



Application Note AN-V-019

Lead in a nickel plating bath

Direct determination of lead stabilizers using anodic stripping voltammetry

SUMMARY

Lead is commonly used as stabilizer in electroless nickel plating processes. The regular and precise determination of the electrochemically active Pb^{2+} concentration is essential to keep the plating process running optimally under stable conditions.

Electroless nickel plating is used in various industrial production processes (e.g., production of hard disks, or as protection against corrosion or wear). The ENIG (electroless nickel, immersion gold) and ENEPIG (electroless nickel, electroless palladium, immersion gold) processes in the production of printed circuit

boards (PCB) are very reliant on the success of this method as electroless nickel plating is the first step in the process. Reducing the amount of out-of-specification product due to plating errors can save manufacturers significant costs.

Differential pulse anodic stripping voltammetry can be used to determine the active lead content after dilution. The voltammetric determination 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 polarographic determination of lead 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 of lead is determined by two additions of Pb standard addition solution.



Figure 1. 884 Professional VA.

Table 1. Parameters for the Pb determination

Parameter	Setting
Working electrode	HMDE
Mode	DP – Differential Pulse
Deposition potential	-0.6 V
Deposition time	90 s
Start potential	-0.6 V
End potential	-0.35 V to -0.25 V
Peak potential Pb	-0.4 V to -0.44 V

ELECTRODES

- Working electrode: Multi-Mode Electrode pro with silanized glass 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 Pb^{2+} 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 in the electroless nickel bath.

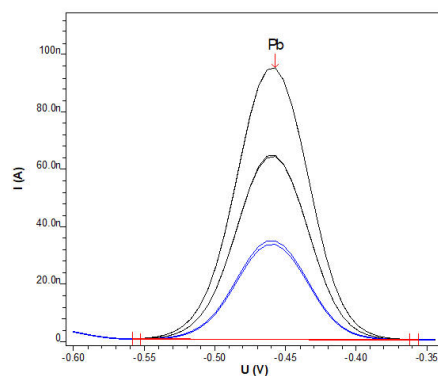


Figure 2. Determination of Pb^{2+} in electroless nickel bath with two standard additions.

Table 2. Result

Sample	Concentration Pb^{2+} [mg/L]
Electroless nickel bath	1.1

Internal references: AW DE4-0226-122009; AW DE4- 0166-112004

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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.

