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METHODS FOR THE ANALYSIS OF IRON AND STEEL

Part 18—DETERMINATION OF PHOSPHORUS (SPECTROPHOTOMETRIC METHOD)

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The following interests are represented on Committee CH/10:

Aluminium Development Council
Australasian Institute of Mining and Metallurgy
Australian Lead Development Association
Australian Mineral Development Laboratories
Australian Tin Information Centre
Australian Zinc Development Association
Bureau of Steel Manufacturers of Australia
Confederation of Australian Industry
Copper Producers Association of Australia
Department of Defence
Electricity Supply Association of Australia
Metal Trades Industry Association of Australia
National Association of Testing Authorities
Railways of Australia Committee
Royal Australian Chemical Institute

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PREFACE

This standard was prepared by the Association's Committee on the Analysis of Metals under the direction of the Chemical Standards Board as a further part of AS 1050. It supersedes AS K1, Part 18—1963.

Whereas the previous method used a titrimetric or gravimetric finish, this method uses a spectrophotometric finish based on that given in the draft revision of ISO 2732 (doc ISO/TC 17/SC 1 N463).

The committee organized an inter-laboratory test program to obtain information on the repeatability and reproducibility of the method. Laboratories from the following organizations participated in the test program to provide the data given in Table 1:

Australian Iron and Steel Pty Ltd, Port Kembla (2 analysts)
Commonwealth Steel Co. Ltd
State Rail Authority, N.S.W.
The Broken Hill Proprietary Co. Ltd, Newcastle
The Broken Hill Proprietary Co. Ltd, Whyalla

STANDARDS ASSOCIATION OF AUSTRALIA

Australian Standard

METHODS FOR THE ANALYSIS OF IRON AND STEEL

PART 18—DETERMINATION OF PHOSPHORUS (SPECTROPHOTOMETRIC METHOD)

1 SCOPE. This standard sets out a spectrophotometric method for the determination of the phosphorus content of iron and steel.

2 APPLICATION. The method is applicable to the determination of phosphorus contents between 0.005 percent and 2.0 percent, provided that the following elements are not present in amounts greater than that specified:

niobium	1 percent
tantalum	1 percent
titanium	2 percent
tungsten	11 percent
zirconium	1 percent

3 REFERENCED DOCUMENTS. The following standards are referred to in this standard:

AS 1213 Iron and Steel—Methods of Sampling

AS 2164 One-mark Volumetric Flasks

AS 2166 One-mark Pipettes

AS 2167 Straight Pipettes

AS CK19 Code of Recommended Practice for the Chemical Analysis of Materials by Ultraviolet Visible Spectrophotometry

BS 4237 Report on Reproducibility of Methods of Chemical Analysis Used in the Iron and Steel Industry.

4 PRECISION. A planned trial of the method was carried out in accordance with BS 4237. The reproducibility index ($2s$) is obtained from the following formula:

$$2s = 2\sqrt{(s_b^2 + s_w^2)}$$

where

s_b = between-operator standard deviation

s_w = within-operator standard deviation.

95 percent of the results obtained by any one analyst should be reproducible to within two standard deviations of the overall mean value derived from all laboratories (i.e. $\bar{x} \pm 2s$).

For further information, see BS 4237.

The planned trial was carried out by six analysts, from five different laboratories. Five tests were carried out by each analyst on each of five samples.

From the results obtained, the 95 percent confidence limits ($2s$, Table 1) have been calculated.

TABLE 1

PRECISION DATA FOR PHOSPHORUS DETERMINATIONS

Sample type	Phosphorus certificate value percent	Phosphorus content found percent	Components of standard deviation		Reproducibility index $2s$
			s_b	s_w	
SRM106 b Steel (Al 1, Cr 1, Mo 0.2)	0.008	0.007	0.0011	0.0003	0.0023
BCS 483 Tool steel (Co 2, Cr 3, V 0.5, W 11)	0.019	0.019	0.0006	0.0007	0.0018
BCS 404 Low alloy steel (Cr 0.7, Cu 0.3, Ni 0.5, Si 1)	0.050	0.049	0.0011	0.0009	0.0028
SRM 6f Cast iron	0.53	0.517	0.009	0.008	0.023
BCS 206/3 Cast iron (Si 3)	1.63	1.62	0.022	0.021	0.061

5 PRINCIPLE. Dissolution of the test portion in an oxidizing acid mixture followed by conversion of the phosphorus to phosphovanadomolybdate in perchloric acid/nitric acid solution. Extraction of the phosphovanadomolybdate into 4-methyl-2-pentanone with citric acid present to complex any arsenic. Spectrophotometric measurement at a wavelength of about 425 nm.

