BSI

金属的分析试验

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METHODS FOR THE
ANALYSIS OF NICKEL
FOR USE IN ELECTRONIC TUBES AND VALVES

Part 1

Determination of Aluminium

(Photometric Method)

B.S. 3727 : Part 1 : 1966

Price 3/- net

BRITISH STANDARDS INSTITUTION

INCORPORATED BY ROYAL CHARTER

BRITISH STANDARDS HOUSE, 2 PARK ST., LONDON, W.1

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THIS BRITISH STANDARD, having been approved by the Telecommunication Industry Standards Committee and endorsed by the Chairman of the Engineering Divisional Council, was published under the authority of the General Council on 12th December, 1966.

The Institution desires to call attention to the fact that this British Standard does not purport to include all the necessary provisions of a contract.

In order to keep abreast of progress in the industries concerned, British Standards are subject to periodical review. Suggestions for improvements will be recorded and in due course brought to the notice of the committees charged with the revision of the standards to which they refer.

A complete list of British Standards, numbering over 4000, fully indexed and with a note of the contents of each, will be found in the British Standards Yearbook, price 15s. The B.S. Yearbook may be consulted in many public libraries and similar institutions.

This standard makes reference to the following British Standard:
B.S. 1499. Sampling non-ferrous metals.

British Standards are revised, when necessary, by the issue either of amendment slips or of revised editions. It is important that users of British Standards should ascertain that they are in possession of the latest amendments or editions.

The following B.S.I. references relate to the work on this standard:
Committee references TLE/5, TLE/5/3 and TLE/5/3/1
Draft for comment D65/9196

CO-OPERATING ORGANIZATIONS

The Telecommunication Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

British Broadcasting Corporation
British Electrical and Allied Manufacturers' Association
*British Radio Equipment Manufacturers' Association
*British Radio Valve Manufacturers' Association
British Railways Board
Cable and Wireless Ltd.
Crown Agents for Overseas Governments and Administrations
Electrical Contractors Association (Incorporated)
Electrical Research Association
Electricity Council, the Generating Board and the Area Boards in England and Wales
*Electronic Engineering Association
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*Post Office
Radio and Electronic Component Manufacturers' Federation
Relay Services Association of Great Britain
Science Research Council—Radio and Space Research Station
Telecommunication Engineering and Manufacturing Association

The Government departments and scientific and industrial organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this standard:

Scientific Instrument Manufacturers' Association
United Kingdom Atomic Energy Authority
Manufacturers of nickel alloys

BRITISH STANDARD METHODS FOR THE ANALYSIS OF NICKEL FOR USE IN ELECTRONIC TUBES AND VALVES

Part 1. Determination of Aluminium (Photometric Method)

FOREWORD

This method for the determination of aluminium is the first of a series of methods which will form a complete British Standard under the collective title 'Analysis of nickel for use in electronic tubes and valves', each method being published as a separate part. Other parts in the series are as follows:

*Part 2. Boron
Part 3. Carbon
Part 4. Chromium
Part 5. Cobalt
Part 6. Copper
Part 7. Iron
Part 8. Manganese
*Part 9. Magnesium
Part 10. Silicon 0·02-0·25 per cent
Part 11. Silicon 0·001-0·02 per cent
*Part 12. Sulphur
Part 13. Titanium
Part 14. Tungsten
*Part 15. Zinc
Part 16. Determination of free and combined magnesium
Part 17. } Not yet allocated
Part 18. }
Part 19. }
Part 20. Spectrographic method
Part 21. Magnesium (atomic absorption method)
Part 22. Zinc (atomic absorption method)

These methods have been found to give reliable and reproducible results and are primarily intended as reference methods to be used in cases of dispute.

INTRODUCTION

a. Principle. The sample is decomposed by nitric acid and, after fuming with sulphuric acid, most interfering elements are complexed by cyanide and sulphide in alkaline buffered solution. Aluminium and magnesium are extracted into chloroform as the oxine complexes and the magnesium interference is overcome by means of EDTA solution. The optical density of the aluminium oxine complex is then determined.

b. Range. 0·002 to 0·10 per cent aluminium.

c. Reproducibility. Experiments have been carried out independently by a number of analysts, using the method recommended in this standard. The degree of reproducibility that can be expected is shown by the following analysis of the results obtained:

*In course of preparation.

