

VA APPLICATION NOTE V-019

Lead in electroless nickel baths

Direct determination of lead stabilizers using anodic stripping voltammetry

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.

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

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

RESULTS

The determination of Pb²⁺ 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.

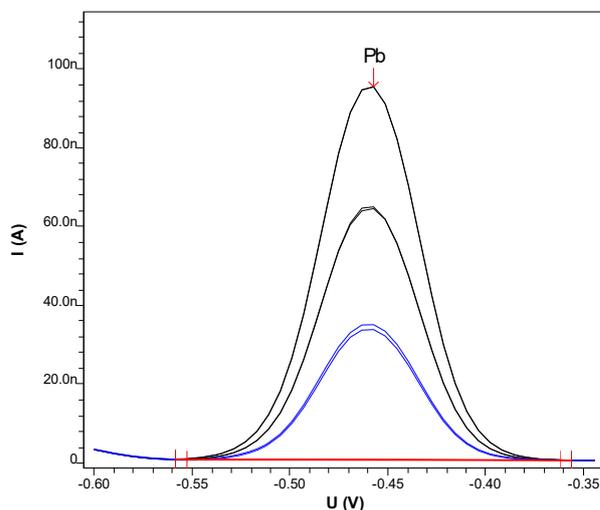


Figure 2. Determination of Pb²⁺ in electroless nickel bath with two standard additions.

Table 2. Result

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

Analytes: Transition metals – lead
Matrix: Plating baths – nickel
Method: Voltammetry, Polarography
Industry: Electronics & semiconductors; Plating & galvanics