



Application Note AN-V-237

Lead stabilizer in an electroless Ni plating bath

Direct lead determination over a wide concentration range with the Bi drop electrode

Electroless nickel plating processes are a low-cost solution for resistance against wear and corrosion. These processes offer the ability to plate on large and complex substrates with a uniform thickness. When manufacturing printed circuit boards, the efficiency of the ENIG (electroless nickel, immersion gold) and ENEPIG (electroless nickel, electroless palladium, immersion gold) processes strongly depends on the exact composition of electroless nickel plating bath. Monitoring the concentration of bath stabilizers such

as lead is crucial to ensure high-quality coatings that meet specified standards. The typical concentration of lead stabilizer in an electroless Ni bath is around 1 mg/L Pb.

The mercury-free Bi drop electrode offers a non-toxic approach for voltammetric analysis of heavy metals. This environmentally friendly sensor allows the determination of lead in an electroless Ni plating bath with anodic stripping voltammetry (ASV).

SAMPLE

Electroless Ni plating solution

EXPERIMENTAL

Add water, the Ni plating bath sample, and the supporting electrolyte into the measuring vessel. The determination of lead is carried out with the 884 Professional VA (Figure 1) using the parameters specified in Table 1. The concentration is determined by two additions of lead standard addition solution. Activate the Bi drop electrode electrochemically prior to the first determination.



Figure 1. 884 Professional VA fully automated for VA.

Table 1. Parameters

Parameter	Setting
Mode	DP – Differential Pulse
Deposition potential	-0.65 V
Deposition time	60 s
Start potential	-0.65 V
End potential	-0.3 V
Peak potential Pb	-0.5 V

ELECTRODES

- Working electrode: Bi drop
 - Reference electrode: Ag/AgCl/KCl (3 mol/L)
- Auxiliary electrode: Glassy carbon rod

RESULTS

The limit of detection (LOD) for a 60 s deposition time is 0.1 mg/L Pb. This outstanding sensitivity is more than sufficient to monitor the typical concentrations of lead stabilizer in electroless Ni plating baths.

This method is best suited for automated systems or process analyzers, allowing the fully automatic determination of lead in large sample series.



Figure 2. Determination of lead in an electroless Ni bath sample containing $\beta(\text{Pb}) = 1.2 \text{ mg/L}$ (sample volume: $50 \text{ }\mu\text{L}$).

Table 2. Result

Sample	Pb (mg/L)
Ni plating bath containing $\beta(\text{Pb}) = 1.2 \text{ mg/L}$	1.16

REFERENCES

1. [Application Bulletin 438](#): Determination of cadmium and lead in water samples by anodic stripping voltammetry with a Bi drop electrode.

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CONFIGURATION



884 Professional VA manual for Multi-Mode Electrode (MME)

884 Professional VA manual for Multi-Mode Electrode (MME) is the entry-level instrument for high-end trace analysis with voltammetry and polarography with the Multi-Mode Electrode pro or the scTRACE Gold or the Bismuth drop electrode. 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.

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

The 884 Professional VA manual for 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.