Aluminium content per cent	Standard deviation
0·01	0·001
0·05	0·0025

d. Application. This method is applicable to nickel primarily intended for use in electronic devices. Such nickel contains not more than:

Aluminium	0·1 per cent
Chromium	0·03 per cent
Cobalt	1 per cent
Copper	0·2 per cent
Iron	0·25 per cent
Manganese	0·25 per cent
Magnesium	0·15 per cent
Silicon	0·25 per cent
Titanium	0·03 per cent
Tungsten	5 per cent

APPARATUS

a. Class A volumetric glassware shall be used throughout complying with the relevant British Standard.

b. Any instrument suitable for measuring the optical density of the solution at a wavelength of 390 $m\mu$ may be used. Cell size 4 cm or 2 cm.

REAGENTS REQUIRED

All reagents shall be of suitable purity*, distilled water shall be used throughout and the solutions shall be freshly prepared.

Standard aluminium solution. Dissolve 500 mg of pure aluminium in 40 ml of hydrochloric acid (50 per cent v/v), then dilute with water to one litre. Transfer 20 ml of this primary aluminium solution to a one litre volumetric flask, add 5 ml of hydrochloric acid (50 per cent v/v) and dilute to the mark with water to give the standard aluminium solution (1 ml contains 10 μg of aluminium).

Hydrochloric acid (50 per cent v/v). Dilute 500 ml of hydrochloric acid (*d* 1·16-1·18) to 1 litre with water.

Sulphuric acid (1 per cent v/v). Add 10 ml of sulphuric acid (*d* 1·84) to 200 ml of water and dilute to 1 litre with water.

Nitric acid (50 per cent v/v). Dilute 500 ml of nitric acid (*d* 1·42) to 1 litre with water.

Sulphuric acid (d 1·84).

*Analytical grade reagents have been found suitable.

Thymol blue indicator (0·01 per cent w/v). Dissolve 0·01 g of thymol blue indicator in 20 ml of methanol and dilute to 100 ml with water.

Ammonia solution (50 per cent v/v). Dilute 500 ml of ammonium hydroxide (*d* 0·88) to 1 litre with water.

Potassium cyanide solution (10 per cent w/v). Dissolve 100 g of potassium cyanide in water and dilute to 1 litre.

Sodium sulphide solution (10 per cent w/v). Dissolve 100 g of sodium sulphide in water and dilute to 1 litre.

Ammonium sulphate. Solid.

Oxine solution (1·5 per cent w/v). Dissolve 1·5 g of recrystallised 8-hydroxyquinoline (Note 1) in 100 ml chloroform.

EDTA solution (0·1 per cent w/v). Dissolve 0·1 g ethylene-diamine tetra-acetic acid (disodium salt) in water and dilute to 100 ml.

Sodium sulphate (anhydrous). Solid.

SAMPLING

Recommended methods of obtaining a suitable sample for the analytical procedure given below are described in B.S. 1499, 'Sampling non-ferrous metals'.

CALIBRATION

Transfer 20 ml of sulphuric acid (1 per cent v/v) to each of six 100 ml beakers and make additions of standard aluminium solution as follows: 0, 1·0, 2·0, 3·0, 4·0 and 5·0 ml.

Add 2 or 3 drops of thymol blue indicator solution (0·01 per cent w/v) and neutralise to pH 9 - 9·5 using ammonia solution (50 per cent v/v).

Add 5 ml of potassium cyanide solution (10 per cent w/v) and heat on a boiling water bath. Add 5 ml of sodium sulphide solution (10 per cent w/v) and allow to stand for 3 minutes. Cool. Add 2 g of ammonium sulphate and transfer to a 150 ml separating funnel, washing the beaker with water and adjusting the volume to 50 ml ± 5 ml in the separating funnel.

Add 10 ml of oxine solution (1·5 per cent w/v), insert the stopper and shake vigorously for 3 minutes (Note 2). Add 5 ml of EDTA solution (0·1 per cent w/v) and shake for a further 3 minutes. Allow the layers to separate and run off the chloroform layer into a 25 ml volumetric flask. Add 5 ml of chloroform to the separating funnel, swirl, allow to separate and then rinse the stem of the funnel, collecting the chloroform wash in the 25 ml volumetric flask. Dilute to 25 ml with chloroform, add 0·5 g of anhydrous sodium sulphate and mix.

Measure the optical density at 20 ± 1°C using the conditions specified under 'Apparatus'. Use the blank solution in the compensating cell.

