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Introduction
Metal traces: total metal and species analysis – quick and easy with voltammetry

Voltammetry instruments from Metrohm are affordable, have low running costs, compact dimensions and are great in detecting.

Traces of toxic heavy metals can be analyzed at extremely low concentrations without much trouble. The detection limits are all in the ppt range, so that the necessary sensitivity can always be achieved. For speciation of analytes voltammetry is the method of choice.

Trace metal analyses can be carried out with the same or better sensitivity for a fraction of the cost of acquiring an AAS or ICP instrument. The low running costs are an additional bonus point for voltammetry. Apart from small amounts of reagents only small volumes of pure nitrogen are required. No expensive combustible gases, no reconstruction of the laboratory for special gas supplies and fume hoods, no expensive lamps. And no time-consuming calibration of the analytical system.

Convincing applications

Analysis of metal traces
Total concentration can be determined by both voltammetry and spectroscopic methods. With voltammetry, however, it is possible to distinguish between the different oxidation states of metal ions and to specify the biological availability of heavy metals. Free and bound metal ions can be differentiated from each other. This makes voltammetry an essential tool for environmental analysis. Spectroscopy allows comparable statements to be made only after complicated separation of metal species. Due to the compact size the instrument can also be used in mobile laboratories.

Samples with a high ionic concentration are not a problem for voltammetry. Voltammetry is also predestined for the analysis of:

• water, wastewater and sea water
• foodstuffs
• salts, pure chemicals
• electroplating baths

Specific analysis of organic chemicals
Not only metals but also various organic compounds can be determined by voltammetry. The technique is used in organic chemistry, e.g. for the analysis of contaminants, and in the pharmaceutical industry for the determination of the concentration of the active substance.

Examples of interesting determinations
• 4-carboxybenzaldehyde in terephthalic acid
• free styrene in polystyrene
• vitamins in fruit and vegetable juices, vitamin formulations

Determination of anions
Some anions can also be determined by voltammetry. The analysis of the following anions is particularly interesting:

• cyanide
• sulfide
• nitrite
• nitrate
• iodide

<table>
<thead>
<tr>
<th>Anion</th>
<th>Typical detection limit in ppt</th>
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<tbody>
<tr>
<td>Sb(III) / Sb(V)</td>
<td>200 ppt</td>
</tr>
<tr>
<td>As(III) / As(V)</td>
<td>100 ppt</td>
</tr>
<tr>
<td>Bi</td>
<td>500 ppt</td>
</tr>
<tr>
<td>Cd</td>
<td>50 ppt</td>
</tr>
<tr>
<td>Cr(III) / Cr(VI)</td>
<td>25 ppt</td>
</tr>
<tr>
<td>Co</td>
<td>50 ppt</td>
</tr>
<tr>
<td>Cu</td>
<td>50 ppt</td>
</tr>
<tr>
<td>Fe(II) / Fe(III)</td>
<td>50 ppt</td>
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<td>Pb</td>
<td>50 ppt</td>
</tr>
<tr>
<td>Hg</td>
<td>100 ppt</td>
</tr>
<tr>
<td>Mo(VI) / Mo(VII)</td>
<td>50 ppt</td>
</tr>
<tr>
<td>Ni</td>
<td>50 ppt</td>
</tr>
<tr>
<td>Pt</td>
<td>0.1 ppt</td>
</tr>
<tr>
<td>Rh</td>
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<tr>
<td>Se(IV) / Se(VI)</td>
<td>300 ppt</td>
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<td>Ti</td>
<td>50 ppt</td>
</tr>
<tr>
<td>W</td>
<td>200 ppt</td>
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<tr>
<td>U</td>
<td>25 ppt</td>
</tr>
<tr>
<td>Zn</td>
<td>50 ppt</td>
</tr>
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</table>

1 ppt = part per trillion = 1 ng/kg

Typical detection limits in voltammetric trace analysis
Cyclic Voltammetric Stripping Analysis (CVS) and Cyclic Pulse Voltammetric Stripping Analysis (CPVS) are widespread methods used in the plating industry for the determination of organic additives in electroplating baths. A simple, robust and favorably-priced rotating disk electrode made of platinum is used for these analyses; this electrode is built into the VA stand instead of the otherwise used Multi-Mode Electrode.

For many technical coatings, particularly in PCB manufacture in the electronics industry, this method is an essential part of the production control process. The most important fields of application are acid copper baths as well as tin-lead baths. The quantitative determination of the additives is carried out using their influence on the deposition of the main components of the electroplating bath. As the measurement corresponds to a process which is similar to the production process, the activity of the additives and therefore their effectiveness in the electroplating process is measured directly.

Quantification of the various types of additives requires special calibration techniques; these are all implemented in the 797 VA Computrace: The so-called brighteners are determined by using the Linear Approximation Technique (LAT) or the Modified Linear Approximation Technique (MLAT). Dilution Titration (DT) is used for the determination of the suppressors.