6 REAGENTS.

6.1 General. Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

Carry out blank tests to verify that all reagents contain a minimum amount of phosphorus. If the result of the blank test (Clause 9.3) is >0.005 percent phosphorus, the cause of high phosphorus content should be located and the particular reagent replaced by one with a much lower phosphorus content.

6.2 Solids.

6.2.1 *Iron metal.* High purity iron metal.

6.3 Solutions.

6.3.1 *Hydrochloric acid* (ρ_{20} 1160 kg/m³ to 1180 kg/m³).

6.3.2 *Nitric acid* (ρ_{20} 1420 kg/m³).

6.3.3 *Nitric acid* (200 mL/L). To 400 mL of water add 100 mL of nitric acid (ρ_{20} 1420 kg/m³).

6.3.4 *Perchloric acid* ($\rho_{20} \sim$ 1540 kg/m³).

NOTES:

1. Particular care should be taken to ensure that this acid has a low phosphorus content (see Clause 6.1).
2. Acid with a density of about 1670 kg/m³ may be used. 100 mL of this acid is equivalent to 127 mL of perchloric acid with a density of 1540 kg/m³.

6.3.5 *Citric acid solution* (500 g/L). Dissolve 500 g of citric acid ($C_6H_8O_7.H_2O$) in water, dilute to 1 L and mix.

6.3.6 *4-Methyl-2-pentanone* (isobutyl methyl ketone).

6.3.7 *Ammonium molybdate solution* (150 g/L). Dissolve 15 g of hexammonium heptamolybdate tetrahydrate [$(NH_4)_6Mo_7O_{24}.4H_2O$] in water, dilute to 100 mL and mix.

6.3.8 *Ammonium metavanadate solution* (2.5 g/L). Dissolve 2.5 g of ammonium metavanadate (NH_4VO_3) in water, dilute to 1 L and mix.

6.3.9 *Boron fluoride solution* (40 g/L). Dissolve 40 g of boric acid (H_3BO_3) in 300 mL of water in a plastics beaker. Add 100 mL of hydrofluoric acid (40% V/V, ρ_{20} 1130 kg/m³), dilute to 1 L and mix. Store the solution in a plastics bottle.

6.3.10 *Potassium permanganate solution* (10 g/L). Dissolve 10 g of potassium permanganate ($KMnO_4$) in water. Dilute to 1 L and mix.

6.3.11 *Sodium nitrite solution* (50 g/L). Dissolve 50 g of sodium nitrite ($NaNO_2$) in water. Dilute to 1 L and mix.

6.4 Standard solutions.

6.4.1 *Stock phosphorus solution* (0.1 g of phosphorus per litre). Weigh, to the nearest 0.0001 g, 0.4393 g of potassium dihydrogen orthophosphate (KH_2PO_4) previously dried to constant mass at 105°C and cooled in a desiccator.

Dissolve in water, transfer the solution quantitatively to a 1 L volumetric flask, dilute to the mark and mix.

1 mL of this solution contains 0.1 mg of phosphorus.

6.4.2 *Standard phosphorus solution* (0.01 g of phosphorus per litre). Transfer 100.0 mL of the stock solution (6.4.1) to a 1 L volumetric flask, dilute to the mark with water and mix.

1 mL of this solution contains 0.01 mg of phosphorus.

7 APPARATUS.

7.1 Volumetric glassware. Grade A volumetric glassware complying with AS 2164, AS 2166 and AS 2167 shall be used.

7.2 Spectrophotometer. A spectrophotometer capable of measuring absorbance with a spectral slit width of less than 5 nm at 425 nm shall be used. The wavelength measurements shall be accurate to ±2 nm or less. In the absorbance range 0.05 to 0.85 absorbance measurements shall be repeatable to ±0.003 absorbance or better.

7.3 Spectrophotometric cells. Plain type cells as specified in BS 3875 shall be used.

8 SAMPLING. Samples shall be taken by the procedures specified in AS 1213.

9 PROCEDURE.

WARNING. Perchloric acid vapour may cause explosions in the presence of ammonia, or organic material.

9.1 Test portion. Weigh to the nearest 0.001 g, approximately 0.5 g of the test sample.