## 896 Professional Detector 850 IC Amperometric Detector



Amperometric detection for determining electroactive components



# Amperometric detection: the answer for complex analytical tasks

Metrohm complements its portfolio of intelligent ion chromatography instruments with an amperometric detector. As an alternative to conductivity and UV/VIS detectors it may always be used when electroactive – i.e. oxidizable or reducible – components are to be determined. The new 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 new amperometric detector is available as an detector block (the **850 IC Amperometric Detector**) and as a stand-alone detector (the **896 Professional Detector**). Both instruments can be operated either with an 850 Professional IC or with an 881 or 882 Compact IC. Control, data recording and data evaluation are carried out by the proven ion chromatography software MagIC Net<sup>™</sup>.

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 generation: irrespective of whether the 896 Professional Detector or the 850 IC Amperometric Detector is used, the new 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, 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  $Net^{TM}$  the proven software for ion chromatography



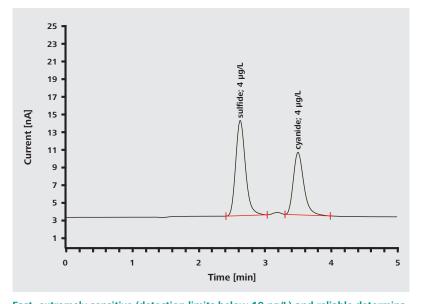
## Direct current amperometry: DC mode

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) 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, 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

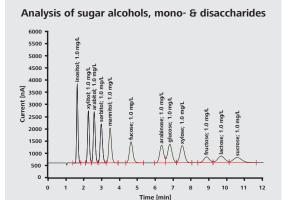
## Pulsed amperometry: PAD mode

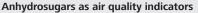
Pulsed amperometry is used when analytes form a deposit on the surface of the working electrode as a result of the electrochemical reaction, making detection in DC mode impossible. In PAD mode a constant working potential is again applied. However, unlike in DC amperometry, the surface of the working 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.

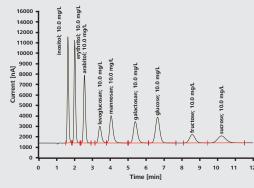
PAD mode is used primarily to detect carbohydrates and their derivatives. The applications can be found in all sectors: in water analysis and environmental analysis, in the pharmaceutical and food industries, in forensic analysis, for the quality control of biofuels and cosmetics, and in the life science field. 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 also offers a complete cell equipment comprising intelligent measuring cell, gold working electrode, palladium reference electrode and appropriate accessories.

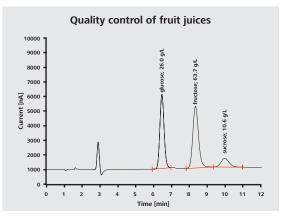
### **Typical PAD applications**

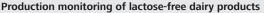
- Sugars, e.g. mono-, di-, oligo- and polysaccharides ...
- Sugar alcohols, polyols, glycols, aliphatic alcohols, ...
- Sugar amines, e.g. glucosamine, galactosamine, ...
- Anhydrosugars, e.g. levoglucosan, galactosan, mannosan, ...
- Sugar acids, e.g. glucuronic acid, gluconic acid, sialic acids, ...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

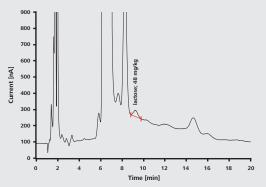










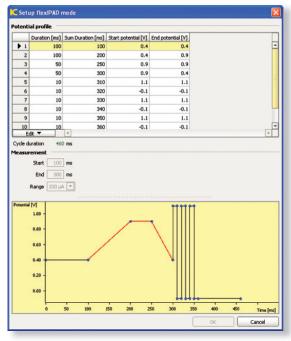


**Determination of sugar components in different matrices;** column: Metrosep Carb 1 - 150/4.0; eluent: 100 mmol/L NaOH, 1.2 mL/min; column temperature: 35 °C; detector: PAD mode, WE: Au, RE: Pd, working potential: 0.05 V, temperature: 35 °C; sample volume: 20 µL

# Flexible integrated pulsed amperometry: flexIPAD mode

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.

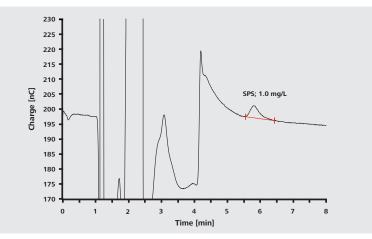
The range of uses of the flexIPAD mode comprises all applications of the PAD mode and a few more besides, including the determination of sulfur-containing analytes of the type present, for example, in electroplating baths or in pharmaceutical products. In addition, the flexIPAD mode is ideal for research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



**Complete flexibility in the creation of the potential wave.** The example shows the potential profile for the determination of the brightener SPS (see chromatogram below).

