

Antimony

Function: Differential Pulse Voltammetry (DPV/a)

Start Potential (mV)	-500
End Potential (mV)	-50
Current range	2,048 μ A
Scan Speed (mV/s)	30
Deposition time (s)	60
Deposition Pot. (mV)	-500
Number of cycles	3
Delay before sweep (s)	5
Purge and stir time (s)	20
Stirring speed (rpm)	300
Drop Size (a.u.)	30

Antimony concentrated standard solution (1 g/l)

Dissolve 2.743 g of potassium antimonil tartrate, $K(SbO)C_4H_4O_6 \cdot 1/2H_2O$ in 1 l of distilled water, in a volumetric flask. ($MM_{K(SbO)C_4H_4O_6 \cdot 1/2H_2O} = 333.93$; $MM_{Sb} = 121.8$).

Supporting electrolyte

1.5 % HCl for samples free of copper

12% HCl for samples containing copper.

Procedure

Samples containing no copper

Add 0.4 ml of 37% HCl to 10 ml of neutralised sample.

Samples containing copper

Add 5 ml of 37% HCl to 10 ml of neutralised sample.

Working standard solution (10 mg/l)

Dilute 1+99 the concentrated standard solution with distilled water. Prepare the solution at the moment of the analysis

Analytical Report

Analysis: digested soil solution

Sample Concentration = 12.7 µg/l (sol.)

Sample Concentration = 1.27 mg/Kg (soil)

Volumes table

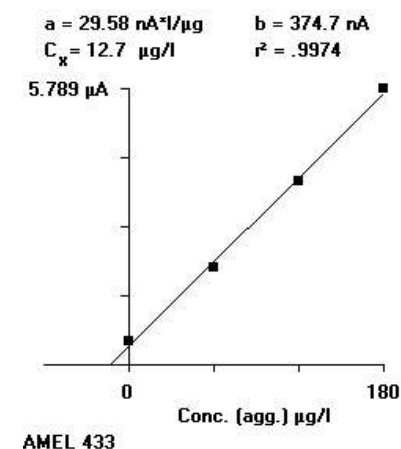
Solvent Volume	5 (ml)
Supporting Sol.	5 (ml)
Sample Volume	5 (ml)
Standard Conc.	10000 (µg/l)

