a detector block (the **IC Amperometric Detector**) and as a stand-alone detector (the **945 Professional Detector Vario**). Both instruments can be operated either with a 940 Professional IC Vario or with a 930 Compact IC Flex. Control, data recording, and data evaluation are carried out by the proven ion chromatography software MagIC Net.

The user-friendly measuring cells are intelligent, require little maintenance, and are optimized for the various applications. Their three-electrode configuration ensures remarkably low noise levels and excellent signal strength. Depending on the particular application, the instrument can be operated in direct current mode (DC), in pulsed amperometric mode (PAD), in flexible integrated pulsed amperometric mode (flexIPAD), or in cyclic voltammetric mode (CV). The flexibility of the detector includes the choice of measurement signal: no matter whether current or charge is selected, users always obtain a reliable result.

The amperometric detector of the Professional IC Vario generation: irrespective of whether the 945 Professional Detector Vario or the IC Amperometric Detector is used, the amperometric detector can be incorporated in any Metrohm IC system and helps users to perform even the most demanding analytical tasks.

Highlights

- Amperometric detector for determining electroactive components
- Flexible setup as stand-alone detector or detector block for IC and HPLC
- Outstanding selectivity due to different measuring modes: DC, PAD, flexIPAD, CV
- High sensitivity through excellent signal/noise ratio
- Large measuring range for demanding applications
- Easy to use due to intelligent and robust measuring cells with wall-jet and thin-layer design
- Large variety of working electrodes: Au, Ag, Pt, Cu, GC
- Different maintenance-free reference electrodes
- Very fast start-up without long conditioning times
- Leak sensor in the cell compartment
- Fully compliant with Metrohm Inline Sample Preparation techniques
- Control and monitoring by MagIC Net – the proven software for ion chromatography



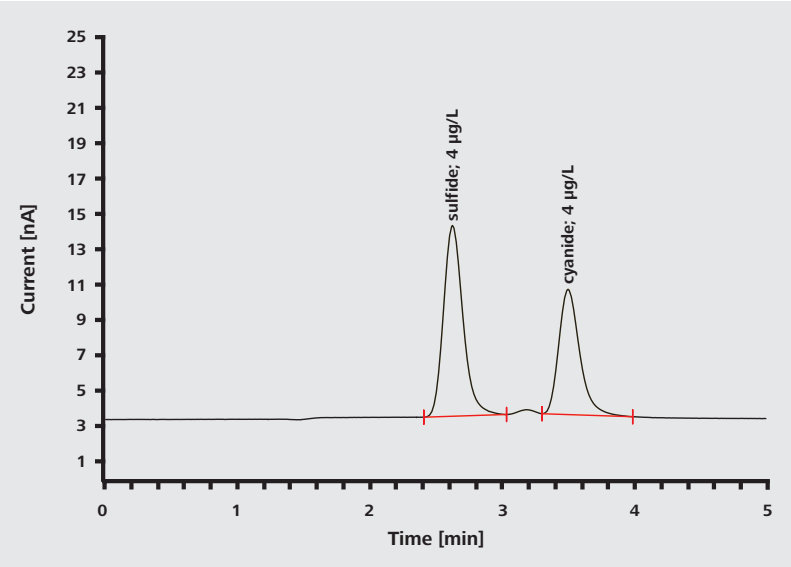
Direct current amperometry: DC mode

The most well-known method of amperometric measurement is direct current amperometry, which is also known as DC mode. It is used primarily to determine inorganic anions, but can also be used to determine organic substances such as phenols or catecholamines. When a constant working potential is applied between the working electrode and the reference electrode, the analytes are, for example, oxidized. Depending on their concentration, a current flows to the auxiliary electrode, which provides the measurement signal. In DC mode it is possible to measure without a specific range. For this, auto range is available.

