

Application Bulletin



Of interest to:
Metallurgical laboratories,
Material testing laboratories

No. 4/1 e

Biamperometric titration method for determining antimony in lead

Summary	An automatic titration method with biamperometric end-point indication for determining antimony in antimonious cable-sheathing lead is described (i.e. lead with app. 1% Sb). An 0.01 mol/L KBrO_3 solution is used as the titrating reagent.																
Reagents	<p>► $c(\frac{1}{6} \text{KBrO}_3) = 0.01 \text{ mol/L}$: Dissolve 0.2783 g KBrO_3 and 1 g KBr, both analytical grade, in distilled water and make up to 1 litre. Standardise by titrating against pure As_2O_3 in HCl at 60°C. 1 mL of 0.01 mol/L $\text{KBrO}_3 \hat{=} 0.4946 \text{ mg As}_2\text{O}_3$.</p> <p>► conc. HCl, analytical grade (no free chlorine!)</p> <p>► conc. sulphuric acid analytical grade</p>																
Apparatus	<p>► 536 Potentiograph with 665 Titrating Stand</p> <p>► 585 Polarizer with 6.0309.000 double platinum foil electrode</p> <p>► Thermostat with 6.5704.220 water-jacketed universal titration vessel</p>																
Method	<p>Weigh out accurately a quantity of about 500 mg of sample metal (drillings or turnings), and place the sample in a 50 mL Erlenmeyer flask. Add 6 mL conc. H_2SO_4, cover with a watch-glass and heat strongly. When all the lead has been converted to the sulphate, continue heating for a further 30 minutes in order to oxidise out any sulphur and sulphur dioxide condensing on the watch-glass. After cooling, wash out into the titration vessel with distilled water and conc. HCl (heating if necessary), and titrate slowly at 60°C. The following instrument settings should be used:</p> <table> <tr> <th></th><th><u>Potentiograph</u></th><th><u>Polarizer</u></th></tr> <tr> <td>Voltage range</td><td>1000 mV</td><td>---</td></tr> <tr> <td>Titration speed</td><td>30 min/100 % vol.</td><td>---</td></tr> <tr> <td>U_{pol}</td><td>---</td><td>200 mV</td></tr> <tr> <td>Sensitivity</td><td>---</td><td>50 $\mu\text{A/V}$</td></tr> </table> <p>A very well-marked inflection appears in the curve at end-point.</p>			<u>Potentiograph</u>	<u>Polarizer</u>	Voltage range	1000 mV	---	Titration speed	30 min/100 % vol.	---	U_{pol}	---	200 mV	Sensitivity	---	50 $\mu\text{A/V}$
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Calculation	1 mL 0.01 mol/L $\text{KBrO}_3 \hat{=} 0.6088 \text{ mg Sb}$																
Remarks	<p>► The presence of arsenic interferes with the determination.</p> <p>► If the titration is carried out too quickly, the free bromine produced by the reaction tends to escape from the titration vessel.</p>																
Literature	► See data published by the Material Testing Department of the Swiss Federal Postal Administration (PTT), Berne																