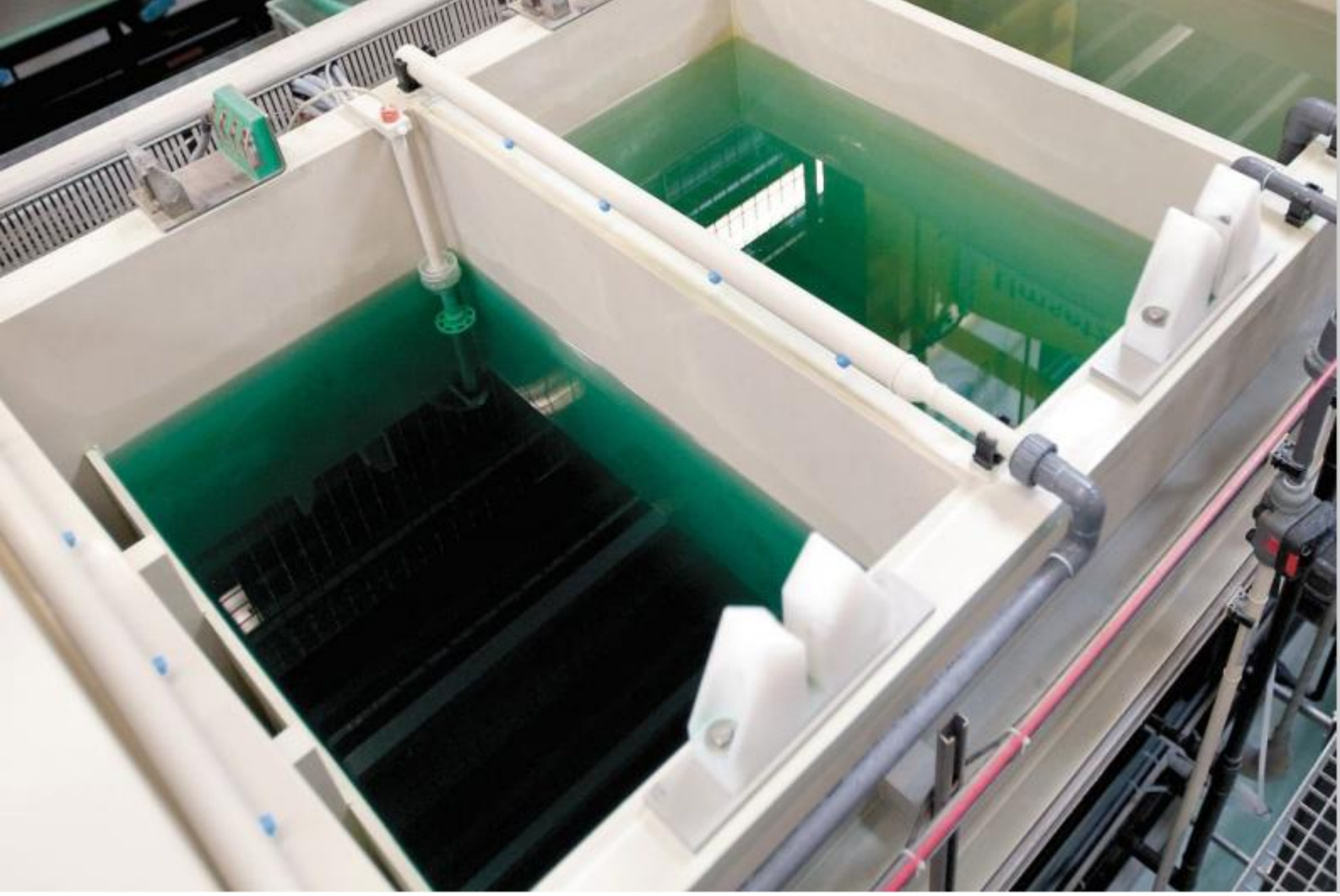

Application Note AN-V-196

Antimony and bismuth in electroless nickel baths

Stabilizer determination using anodic stripping voltammetry

Lead was commonly used as a stabilizer in electroless nickel plating processes in the past. The regular and precise determination of the stabilizer concentration is essential to keep the plating process running successfully under stable conditions. With the increasing number of restrictions in recent years on the use of lead in consumer products, particularly electronics, alternative stabilizers were developed and introduced. Two of these alternative stabilizers used to replace lead are antimony and bismuth.