DC amperometry is a highly sensitive analytical method that is characterized by detection limits in the lowest ng/L range. Another outstanding feature is its high selectivity, which enables matrix effects to be suppressed in the chromatogram, so reliable analysis can be carried out successfully even in the most difficult sample matrices (e.g., glacial acetic acid, urine, wastewater). To ensure optimum analysis of samples, Metrohm offers a wide selection of working electrode materials: gold (Au), silver (Ag), platinum (Pt), copper (Cu), and glassy carbon (GC). Complete cell equipments with an intelligent measuring cell, including the permanently installed auxiliary electrode, suitable working electrode, and reference electrode, are also available to users for the standard DC mode applications, such as cyanide determination, and for the analysis of inorganic anions.

Typical DC applications

- Anions, such as cyanide, sulfide, nitrite, sulfite, thiosulfate, iodide, chlorite, hypochlorite, bromide, arsenite, ...
- Cations such as amines and aromatic amino acids, ...
- Organic substances such as phenols, catecholamines, ascorbic acid, alcohols, vitamins, ...



Fast, extremely sensitive (detection limits below 10 ng/L), and reliable determination of cyanide and sulfide is possible with DC amperometry. Analysis of a standard solution containing 4 µg/L cyanide and 4 µg/L sulfide; column: Metrosep A Supp 10 - 100/2.0; eluent: 100 mmol/L NaOH + 7 µmol/L EDTA, flow: 0.25 mL/min; column temperature: 35 °C; detector: DC mode, WE: Ag, RE: Ag/AgCl, working potential: 0 V, temperature: 35 °C; sample volume: 20 µL

Pulsed amperometry: PAD mode

Pulsed amperometry is used when analytes form a deposit on the surface of the working electrode as a result of the electrochemical reaction, making detection in DC mode impossible. In PAD mode, a constant working potential is applied at the beginning. However, unlike in DC amperometry, the surface of the working electrode is then cleaned continuously through the application of cleaning potentials. A total of up to 40 different potentials can be applied for this «potential wave». In this way, the application can be optimized, so there is always a fresh working electrode surface available. Robust and reliable detection is guaranteed.

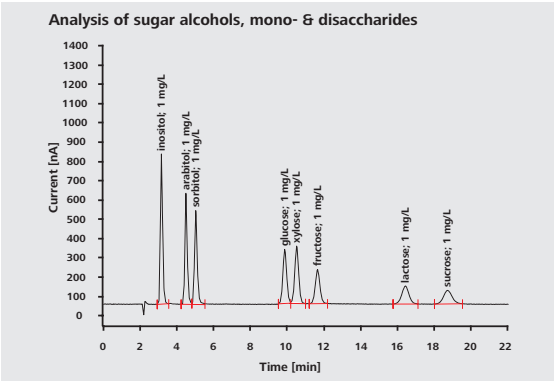
PAD mode is used primarily to detect carbohydrates and their derivatives. Multiple potential profiles make it possible to find the optimum working conditions for each analyte.

In PAD mode, all working and reference electrodes are available to the user. Specifically for carbohydrate analysis, however, Metrohm offers a complete cell equipment comprising intelligent measuring cell, gold working electrode, palladium reference electrode, and appropriate accessories.

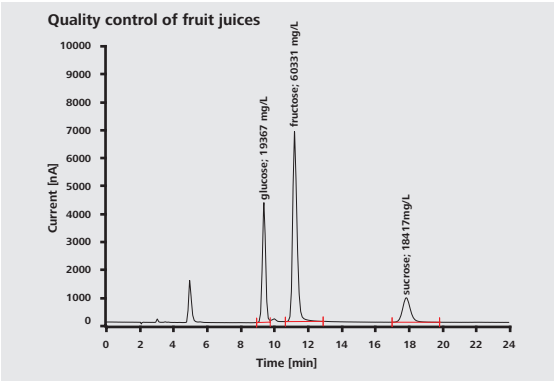
Typical PAD applications

- Sugars, e.g., mono-, di-, oligo-, and polysaccharides ...
- Sugar alcohols, polyols, glycols, aliphatic alcohols, ...
- Sugar amines, e.g., glucosamine, galactosamine, ...
- Anhydrosugars, e.g., levoglucosan, galactosan, mannosan, ...
- Sugar acids, e.g., glucuronic acid, gluconic acid, sialic acids, ...
- Substituted sugar compounds, e.g., glucose-6-phosphate, 2-fluoro-2-deoxy-D-glucose, ...
- Amino acids