Plot the optical density values obtained against percentage aluminium and prepare calibration graphs for 2 cm cells (0·0 - 0·10 per cent aluminium) and for 4 cm cells (0·0 - 0·05 per cent aluminium).

PROCEDURE

Weigh 500 mg of the sample and transfer to a 150 ml beaker. Carry a blank determination through the entire procedure. Add 10 ml of nitric acid (50 per cent v/v), warm gently until dissolved, add 2 ml of sulphuric acid (*d* 1·84) and carefully evaporate to fumes of sulphuric acid. Cool, add about 50 ml of water and warm to dissolve the nickel salts. Filter through a suitable filter paper* into a 200 ml volumetric flask and wash with water. Discard the filter paper. Cool the solution and dilute the filtrate to 200 ml.

Transfer a 20 ml portion (= 50 mg of sample) to a 100 ml beaker. Add 2 or 3 drops of thymol blue indicator solution (0·01 per cent w/v), and neutralise to pH 9 - 9·5 using ammonia solution (50 per cent).

Proceed as described under 'Calibration'.

Obtain the percentage aluminium in the sample by reference to the appropriate calibration graph.

NOTES

1. *Recrystallization of 8-hydroxyquinoline*. Dissolve 40 g of 8-hydroxyquinoline in 100 ml of methanol at 55°C. Filter through a filter paper* into a 250 ml beaker, discard the filter paper and allow the filtrate to cool to room temperature. Filter through a sintered glass crucible (porosity No. 1) and rinse the crystals with three small washes of methanol. Transfer the crystals to a large watch glass and dry in a desiccator.
2. The complexing and extraction stage should be carried out in diffused light. Strong sunlight causes a darkening of the extract.

*A Whatman No. 541 paper has been found satisfactory.

BRITISH STANDARDS INSTITUTION

The British Standards Institution was founded in 1901 and incorporated by Royal Charter in 1929.

The principal objects of the Institution as set out in the charter are to co-ordinate the efforts of producers and users for the improvement, standardization and simplification of engineering and industrial materials; to simplify production and distribution; to eliminate the waste of time and material involved in the production of an unnecessary variety of patterns and sizes of articles for one and the same purpose; to set up standards of quality and dimensions, and to promote the general adoption of British Standards.

In carrying out its work the Institution endeavours to ensure adequate representation of all viewpoints. Before embarking on any project it must be satisfied that there is a strong body of opinion in favour of proceeding and that there is a recognized need to be met.

The Institution is a non-profit-making concern. It is financed by subscriptions from firms, trade associations, professional institutions and other bodies interested in its work, by a Government grant and by the sale of its publications. The demands on the services of the Institution are steadily increasing and can only be met if continuing and increased financial support is provided.

Membership of the Institution is open to British subjects, companies, technical and trade associations, and local and public authorities.

(Printed in England by I.L. Ltd.)

METHODS FOR THE

ANALYSIS OF NICKEL

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TUBES AND VALVES

Part 2

Determination of Boron

(Photometric Method)

BS 3727 : Part 2: 1968

Price 4/- net

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INCORPORATED BY ROYAL CHARTER

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THIS BRITISH STANDARD, having been approved by the Telecommunication Industry Standards Committee and endorsed by the Chairman of the Engineering Divisional Council, was published under the authority of the General Council on 8th February, 1968.

SBN: 580 00055 9

This standard makes reference to the following British Standards:

BS 1499. Sampling non-ferrous metals.
BS 3591. Industrial methylated spirits.

The following BSI references relate to the work on this standard:
Committee references TLE/5, TLE/5/3 and TLE/5/3/1
Draft for comment 66/18108

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Ministry of Defence, Army Department
*Ministry of Defence, Navy Department
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Part 2. Determination of Boron (Photometric Method)

FOREWORD

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Part 1. Aluminium
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Part 18. }
Part 19. }
Part 20. Spectrographic method
Part 21. Magnesium (atomic absorption)
Part 22. Zinc (atomic absorption)

These methods have been found to give reliable and reproducible results and are primarily intended as reference methods to be used in cases of dispute.

1. INTRODUCTION

1.1 Principle. The sample is decomposed by perchloric acid and the resulting solution treated with a solution of curcumin in glacial acetic acid. Sulphuric acid and acetic acid are then added and the mixture diluted to volume with industrial methylated spirit. The optical density of the boron-curcumin complex is then measured.

1.2 Range. 1 - 10 p.p.m., using 4 cm cells.
1 - 25 p.p.m., using 1 cm cells.

*In course of preparation.