

VA Application Note No. V - 170

Title: Selenium(IV) in zinc plant electrolyte

Summary: The concentration of Se(IV) in zinc plant electrolyte is determined by cathodic stripping voltammetry (CSV) in ammonium sulfate electrolyte containing EDTA and Cu. The Cu concentration has to be adapted to the sample and the deposition time. With voltammetry only free selenium is determined, therefore it has to be taken into consideration that selenium forms sparingly soluble compounds with numerous cations (e.g. $\text{Fe}_2(\text{SeO}_3)_3$ with $K_s = 2 \cdot 10^{-31}$).

Sample: Zinc plant electrolyte (concentrated ZnSO_4 solution)

Sample preparation: None

Analysis of Se(IV)

Supporting electrolyte $c((\text{NH}_4)_2\text{SO}_4) = 2.5 \text{ mol/L}$

EDTA solution $c(\text{Na}_2\text{EDTA}) = 0.1 \text{ mol/L}$

Na₂EDTA: Ethylenediaminetetraacetic acid disodium salt

Cu solution $\beta(\text{Cu}) = 1 \text{ g/L}$

Measuring solution
 10 mL supporting electrolyte
 + 1 mL EDTA solution
 + 0.2 mL Cu solution
 + 20 μL zinc plant electrolyte
 pH 2.2 adjusted with H_2SO_4

Working electrode (WE) **MME** (Multi Mode Electrode) 6.1246.020

Auxiliary electrode (AE) **Pt** 6.0343.000

Reference electrode (RE) Reference system: Ag/AgCl/KCl (3 mol/L) 6.0728.020
 Intermediate electrolyte: $c(\text{KCl}) = 3 \text{ mol/L}$ 6.1245.010

Parameters

Working electrode	HMDE
Stirrer speed	2000 rpm
Mode	DP
Purge time	300 s
Deposition potential	-0.4 V
Deposition time	120 s
Equilibration time	10 s
Pulse amplitude	0.08 V

Start potential	-0.4 V
End potential	-0.95 V
Voltage step	0.004 V
Voltage step time	0.1 s
Sweep rate	0.04 V/s
Peak potential Se	-0.73 V

Results:	Se
	129.1 µg/L

Determination of Se