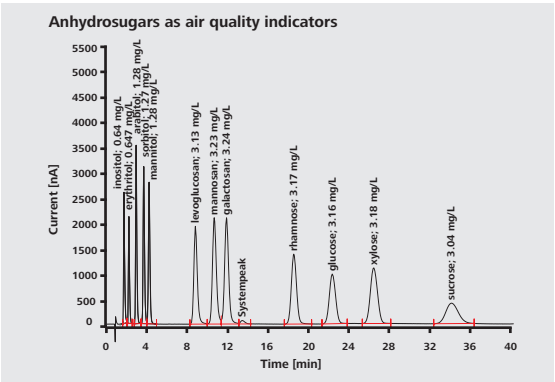
All separations are achieved on Metrosep Carb 2 - 150/4.0 column



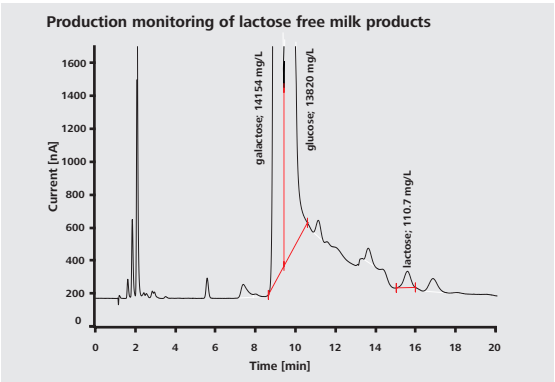
Eluent: 100 mmol/L sodium hydroxide / 10 mmol/L sodium acetate, column temperature: 30 °C; sample volume: 20 µL, flow rate: 0.5 mL/min, amperometric detector: PAD mode, WE: Au, RE: Pd, working potential: 50 mV



Eluent: 100 mmol/L sodium hydroxide / 10 mmol/L sodium acetate, column temperature: 30 °C; sample volume: 20 µL, flow rate: 0.5 mL/min, amperometric detector: PAD mode, WE: Au, RE: Pd, working potential: 50 mV, dilution 1:1000



Eluent: 10 mmol/L sodium hydroxide, column temperature: 45 °C; sample volume: 100 µL, flow rate: 1.0 mL/min, amperometric detector: PAD mode, WE: Au, RE: Pd, working potential: 50 mV



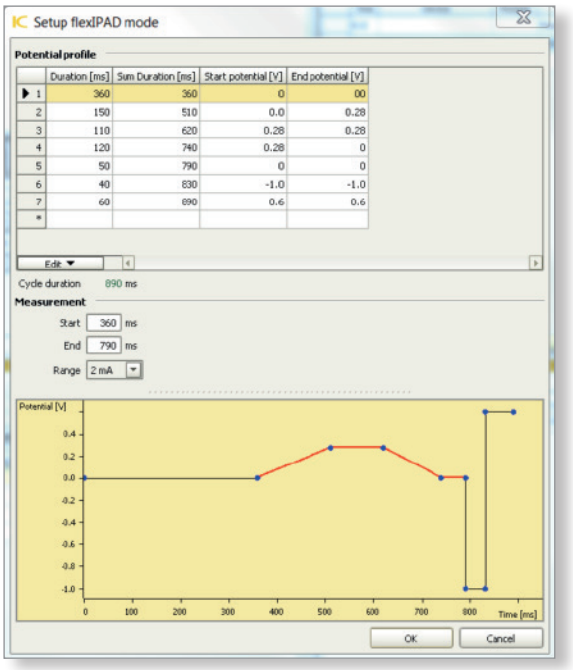
Eluent: 5 mmol/L sodium hydroxide / 2 mmol/L sodium acetate, column temperature: 40 °C; sample volume: 20 µL, flow rate: 0.8 mL/min, amperometric detector: PAD mode, WE: Au, RE: Pd, working potential: 50 mV. Sample: diluted 1:100, spiked with 100 mg/L lactose

