

Ti Application Note No. T- 24

Title: Metal contents of alkaline plating baths for cadmium, copper, lead or zinc

Summary: Determination of cadmium, copper, lead and zinc in alkaline plating baths by potentiometric titration with EDTA using the Cu ISE.

Sample: Alkaline plating bath for cadmium, copper, lead or zinc

Sample Preparation: Work has to be performed in the fume hood!!!
Pipette 5.00 mL sample into a Kjeldahl flask. Add 10 mL dist. water and then carefully conc. HNO₃ in excess until the mixture is strongly acidic. Boil to dissolve and to remove cyanide completely. Allow the solution to cool, rinse it into a 50 mL volumetric flask with dist. water, fill the flask to the mark and mix the contents.

Instruments and Accessories: 702, 716 or 736 Titrino or 726 Titroprocessor, 6.0502.140 Cu ISE and 6.0726.100 reference electrode

Analysis:

Determination of Cd and Zn:
Pipette a defined volume of the prepared sample solution into a beaker and dilute with dist. water to ca. 40 mL. Add 5 mL NH₃/NH₄Cl buffer pH = 10 (1 mol/L NH₃ + 1 mol/L NH₄Cl) and 1 mL CuEDTA. Stir for 1 min and titrate with c(EDTA) = 0.1 mol/L using the MET mode.

Determination of Cu:
The same as for Cd and Zn, but without addition of CuEDTA.

Determination of Pb:
The same as for Cd and Zn, but instead of NH₃/NH₄Cl buffer pH = 10 add 5 mL acetate buffer pH = 4.6 (0.5 mol/L acetic acid + 0.5 mol/L Na acetate).

Calculation: 1 mL c(EDTA) = 0.1 mol/L corresponds to

11.241 mg Cd ²⁺
6.3546 mg Cu ²⁺
20.72 mg Pb ²⁺
6.538 mg Zn ²⁺

Calculation: $\text{g/L metal} = \text{EP1} * \text{C01} / \text{C00}$

EP1 = titrant consumption in mL

C00 = mL of original sample contained in the sample volume used for the titration

C01 = 11.241 or 6.3546 or 20.72 or 6.538 (Cd^{2+} or Cu^{2+} or Pb^{2+} or Zn^{2+} equivalent in mg/mL, see above)

Remarks: If the analysis is performed at only one pH value it is not possible to determine the different metals in a mixture as only the sum of the metals is determined.