VA Application Note No. V - 78

Title:	**Determination of antimony in zinc plant electrolytes (concentrated $ZnSO_4$ solutions)**

Summary:	The concentration of total Sb in zinc plant electrolytes is determined by anodic stripping voltammetry (ASV) in 5 mol/L HCl. If 0.6 mol/L HCl is used only the concentration of antimony(III) is determined selectively. The interference of an excess of Cu is suppressed by the selective oxidation of Cu. Nevertheless the concentration of Cu in the sample limits the amount of sample that can be used for the determination.

Sample:	Zinc plant electrolyte (concentrated $ZnSO_4$ solution)
Sample preparation:	None

Analysis of Sb(total)

HCl solution	w(HCl) = 30%	
Measuring solution	5 mL H_2O + 5 mL HCl solution + 0.1 mL zinc plant electrolyte	
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 1	-0.4 V
Deposition time 1	180 s
Deposition potential 2 (selective oxidation)	-0.27 V
Deposition time 2 (selective oxidation)	10 s
Equilibration time	10 s
Pulse amplitude	**0.01 V**
Start potential	-0.4 V
End potential	-0.1 V

	Voltage step	0.004 V
	Voltage step time	0.1 s
	Sweep rate	0.04 V/s
	Peak potential Sb	-0.18 V

Analysis of Sb(III)

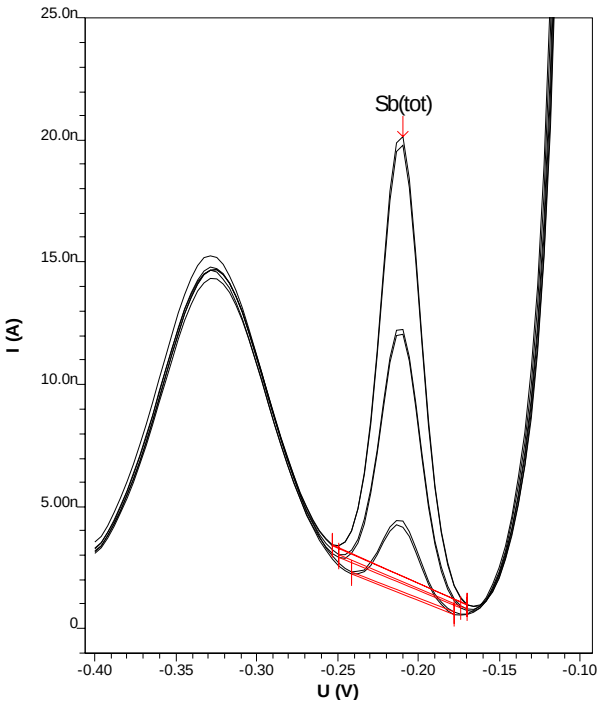
HCl solution	w(HCl) = 30%	
Measuring solution	10 mL H_2O + 0.6 mL HCl solution + 1 mL zinc plant electrolyte	
Working electrode (WE)	**MME** (Multi Mode Electrode)	6.1246.020
Auxiliary electrode (AE)	**Pt**	6.0343.000
Reference electrode (RE)	Reference system: Ag/AgCl/KCl (3 mol/L) Intermediate electrolyte: c(KCl) = 3 mol/L	6.0728.020 6.1245.010

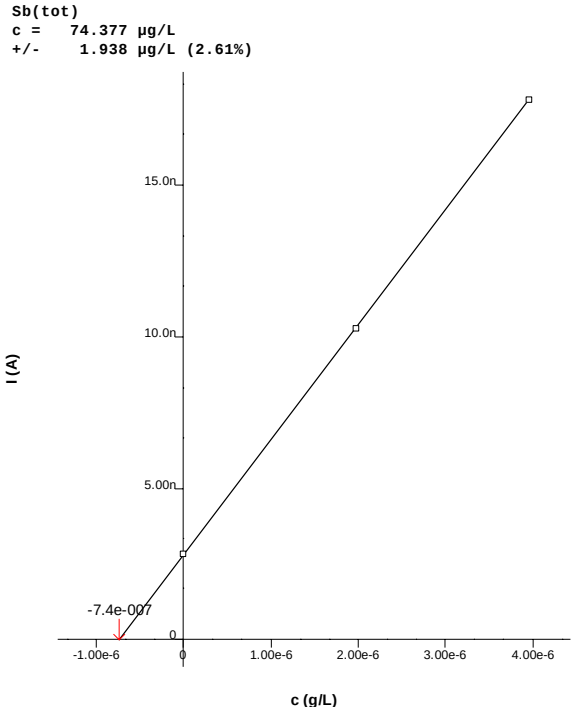
Parameters

Working electrode	HMDE
Stirrer speed	2000 rpm
Mode	DP
Purge time	300 s
Deposition potential 1	-0.4 V
Deposition time 1	120 s
Deposition potential 2 (selective oxidation)	-0.2 V
Deposition time 2 (selective oxidation)	60 s
Equilibration time	10 s
Pulse amplitude	**0.01 V**
Start potential	-0.2 V
End potential	-0.05 V
Voltage step	0.004 V
Voltage step time	0.1 s
Sweep rate	0.04 V/s
Peak potential Sb	-0.13 V

Results:	Sb(total)	Sb(III)
	74 µg/L	21 µg/L

Determination of Sb(total)





Determination of Sb(III)