Electroless nickel plating is used in various industrial production processes. The ENIG (electroless nickel, immersion gold) and ENEPIG (electroless nickel, electroless palladium, immersion gold) processes in the production of printed circuit boards are very reliant on the success of this method as electroless nickel plating is the first step in the process.

Differential pulse anodic stripping voltammetry has been established as a straightforward, sensitive, selective, and interference-free method for this application.

SAMPLE

Electroless nickel plating bath

EXPERIMENTAL

After diluting the sample in supporting electrolyte, the voltammetric determination of antimony and bismuth is carried out on the 884 Professional VA with the Multi-Mode Electrode pro as working electrode using the parameters listed in Table 1. The concentration is determined by two additions of antimony and bismuth standard addition solution.

Figure 1. 884 Professional VA.

Table 1. Parameters for the determination of Sb3+ and Bi.

Parameter	Setting
Working electrode	HMDE
Mode	DP – Differential Pulse
Deposition potential	-0.4 V
Deposition time	30 s
Start potential	-0.3 V
End potential	+0.05 V
Peak potential Sb(III)	-0.16 V
Peak potential Bi	-0.04 V

ELECTRODES

- Working electrode: Multi-Mode Electrode pro with silanized capillaries
- Reference electrode: Ag/AgCl/KCl (3 mol/L) reference electrode with electrolyte vessel. Bridge electrolyte: KCl (3 mol/L)
- Auxiliary electrode: Platinum rod electrode

RESULTS

The determination of antimony and bismuth in electroless nickel plating baths can be carried out in a simple and straightforward manner. The method is selective and free of interferences. It is suitable for concentrations in the low to mid mg/L range.

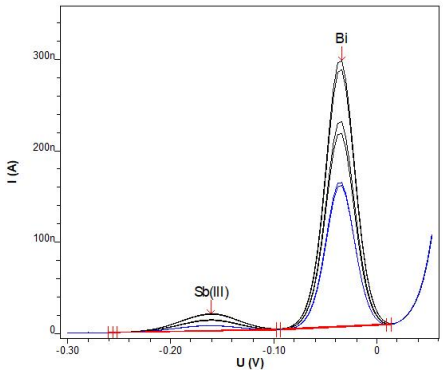

Figure 2. Determination of antimony and bismuth in electroless nickel bath with two standard additions.

Table 2. Results in electroless nickel bath

Analyte	Concentration [mg/L]
Sb(III)	0.4
Bi	4.7

CONTACT

Metrohm USA
9250 Camden Field Pkwy
33578 Riverview, FL
info@metrohmusa.com

CONFIGURATION

884 Professional VA semiautomated für Multi-Mode Electrode (MME) with 2 Dosinos

884 Professional VA semiautomated for Multi-Mode Electrode (MME) is a convenient high-end routine analyzer for trace determinations with voltammetry and polarography with the Multi-Mode Electrode pro or the scTRACE Gold. The proven Metrohm electrode methods in combination with a high-performance potentiostat/galvanostat and the extremely flexible viva software open up new perspectives for the determination of heavy metals. The potentiostat with a certified calibrator readjusts itself automatically before each measurement, thus guaranteeing maximum precision.

Determinations with rotating disc electrodes can also be performed with the instrument, e.g. determinations of organic additives in electroplating baths with "Cyclic Voltammetric Stripping" (CVS), "Cyclic Pulse Voltammetric Stripping" (CPVS), and chronopotentiometry (CP). The replaceable measuring head enables rapid changes between the various applications with different electrodes.

2x 800 Dosinos (supplied) permit the automatic addition of auxiliary solutions during the determination, e.g., electrolyte, buffer or standard solutions.

The viva software is required for control, data collection, and evaluation.

The 884 Professional VA semiautomated for Multi-Mode Electrode (MME) is supplied with extensive accessories and a measuring head for the Multi-Mode Electrode pro. Electrode set and viva license need to be ordered separately.

VA electrode equipment with Multi-Mode Electrode pro for Professional VA instruments

Complete electrode set for polarographic and voltammetric determinations. Includes Multi-Mode Electrode pro, reference electrode, platinum auxiliary electrode, measuring vessel, stirrer, electrolyte solution and additional accessories for setting up and operating the Multi-Mode Electrode.