Flexible integrated pulsed amperometry: flexIPAD mode

06

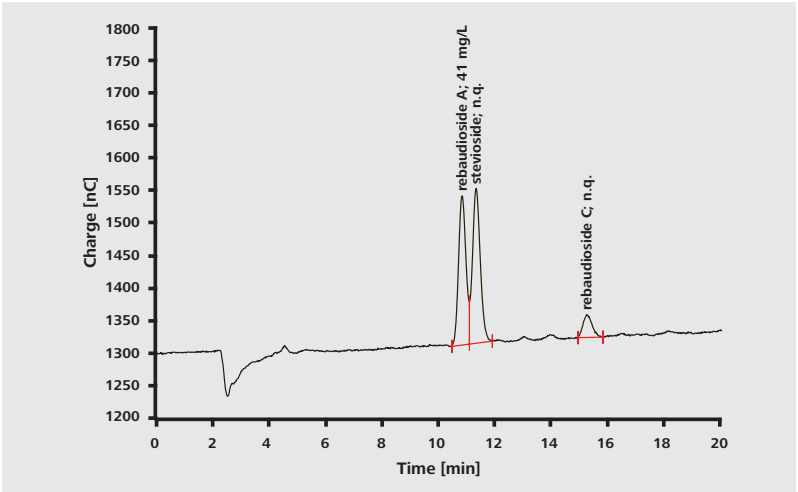
In flexIPAD mode the focus is entirely on flexibility. This enables a potential-time pattern to be preset at will. Up to 40 constant potentials (steps) and linear potential changes (ramps) can be combined. Whatever the requirements of the application are, they can be fulfilled in this working mode. The range over which the measurement signal is to be integrated can be defined at the user's discretion. Either charge or current can be chosen as the measurement signal. The fast switches of the amperometric detector enable the potential to be applied very rapidly. Consequently there is hardly any need for conditioning steps; the potential wave can be made short, so a higher data rate is possible.

The range of uses of the flexIPAD mode comprises all applications of the PAD mode and a few more besides, including the determination of sulfur-containing analytes, for example, in electroplating baths, in pharmaceutical products, or in food and beverage. In addition, the flexIPAD mode is ideal for research applications owing to the freedom one has to choose the potential profile.



Complete flexibility in the creation of the potential wave of the sweetener Stevia (see chromatogram below).

Typical flexIPAD applications
• Carbohydrates and their derivatives, sweeteners
• Amino acids and sugar amines
• Organosulfur compounds
• Antibiotics
• Biogenic amines
• Research applications

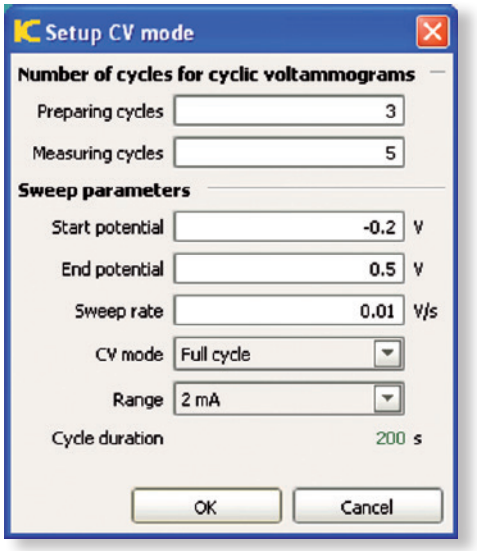


Glycoside analysis in sweetener – flexIPAD mode. Noncaloric stevia glycosides are widely used in food and beverages to replace calorie-rich sugars. One of the main stevia glycosides is rebaudioside A. The chromatogram shows the rebaudioside and stevioside peaks detected in a highly diluted sweetener sample (41 mg/L). Column: Luna 5 µm C18, 250 × 4.6 mm (Phenomenex); eluent: 10 mmol/L NaH_2PO_4, pH = 4.5 / acetonitrile 70:30 (v/v), flow: 1.0 mL/min; column temperature: 40 °C; PCR solution: 400 mmol/L NaOH, 0.3 mL/min; detector: flexIPAD mode (see Fig. above), WE: Au, RE: Pd, working potential: -1.0 to 0.6 V, temperature: 35 °C; sample volume: 20 µL

Cyclic voltammography: CV mode

The method development for determining unknown analytes is complex, especially when work is done using PAD or flexIPAD mode. CV mode helps the user to meet this challenge. Through interpretation of a cyclic voltammogram it is possible to arrive at the optimum working potential for the particular application. The cyclic voltammogram also shows the potentials with which the surface of the working electrode can be oxidized and reduced, in order to achieve continuous cleaning. In this way the optimum potential profile for each analyte can be discovered.

