Application Bulletin

Metrohm

Of interest for:
Metallurgical and materialtesting laboratories

No. 37/1 e

Determination of chromium in iron and steel

Summary

Two methods of chromium determination are described, one being a biamperometric titration and the other a polarographic analysis.

1. Biamperometric titration

Reagents

$(NH_4)_2Fe(SO_4)_2$ 0.01 mol/L

$NaNO_2$ 5 %, $(NH_4)_2S_2O_8$ 10 %, $AgNO_3$ 1 % (all g/V)

HNO_3 $d = 1.4$, H_3PO_4 app. 80 %, H_2SO_4 app. 20 %

Apparatus

- 536 Potentiograph with 665 Titrating Stand or pH-Meter with Titrating Stand
- 585 Polarizer with 6.0309.000 double platinum-sheet electrode

Preparation and analysis

1...2 g of the sample metal is dissolved in 25 mL of the sulphuric acid plus 5 mL of the phosphoric acid. The ferrous iron is then oxidised to ferric iron with nitric acid. After the brown fumes have been driven off, the solution is further diluted with 50 mL distilled water and 5 mL of silver nitrate added. The chromium is then oxidised by boiling with the addition of 10 mL of ammonium persulphate. The excess persulphate is decomposed by boiling. Any permanganates which may form are reduced with sodium nitrite, the excess of nitrite also being decomposed by boiling. After cooling, carry out a biamperometric titration against ferrous ammonium sulphate until a sharp jump in potential occurs. Titrate with the following settings:

measuring range	500 mV	U pol.	200 mV
counter-voltage	– 200 mV	sensibility	$1\ V \mathrel{\hat{=}} 50\ \mu A$
feeding time	20 min/100 % Vol		

Calculation

1 mL 0.01 mol/L $(NH_4)_2Fe(SO_4)_2$ is equivalent to 0.16675 mg chromium

Limit

0.5 mg chromium per sample

Determination of chromium in iron and steel

2. Polarographic determination

Reagents

$HClO_4$	$d = 1.62$
HNO_3	$d = 1.4$
NaOH	2 mol/L

Apparatus

506 Polarecord and 663 VA Stand

Preparation and analysis

1 g of the sample metal is weighed out and dissolved in 15 mL perchloric acid and 1 mL nitric acid and then heated to drive off the brown fumes. After cooling dilute with 50 mL distilled water and then boil again for 5 minutes.

Now add sodium hydroxide so as to bring the pH of the solution up to at least pH 9, stir well and filter off the hydroxide precipitate so formed. Rinse the precipitate two or three times with hot sodium hydroxide solution (about 0.01 mol/L), combine the two filtrates and make up to 100 mL with NaOH 2 mol/L. After thorough mixing, pipette off a portion of this solution into the polarography vessel, deaerate with nitrogen and then record the polarogram. The half-wave potential is about –0.9 V.

For an AC polarogram, work with the following settings:

Initial voltage	– 0.6 V
Voltage range	– 2 V
AC voltage	10 mV

Calculation

Evaluate the results by means of a calibration curve
(always work under identical conditions!)

Limit

0.1 mg chromium per sample.