भारतीय मानक

इस्पात के रासायनिक विश्लेषण की पद्धतियाँ

भाग 24 अक्रिय गैस संलयन द्वारा इस्पात में नाइट्रोजन का निर्धारण — तापीय चालकता पद्धति (नाइट्रोजन 0.001 से 0.2 प्रतिशत)

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF STEELS

PART 24 DETERMINATION OF NITROGEN IN STEEL BY INERT GAS FUSION — THERMAL CONDUCTIVITY METHOD (NITROGEN 0.001 TO 0.2 PERCENT)

ICS 77.080.20

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

May 2003

Price Group 1

FOREWORD

This Indian Standard (Part 24) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 228, which was first published in 1952 and subsequently revised in 1959, covered the chemical analysis of plain carbon and low alloy steels, alongwith pig iron and cast iron. It was revised again to make it comprehensive in respect of steel analysis and to exclude pig iron and cast iron which were being covered in separate standards. During its second revision the standard has been split up in several parts.

Determination of nitrogen in steel by steam distillation, has been covered in IS 228 (Part 19) and Determination of nitrogen in steel by optical emission spectrometer is under preparation and will be covered in a separate standard which will form a part of the above series.

This part covers the method for determination of nitrogen (0.001-0.2 percent) in steel by inert gas fusion thermal conductivity method. The other parts of this series are:

(Part 1) : 1987	Determination of carbon by volumetric method (for carbon 50 percent) (*third revision*)
(Part 2) : 1987	Determination of manganese in plain carbon and low alloy steels by arsenite method (*third revision*)
(Part 3) : 1987	Determination of phosphorus by alkali-metric method (*third revision*)
(Part 4) : 1987	Determination of total carbon by gravimetric method (for carbon greater than or equal to 0.1 percent) (*third revision*)
(Part 5) : 1987	Determination of nickel by dimethyl glyoxime (gravimetric) method (for nickel greater than or equal to 0.1 percent) (*third revision*)
(Part 6) : 1987	Determination of chromium by persulphate oxidation method (for chromium greater than or equal to 0.1 percent) (*third revision*)
(Part 7) : 1990	Determination of molybdenum by alphabenzoinoxime method (for molybdenum greater than 1 percent) (*third revision*)
(Part 8) : 1989	Determination of silicon by gravimetric method (for silicon 0.05 to 0.50 percent) (*third revision*)
(Part 9) : 1989	Determination of sulphur in plain carbon steels by evolution method (for sulphur 0.01 to 0.25 percent) (*third revision*)
(Part 10) : 1989	Determination of molybdenum by thiocyanate (photometric) method in low and high alloy steels (for molybdenum 0.01 to 1.5 percent) (*third revision*)
(Part 11) : 1990	Determination of total silicon by reduced molybdosilicate spectrophotometric method in carbon steels and low alloy steels (for silicon 0.01 to 0.05 percent) (*third revision*)
(Part 12) : 1988	Determination of manganese by periodate spectrophotometric method in low and high alloy steels (for manganese 0.01 to 2.0 percent) (*third revision*)
(Part 13) : 1982	Determination of arsenic
(Part 14) : 1988	Determination of carbon by thermal conductivity method (for carbon 0.005 to 2.000 percent)
(Part 15) : 1992	Determination of copper by thiosulphate iodide method (for copper 0.05 to 5 percent)

(*Continued on third cover*)

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF STEELS

PART 24 DETERMINATION OF NITROGEN IN STEEL BY INERT GAS FUSION — THERMAL CONDUCTIVITY METHOD (NITROGEN 0.001 TO 0.2 PERCENT)

1 SCOPE

This standard (Part 24) covers the determination of nitrogen in steel in the range 0.001 to 0.2 percent.