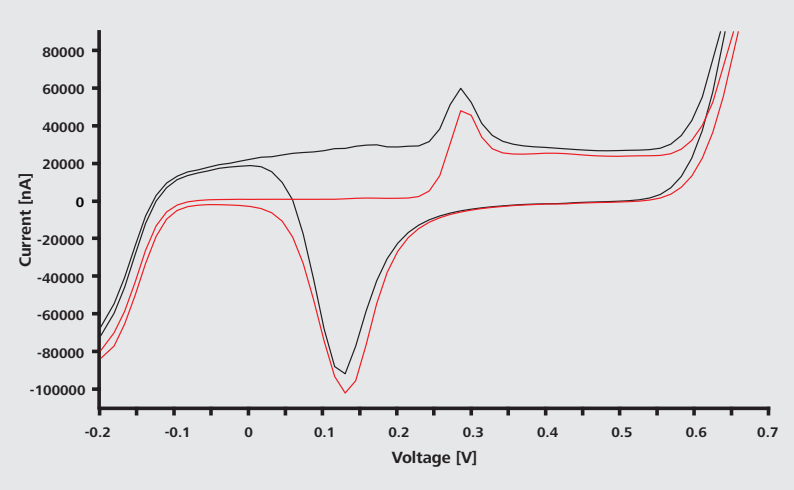
In addition, working, reference, and auxiliary electrodes can be characterized with the help of cyclic voltammograms. Changes over a prolonged period can be documented. Furthermore, it is possible to read off from cyclic voltammograms the potential range within which work can be done (depending on the electrode configuration being used). Thus users can get a better understanding of the electrochemical reactions under the respective application conditions.



A multiple number of cyclic voltammograms in combination with a few preparing cycles guarantees that one actually measures what one is supposed to measure. Artifacts and interferences can be ruled out. Variable settings are possible for the start and end potential, the sweep rate, the measurement range, and also whether full or half cycles should be scanned.

Typical CV applications

- Determination of the optimum working potential
- Determination of regeneration potentials for PAD and flexIPAD modes
- Characterization of electrodes



In CV mode voltammograms are recorded that show the resulting current as a function of the working potential. This can also be done in a peak – during chromatographic separation. Thus, the electrochemical reaction in the cell can be followed exactly as it takes place during detection. The example shows a cyclic voltammogram of glucose in eluent (black CV) by comparison with pure eluent (red CV).

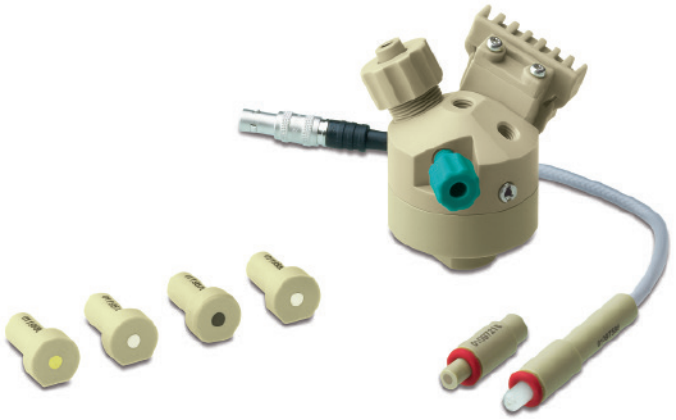
Intelligent measuring cells as the key to success