### Typical flexIPAD applications

- Carbohydrates and their derivatives
- Amino acids and sugar amines
- Organosulfur compounds
- Antibiotics
- Biogenic amines
- Research applications



### The determination of sulfur-containing brightener in copper baths, e.g. for process control, is easy with the flexIPAD

**mode.** 100 µg/L SPS (bis-(sodium sulfopropyl)-disulfide) in 1:10 diluted copper bath with 60 g/L CuSO<sub>4</sub> x 5 H<sub>2</sub>O + 238 g/L H<sub>2</sub>SO<sub>4</sub> + 82 mg/L NaCl + 20 mL/L suppressor analyzed with a 2-D IC configuration with inline cation removal: 1<sup>st</sup> stage: column: Metrosep A Supp 5 - 100/4.0; eluent: 7 mmol/L Na<sub>2</sub>CO<sub>3</sub> + 3 mmol/L NaHCO<sub>3</sub>, 0.7 mL/min; column temperature: 30 °C; sample volume: 20 µL; 2<sup>nd</sup> stage (without preconcentration column): column: Metrosep A Supp 5 - 50/4.0; eluent: 9 mmol/L Na<sub>2</sub>CO<sub>3</sub> + 3 mmol/L H<sub>2</sub>SO<sub>4</sub> + 10% acetone, 0.8 mL/min; column temperature: 30 °C; detector: flexIPAD mode, WE: Au, RE: Ag/AgCl, working potential: 0.4 - 0.9 V (see Fig. above), temperature: 35 °C

## 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.

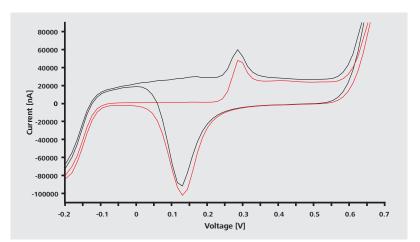
In addition, working, reference and auxiliary electrodes can be characterized with the help of cyclic voltammograms. Changes over a prolonged period can be documented. Furthermore, it is possible to read off from cyclic voltammograms the potential range within which work can be done (depending on the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

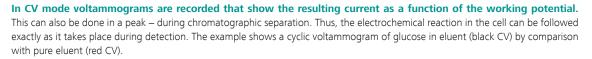
| Number of cycles for cyclic voltammograms — |  |  |  |  |
|---|--|--|--|--|
| 3   |  |  |  |  |
| 5   |  |  |  |  |
|   |  |  |  |  |
| -0.2 V                                      |  |  |  |  |
| 0.5 V                                       |  |  |  |  |
| 0.01 V/s                                    |  |  |  |  |
| -   |  |  |  |  |
| -   |  |  |  |  |
| 200 s                                       |  |  |  |  |
| Cancel                                      |  |  |  |  |
|   |  |  |  |  |

A multiple number of cyclic voltammograms in combination with a few preparing cycles 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**

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





## 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-mmand 3-mm-versions are available in the usual materials, namely gold, silver, platinum and glassy carbon (GC), 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 KCI) 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   | <ul> <li>Mono-, di-, oligo- and polysaccharides, sugar alcohols, sugar amines, sugar acids</li> <li>Amino acids</li> <li>Antibiotics</li> </ul>    |  |  |
| Ag   | <ul> <li>Halogen anions, e.g. bromide, iodide</li> <li>Cyanide and sulfide</li> <li>Thiosulfate</li> <li>Pharmaceuticals</li> </ul>                |  |  |
| Pt   | <ul><li>Alcohols and glycols</li><li>Hydrazines</li><li>Arsenite and hypochlorite</li></ul>  |  |  |
| GC   | <ul> <li>Inorganic anions, e.g. nitrite, sulfite, iodide</li> <li>Phenols</li> <li>Catecholamines and aromatic amines</li> <li>Vitamins</li> </ul> |  |  |

## The right system for every challenge

Metrohm offers the amperometric detector in the form of an detector block (850 IC Amperometric Detector) and as a stand-alone detector (896 Professional Detector). These two instruments can be used both with an 850 Professional IC and with an 881 Compact IC pro or 882 Compact IC plus. 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<sup>™</sup> ion chromatography software.