Peak table

#	Peak Pot.	Height
0	-190.1	160.2 nA
1	-184.5	674.4 nA
2	-182.2	1.278 µA
3	-180.8	1.918 µA

Regression Data

#	Add Conc.	Height x dilution
0	0 µg/l	480.7 nA
1	60.0 "	2.027 µA
2	120 "	3.851 µA
3	180 "	5.789 µA

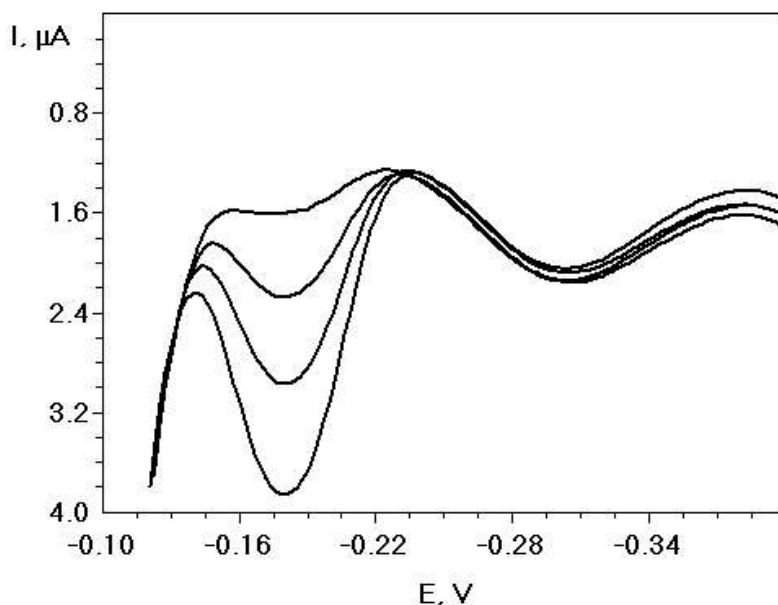


$$y = ax + b$$

$$a = 29.58 \text{ nA}^*/\mu\text{g}$$

$$b = 374.7 \text{ nA}$$

$$r^2 = .9974$$



Interferences

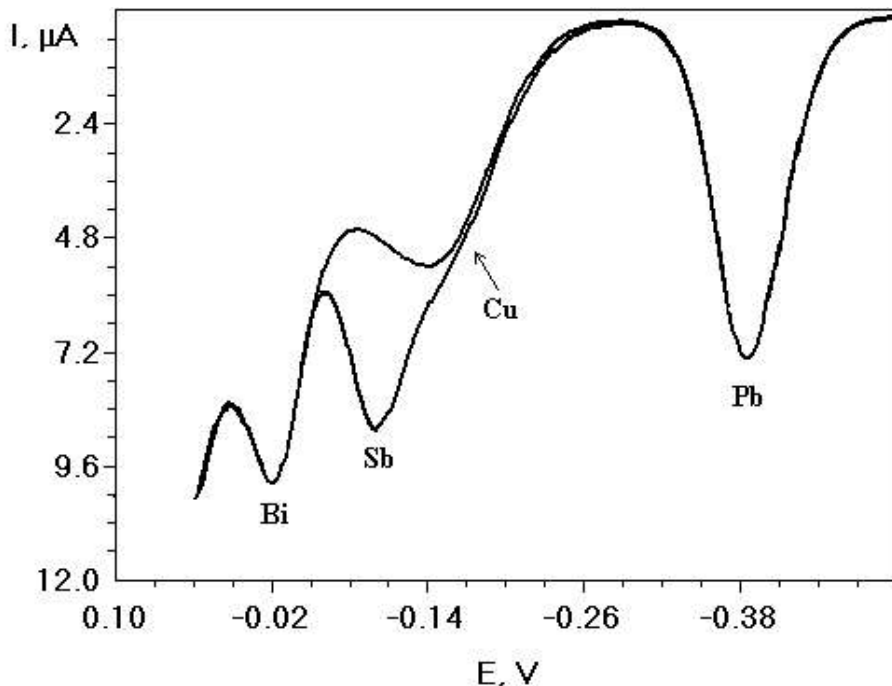


Fig. 1 - Pb, Cu, Sb e Bi in 0.6 M HCl

The antimony peak overlaps the copper peak.

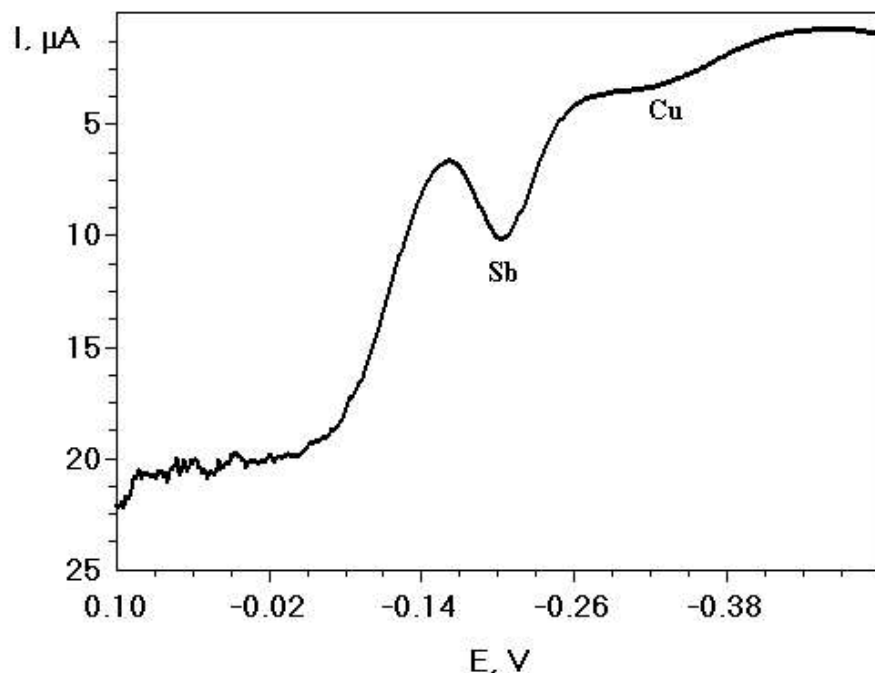


Fig. 2 - Pb, Cu, Sb e Bi in 12 M HCl

The Lead peak does not appear in the voltammogram because its potential is lower than usual. Also the copper peak shifts towards lower potential and does not interfere with the antimony discharge. The bismuth peak cannot be registered because the acid concentration is too high.

Analytical Report

Analysis: Sb in PET

Sample Concentration = 5.21 mg/l

Sample Concentration = 64.6 mg/Kg

Volumes table

Solvent Volume	0 (ml)
Supporting Sol.	12 (ml)
Sample Volume	0.1 (ml)
Standard Conc.	10 (mg/l)

Peak table

#	Peak Pot.	Height
0	-175.3	2.574 μA
1	-173	6.249 μA
2	-173	9.831 μA
3	-171.6	14.03 μA
4	-171.6	18.65 μA
5	-168.3	22.20 μA

Regression Data

#	Add Conc.	Height x dilution	
0	0 mg/l	311.5 μA	$y = ax + b$
1	10.00	762.5 μA	$a = 50.49 \mu\text{A} \cdot \text{l}/\text{mg}$
2	20.0 "	1.209 mA	$b = 263.3 \mu\text{A}$
3	30.0 "	1.740 mA	$r^2 = .9976$
4	40.0 "	2.331 mA	
5	50.0 "	2.798 mA	