Intelligent IC stands for self-monitoring measuring systems, minimization of operating errors, and traceability of the measurement results from sample preparation, through separation and on to detection. In practice, that means reliable and simple operation. The intelligent measuring cells of the amperometric detector are a perfect example. The systems offer the following advantages:

- Automatic recognition of the cells in the system
- Traceability of data to the measuring cell used
- Monitoring of the working and reference electrodes on the basis of operating hours

The intelligent measuring cells are resistant to pressure, require little maintenance, and are optimized for ion chromatography applications. Depending on the application, users can choose between wall-jet cell or thin-layer cell. The closed cell compartment, the electrical shielding, and the thermostatic control also minimize the signal noise. Moreover, the cell compartment is equipped with a leak sensor, so the analytical system is shut down immediately in the event of any escape of liquids. Each cell has a purge connection that ensures easy venting.

The measuring cells are easy to use and equipped with three electrodes: working, reference, and auxiliary electrodes. As the working electrode, a number of 2-mm- and 3-mm-versions are available in the usual materials, namely gold, silver, platinum, copper, and glassy carbon, depending on the particular application. The working electrode can be replaced at any time and is so rugged that it can be used for months or even years. The precision and repeatability of the measurements are impressive. Maintenance-free auxiliary and reference electrodes complete the detection system. Solid-phase palladium electrodes with long-term stability and Ag/AgCl gel electrodes (filled with saturated KCl) are available as reference electrodes. A stainless steel auxiliary electrode is a standard component of every cell.



The intelligent measuring cells and their working and reference electrodes – a perfect team for your application

Typical applications of the different working electrodes

Working electrode	Application
Au	• Mono-, di-, oligo-, and polysaccharides, sugar alcohols, sugar amines, sugar acids • Amino acids • Antibiotics
Ag	• Halogen anions, e.g., bromide, iodide • Cyanide and sulfide • Thiosulfate • Pharmaceuticals
Pt	• Alcohols and glycols • Hydrazines • Arsenite and hypochlorite
Cu	• Mono-, di-, oligo-, and polysaccharides, sugar alcohols
GC	• Inorganic anions, e.g., nitrite, sulfite, iodide • Phenols • Catecholamines and aromatic amines • Vitamins

The right system for every challenge

Metrohm offers the amperometric detector in the form of a detector block (IC Amperometric Detector) and as a stand-alone detector (945 Professional Detector Vario). These two instruments can be used both with 940 Professional IC Vario or with 930 Compact IC Flex. Depending on the task at hand, measurement can be performed in series or in parallel with other analytical channels. System configurations with gradient elution, with inline eluent preparation, with microbore columns, and inline sample preparation techniques (MISP) are also possible. Such systems are controlled and the data are collected and evaluated by MagIC Net ion chromatography software.

The stand-alone detector 945 Professional Detector Vario is a versatile instrument that is offered in three different versions: only as an amperometric detector (945 Professional Detector Vario – Amperometry), only as a conductivity detector (945 Professional Detector Vario – Conductivity), and as a combination of both detectors in a single instrument (945 Professional Detector Vario Conductivity & Amperometry). The 945 Professional Detektor Vario is the interface for a number of optional peripheral devices as for example, 942 Extension Module Vario, 800 Dosino, 891 Professional Detector Out, or USB devices.

Due to the wide variety of possible configurations with the IC Amperometric Detector and the 945 Professional Detector Vario it is not only possible to equip IC systems with additional analytical features, but also to upgrade HPLC systems to an ion chromatograph.

As a detector block or as a stand-alone detector – amperometric detection provides the solution for both selective and sensitive analytical tasks.

