

VA Application Note No. V - 175

Title: Antimony(III) in zinc plant electrolyte with chloranilic acid as complexing agent

Summary: The concentration of Sb(III) in zinc plant electrolyte is determined by adsorptive stripping voltammetry (AdSV) with chloranilic acid as complexing agent. In this method high copper concentrations do not interfere. A approx. 10-fold excess of lead interferes since it shows a signal close to the antimony. With the parameters given below the working range of this method is 1 – 30 µg/L antimony(III) with respect to the concentration in the measuring vessel

Sample: Zinc plant electrolyte (concentrated ZnSO₄ solution)

Sample preparation: None

Analysis of Sb(III)

| | | |
|---------------------------------|--|--------------------------|
| CAA solution | c(CAA) = 5 mmol/L CAA: chloranilic acid | |
| Measuring solution | 10 mL H ₂ O + 1 mL zinc plant electrolyte + 0.1 mL CAA solution pH 3.2 ± 0.2 adjusted with HCl | |
| 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) Intermediate electrolyte: c(KCl) = 3 mol/L | 6.0728.020 6.1245.010 |

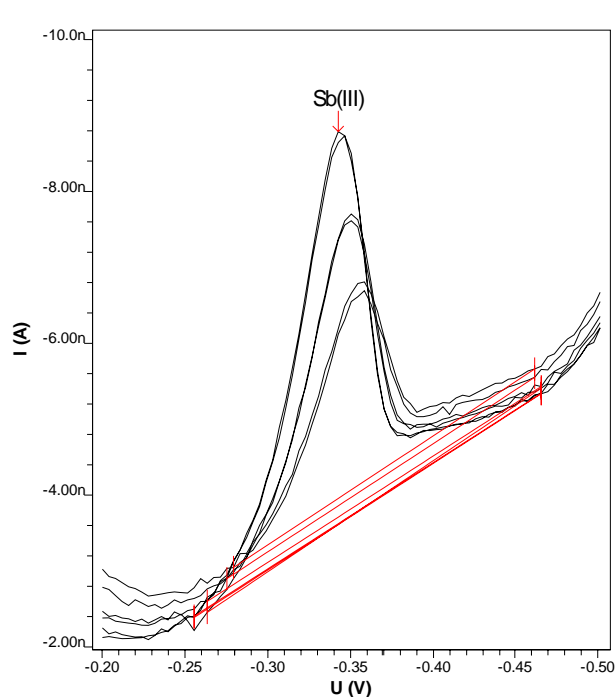
Parameters

| | |
|----------------------|----------|
| Working electrode | HMDE |
| Stirrer speed | 2000 rpm |
| Mode | DP |
| Purge time | 300 s |
| Deposition potential | 0.1 V |
| Deposition time | 10 s |
| Equilibration time | 10 s |
| Pulse amplitude | 0.05 V |
| Start potential | -0.2 V |
| End potential | -0.5 V |
| Voltage step | 0.004 V |

| | | |
|--|-------------------|----------|
| | Voltage step time | 0.2 s |
| | Sweep rate | 0.02 V/s |
| | Peak potential Sb | -0.35 V |

| | |
|-----------------|----------|
| Results: | Sb(III) |
| | 106 µg/L |

Determination of Sb(III)



sb(III)
 c = 106.254 µg/L
 +/- 2.070 µg/L (1.95%)