With CVS or CPVS the concentration of the additives can be determined exactly. The effective concentration of the particular additive in the bath sample is shown directly and printed out in mL additive per liter bath. This means that topping up to achieve the set concentration can be carried out very exactly. This guarantees continuous interference-free operation of the unit. Accuracy of analytical results in particular has helped this method to become widely accepted in the electroplating industry.

Other methods, such as the classical Hull cell method, do not allow the concentration to be determined, but can only be used to assess the quality of the metallic layer that has been deposited.

To carry out the determination one of the pre-installed methods is loaded. After a few parameters have been adapted, the analysis can be started. Ready-to-use methods are included for the most important bath types from leading manufacturers; these methods have been worked out in our application laboratory.
Instruments for voltammetry and CVS

797 VA Computrace

Simple operation
The 797 VA Computrace is a VA Stand with built-in potentiostat/galvanostat connected to a PC via the USB port. Control, data acquisition and evaluation are carried out by the PC software supplied, which runs under Microsoft Windows™ 2000, Windows™ XP Professional, Windows™ Vista and Windows™ 7. Due to the well-laid-out program structure the simplicity of operation is unsurpassed. All the methods described in the Metrohm Application Bulletins and Application Notes have already been pre-installed. Method modifications require changing of only a few parameters.

Fields of application

Classical trace analyses
Trace analysis of metal ions or other analytes after calibration by standard addition or calibration curve down to the ultratrace range (ppt) is one of the most important fields of application of the 797 VA Computrace. Evaluation is carried out automatically and can be controlled and reprocessed at the PC. Standard additions can, of course, be carried out automatically by using Dosinos.

Determination of additives in electroplating baths
These new methods are the second most important field of application. With the 797 VA Computrace the determination of additives in electroplating baths is possible for the first time. All the necessary techniques for data recording, evaluation and calibration have been implemented in the software; methods for important, often used bath chemicals have already been optimized and pre-installed.

Only with the 797 VA Computrace is it possible to carry out CVS analyses and classical trace metal determination with a single instrument. This means that both investment costs and running costs can be reduced, as can the familiarization time for the operating staff.

«Exploratory» mode for method development and training
The «Exploratory» is characterized by an intuitive user interface. This has been reduced to the most important functions that are necessary for carrying out simple electrochemical experiments. After entering a few parameters a voltammogram can be taken; the effects of changing parameter settings on the curve can be seen directly. A direct comparison of the voltammograms is the greatest strength of the «Exploratory» mode. It has been specially developed for training purposes in electrochemistry, but also helps in developing the optimal method for solving an analytical problem.

Proven working electrodes
The time-proven Multi-Mode Electrode (MME) or rotating disk electrodes made from various materials are used as sensors.
MVA accessory kits

MVA-Hg, equipment for mercury determination (6.5327.000)

Complete set of accessories for the determination of mercury as per Application Bulletin 96. Contains rotating gold working electrode, reference electrode, glassy-carbon auxiliary electrode and other accessories.

MVA-As, equipment for arsenic determination (6.5327.010)

Complete set of accessories for the determination of arsenic as per Application Bulletin 226. Contains rotating gold working electrode, reference electrode, glassy-carbon auxiliary electrode and other accessories.

MVA-CVS, equipment for CVS (6.5327.020)

Complete set of accessories for the determination of organic additives in electroplating baths by CVS (cyclic voltammetric stripping). Contains rotating platinum working electrode, reference electrode, platinum auxiliary electrode and other accessories.
Automation in VA

Automation in voltammetry – simple and efficient
Compact sample changer for VA
863 Compact VA Autosampler

The 863 Compact VA Autosampler is the space-saving and affordable sample changer for voltammetric trace analysis in combination with the 797 VA Computrace. It permits the determination of small series of similar samples with a high degree of accuracy.

Up to 18 samples can be analyzed automatically in a series. The sample solution is transferred from the sample rack of the 863 Compact VA Autosampler to the measuring vessel of the 797 VA Computrace using the built-in peristaltic pump. All auxiliary solutions necessary for the voltammetric determination are added from Dosinos during the measuring procedure. The two pumps of the connected 843 Pump Station empty and rinse the vessel after each determination. The 863 Compact VA Autosampler is programmed via the built-in keyboard.

The scope of delivery includes a built-in sample rack, extensive accessories and connection cables for setting up a complete workplace.

Ordering information
2.863.0020 863 Compact VA Autosampler

Options
2.843.0040 843 VA Membrane Pump Station
2.843.0140 843 VA Peristaltic Pump Station
Flexible sample changer for CVS
838 Advanced VA Sample Processor

The 838 Advanced VA Sample Processor together with the 797 VA Computrace permits the fully automatic determination of organic additives in electroplating baths by using «Cyclic Voltammetric Stripping» (CVS). Series of brighteners or suppressors can be analyzed unattended in succession. The sample rack provided accommodates up to 56 samples.

The sample solution is transferred from the sample vessel on the rack of the 838 Advanced VA Sample Processor to the measuring vessel of the 797 VA Computrace either by using the built-in peristaltic pump for determining brighteners or an 800 Dosino for determining suppressors by dilution-titration technique (DT). Further auxiliary solutions necessary for CVS analysis are automatically added by additional 800 Dosinos. The connected 843 Pump Station empties and rinses the vessel after each determination.