Technical information

	IC Amperometric Detector 2.850.9110	945 Professional Detector Vario – Amperometry 2.945.0020
Design	Detector block	Stand-alone detector
Type of detector	Microprocessor-controlled digital signal processing	
Detection modes	DC, PAD, flexIPAD, CV	
Potential range	-5.0 to +5.0 V in 0.001 V steps	
Potential profile	DC mode: 1 potential PAD mode: 40 potentials flexIPAD mode: 40 potentials (steps and ramps)	
Data output channels	Current and charge	
Digital signal range	DC mode: 0.00012 pA to 2 mA, autorange PAD mode: 0.012 pA to 2 mA flexIPAD mode: 0.12 pC to 200 µC CV mode: 0.12 pA to 20 mA	
Electronic noise	DC mode: < 5 pA PAD mode: < 10 pA flexIPAD mode: < 30 pC	
Control, data collection, and data evaluation	MagIC Net 3.1 Compact, Professional and Multi	
Detector temperature	Temperature stability better than 0.05 °C Temperature range: ambient temperature +8 °C to 80 °C	
Liquid-carrying parts	Free of metal	
System readiness	• Automatic function test upon startup • Leak sensor • Monitoring of temperature stability	
Peripheral devices	Can be installed in 940, 930 standard component of 2.945.0020 and 2.945.0030	Can be combined with 940, 930, 883, 944, and 945
Installation	Dedicated and multiple detection (parallel and in series) possible	
Sample preparation and liquid handling	Can be combined with all inline sample preparation techniques (MISP) and injection techniques (full-loop, internal loop, MiPT, MiPuT)	
Analog data output	Via peripheral devices	Possible directly with 891 Professional Analog Out
Measuring cell management	Intelligent measuring cells with automatic recognition and monitoring functions	
Cell geometry	Wall-jet cell and thin-layer cell	
Cell housing	PEEK body purge outlet	
Working electrodes	Replaceable gold, silver, platinum, copper, and glassy carbon working electrodes (2 and 3 mm diameter)	
Reference electrodes	Replaceable solid-phase Pd electrode and Ag/AgCl reference electrodes	
Auxiliary electrode	Stainless steel auxiliary electrode incorporated in the measuring cell	
Cell volume	< 0.1 µL wall-jet cell (with 2 mm working electrode and 25 µm spacer)	

Ordering information

	Detectors
2.850.9110	IC Amperometric Detector
2.945.0010	945 Professional Detector Vario – Conductivity
2.945.0020	945 Professional Detector Vario – Amperometry
2.945.0030	945 Professional Detector Vario – Conductivity & Amperometry
	Equipment with intelligent cell, 25 and 50 µm spacers and accessories
6.5337.000	IC equipment wall-jet cell
6.5337.010	IC equipment wall-jet cell for carbohydrate analysis*
6.5337.020	IC equipment wall-jet cell for cyanide analysis**
6.5337.030	IC equipment wall-jet cell for anion analysis***
6.5337.200	IC equipment thin-layer cell
	Cells and electrodes
6.1257.010	Wall-jet cell (without accessories)
6.1257.100	Thin-layer cell (without accessories)
6.1257.210	Au working electrode (3 mm)
6.1257.220	GC working electrode (3 mm)
6.1257.230	Pt working electrode (3 mm)
6.1257.240	Ag working electrode (3 mm)
6.1257.260	Au working electrode (2 mm)
6.1257.270	Cu working electrode (2 mm)
6.1257.720	Ag/AgCl reference electrode
6.1257.740	Pd reference electrode
	Other accessories
6.1257.810	50 µm spacers for wall-jet cell (3 pieces)
6.1257.820	50 µm spacer for thin-layer cell (3 pieces)
6.1257.830	25 µm spacers for wall-jet cell (3 pieces)
6.1257.840	25 µm spacer for thin-layer cell (3 pieces)
6.1257.500	Connecting cable for cell (set with 3 cables)
6.2061.100	Bottle holder for Professional IC instruments
6.2061.110	Tray with leak sensor for Professional IC instruments
6.2802.200	Maintenance set for 6.1257.2XX working electrodes
6.2802.210	Polishing disks for 6.2802.200 maintenance set
6.2813.040	Dummy cell

* with Au working electrode (3 mm) and Pd reference electrode
** with Ag working electrode (3 mm) and Pd reference electrode
*** with GC working electrode (3 mm) and Ag/AgCl reference electrode

www.metrohm.com

Subject to change without prior notice
Layout by Ecknauer+Schoch ASW, printed by Metrohm AG, CH-9100 Herisau, Switzerland
8.945.5001EN – 2016-01

Metrohm