The stand-alone detector 896 Professional Detector is a versatile instrument that is offered in three different versions: only as an amperometric detector (896 Professional Detector – Amperometry), only as a conductivity detector (896 Professional Detector – Conductivity) and as a combination of both detectors in a single instrument (896 Professional Detector – Conductivity & Amperometry). The 896 Professional Detector is the interface for a number of optional peripheral devices as for example, 872 Extension Modules, 800 Dosinos, 891 Professional Analog Outs, or USB devices.

Due to the wide variety of possible configurations with the 850 IC Amperometric Detector and the 896 Professional Detector 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  | Professional Detector – Amperometry |  |
|---------------------------|---|-------------------------------------|--|
|                           | 2.850.9110  | 2.896.0020                          |  |
| 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   |                                     |  |
|                           | DC mode: 1 potential  |                                     |  |
| Potential profile         | PAD mode: 40 potentials   |                                     |  |
|                           | flexIPAD mode: 40 potentials (steps ar  | nd ramps)                           |  |
| Data output channels      | Current and charge  |                                     |  |
|                           | DC mode: 0.00012 pA to 2 mA, autor  | ange                                |  |
| Digital signal range      | PAD mode: 0.012 pA to 2 mA  |                                     |  |
|                           | flexIPAD mode: 0.12 pC to 200 $\mu$ C   |                                     |  |
|                           | CV mode: 0.12 pA to 20 mA   |                                     |  |
|                           | DC mode: < 5 pA   |                                     |  |
| Electronic noise          | PAD mode: < 10 pA   |                                     |  |
|                           | flexIPAD mode: < 30 pC  |                                     |  |
| Control, data collection  | MagIC Net <sup>™</sup> 2.3 Compact, Professional and Multi                        |                                     |  |
| and data evaluation       |   |                                     |  |
|                           | Temperature stability better than 0.05 °C   |                                     |  |
| Detector temperature      | Temperature range: ambient temperature +8 °C to 80 °C                             |                                     |  |
| Liquid-carrying parts     | Free of metal   |                                     |  |
|                           | Automatic function test upon startu   | р                                   |  |
| System readiness          | Leak sensor   |                                     |  |
|                           | • Monitoring of temperature stability   |                                     |  |
|                           | Can be installed in 850, 881, 882,  | Can be combined with                |  |
| Peripheral devices        | standard component of   | 850, 881, 882, 883, 887 und 896     |  |
|                           | 2.896.0020 and 2.896.0030   | 650, 661, 662, 665, 667 unu 690     |  |
| Installation              | Dedicated and multiple detection (parallel and in series) possible                |                                     |  |
| Sample preparation and    | Can be combined with all inline sample preparation techniques (MISP) and          |                                     |  |
| liquid handling           | injection techniques (full-loop, MiPT, MiPuT)                                     |                                     |  |
| Analog data output        | Via paripharal davisas  | Possible directly with              |  |
|                           | Via peripheral devices  | 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 and glassy carbon working electrodes           |                                     |  |
|                           | (2 and 3 mm diameter)   |                                     |  |
| Reference electrode       | Replaceable solid-phase Pd electrode and Ag/AgCl reference electrode              |                                     |  |
| Auxiliary electrode       | Stainless steel auxiliary electrode incorporated in the measuring cell            |                                     |  |
| Cell volume               | $<$ 0.1 $\mu$ L wall-jet cell (with 2 mm working electrode and 25 $\mu$ m spacer) |                                     |  |



## Ordering information

#### Detectors

- 2.850.9110 850 IC Amperometric Detector
- 2.896.0010 896 Professional Detector Conductivity
- 2.896.0020 896 Professional Detector Amperometry
- 2.896.0030 896 Professional Detector Conductivity and Amperometry

#### Equipment with intelligent wall-jet 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\*\*\*

#### 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)
- 6.1257.260 Au working electrode (2 mm)
- 6.1257.240 Ag working electrode (3 mm)
- 6.1257.230 Pt working electrode (3 mm)
- 6.1257.220 GC working electrode (3 mm)
- 6.1257.720 Ag/AgCl reference electrode
- 6.1257.740 Pd reference electrode

### Other accessories

- 6.1257.810~ 50  $\mu m$  spacers for wall-jet cell (3 pieces)
- 6.1257.830 25 µm spacers for wall-jet cell (3 pieces)
- 6.1257.820 50 µm spacer for thin-layer 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)
- 6.2813.040 Dummy cell
- 6.2061.100 Bottle holder for Professional IC instruments
- 6.2061.110 Tray with leak sensor for Professional IC instruments
- \* with Au working electrode (3 mm) and Pd reference electrode
- $^{\star\star}\,$  with Ag working electrode (3 mm) and Pd reference electrode
- \*\*\* with GC working electrode (3 mm) and Ag/AgCl reference electrode

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