The 838 Advanced VA Sample Processor is equipped with a flexible method editor in order to adapt the analytical sequence to meet the user’s requirements. The separate keyboard allows access to the method editor and all other instrument settings. During the determination of a sample series the 838 Advanced VA Sample Processor is remote controlled by the 797 VA Computrace.

The scope of delivery includes accessories, sample rack, keyboard and all accessories for setting up a complete workplace.

Ordering information
2.838.0310  838 Advanced VA Sample Processor

Options
2.843.0040  843 VA Membrane Pump Station
2.843.0140  843 VA Peristaltic Pump Station
MVA – Metrohm VA systems

Complete voltammetry systems – customized for each application

The Metrohm MVA systems are complete, ready-to-use packages customized to meet a wide range of requirements. Just select the package, connect it to a supply of nitrogen and to a PC and you are ready to start.

From a simple-to-operate voltammetric first-user system up to completely unattended, fully automatic analysis systems for process control, you will find the right system for your demands in our portfolio. Each MVA system is supplied with all the necessary accessories. Not included in the scope of delivery are the PC, and, if required, nitrogen and mercury. For special applications the MVA accessory kits (MVA-UV, MVA-Hg, MVA-As and MVA-CVS) can be used together with the basic MVA systems to extend the range of applications.
MVA systems with the 797 VA Computrace for classical trace analysis

797 VA Computrace system for trace analysis (MVA-01)

Analysis system for voltammetric trace analysis and training. For manual operation. The system is suitable for quantitative analysis in routine and research laboratories. Consisting of 797 VA Computrace. Without PC.

797 VA Computrace system for trace analysis with automatic standard addition (MVA-02)

Analysis system for voltammetric trace analysis and training. Consisting of 797 VA Computrace with two 800 Dosinos for automatic addition of auxiliary solutions. Without PC.

Fully automated 797 VA Computrace system for trace analysis (MVA-03)

Fully automatic analytical system for voltammetric trace analysis and education. Consists of 797 VA Computrace with 863 Compact VA Autosampler and two 800 Dosinos for automatic addition of auxiliary solutions. Automatic processing of max. 18 samples. This system is the optimum solution for automatic analysis of small series of samples that have to be analyzed for a single or two parameters in one run. Without PC.
MVA systems with the 797 VA Computrace for the determination of organic additives in electroplating baths by CVS

797 VA Computrace system for CVS (MVA-11)

Analysis system for the determination of organic additives in electroplating baths by CVS technique («Cyclic Voltammetric Stripping»). Consisting of 797 VA Computrace for CVS. All additions of solutions are carried out manually. Without PC.

797 VA Computrace system for CVS with automatic solution addition (MVA-12)

Analysis system for the determination of organic additives in electroplating baths by CVS technique («Cyclic Voltammetric Stripping»). Consists of 797 VA Computrace for CVS with three 800 Dosinos for automatic addition of auxiliary solutions and sample. Without PC.

797 VA Computrace system for fully automated CVS (MVA-13)

Analysis system for the determination of organic additives in electroplating baths by CVS technique («Cyclic Voltammetric Stripping»). Consists of 797 VA Computrace for CVS, 838 Advanced VA Sample Processor, three 800 Dosinos and rinsing kit. For sample series up to 56 samples. Without PC.
Sample preparation for trace analysis
705 UV Digester

Digestion is decisive for the reliability of trace and ultra trace analyses in natural sample matrices. The 705 UV Digester is a sample digestion unit proven in practice. It is used for sample preparation in spectroscopy, polarography, voltammetry and ion chromatography to eliminate the organic matrix. ICP-AES, graphite-furnace and flame AAS also benefit from the 705 UV Digester.

The instrument has been designed for the digestion of water samples free from suspended matter that contain low to moderate concentrations of organic material, for example, natural surface waters. Owing to their low content in heavy metals, these samples are very sensitive to contamination. The advantage of UV photolysis is that only a little hydrogen peroxide has to be added and consequently the blank values can be kept very low. Digestion by UV photolysis is based on the photolytic generation of OH radicals, which in turn react with organic compounds and degrade them. Hydrogen peroxide is used as an initiator of the radical reaction. The radiant energy of the mercury lamp is converted to heat and this accelerates the digestion.

- Simultaneous digestion in 12 or 16 quartz sample tubes, depending on the sample volume
- As only minimal amounts of expensive digestion chemicals are needed (typically 50 µL), the blank value is virtually zero
- The costs and the load on the environment are minimal

Ordering information

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<th>Code</th>
<th>Description</th>
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<td>2.705.0016</td>
<td>705 UV Digester (220V / 60 Hz)</td>
</tr>
<tr>
<td>2.705.0017</td>
<td>705 UV Digester (220V / 50 Hz)</td>
</tr>
</tbody>
</table>

- The digested solution can be used directly without the need for aliquot sampling or topping up
- The digestion temperature of approx. 90 °C means most effective digestion and practically no losses of volatile elements