2 SAMPLE PREPARATION

The sample may be in the form of chips, drillings or solid pin. The size of the solid pin should be such to permit free introduction through the loading device of the equipment or directly into the graphite crucible. The sample may be cut for nitrogen analysis in such a way that excess heat is not generated. A hacksaw may be used for cutting. If drillings so desired, they should be taken under argon atmosphere avoiding excess heat. The sample to be analyzed may be rinsed in acetone, air-dried and used.

3 PRINCIPLE OF THE METHOD

The sample, contained in a small graphite crucible, is fused under a flowing helium atmosphere. Nitrogen present in the steel is released as molecular nitrogen into the flowing helium stream. The nitrogen is separated from other liberated gases such as hydrogen and carbon monoxide and finally measured by thermal conductivity.

4 APPARATUS

4.1 Any commercial analyzer equipped to carry out the above operations automatically may be used.

4.2 Graphite Crucible

High purity graphite crucible of the size recommended by the manufacturer of the instrument.

4.3 Crucible Tongs

Capable of handling recommended crucibles.

5 REAGENTS

5.1 Helium (99.99 Percent and Above Purity)

5.2 Magnesium Perchlorate (Commonly known as Anhydrone)

5.3 Sodium Hydroxide on Asbestos (Commonly known as Ascarite)

5.4 Rare Earth Copper Oxide

6 CALIBRATION BY STANDARD

6.1 Switch on the instrument and allow the time recommended by the equipment manufacturer for stabilization before use.

6.2 Purge the gas line and the thermal conductivity cell for sometime for the time period as recommended by the manufacturer before starting the analysis.

6.3 Set the analyzer to operate in automatic or manual mode depending upon the nature of the sample.

NOTE — If the standard or sample is in pin form, calibrate with pin standard by automatic mode and subsequently analyze the sample also in automatic mode. However, if the sample/standard is in the form of drillings, calibrate by manual mode and also subsequently analyze the sample in manual mode.

6.4 Weigh the standard sample and enter the weight.

6.5 Place the weighed standard sample in pin form in the loading device for automatic analysis.

6.6 Place an empty graphite crucible on the furnace lower electrode assembly and close the furnace. In automatic mode, the analysis begins automatically and at the end displays the percentage nitrogen on the control console and also prints on the paper. However, in manual mode, after closing the furnace, press the analyzer switch. The purging and degassing will start. Then lower the piston, keep weighed standard in the graphite crucible and again close the furnace. Now press again the analyzer switch. After the analysis cycle, the percentage of nitrogen will get displayed on the control console and also will give a print out.

6.7 Lower the position, remove the used crucible and follow instructions of the manufacturer for cleaning the furnace before starting the next analysis.

6.8 Repeat the analysis of the standard three times and take the mean value as the percentage of nitrogen.

6.9 Calibrate the instrument as given in the instrument manual. Until the certified value of nitrogen is obtained with desired reproducibility.

6.10 Verify the response of the instrument by analyzing a standard sample, after calibration. The value should be within the allowable limits of the certified value of the standard. If not, repeat the calibration and verification.

NOTE — Repeat the calibration when a different lot of crucible is used or the system has not been used for a long time or when the carrier gas has been changed.

6.11 Some instruments have provision for calibration by nitrogen gas dosing. Then carry on calibration by gas dosing as suggested by the manufacturer of the instrument and finally verify the validity of calibration by analyzing a certified reference standard sample.

7 ANALYSIS OF SAMPLE

Weigh around 1 g of the unknown sample enter the weight and carry out the analysis of the sample for nitrogen content by automatic/manual mode as given in **6.5** to **6.7**.

NOTE — Ensure that the calibration standards are of same or similar composition as the sample to be analyzed.

8 PRECISION

The precision of analysis should be within ±10 percent.

(*Continued from second cover*)

(Part 16) : 1992	Determination of tungsten by spectrophotometric method (for tungsten 0.1 to 2 percent)
(Part 17) : 1998	Determination of nitrogen by thermal conductivity method
(Part 18) : 1998	Determination of oxygen by instrumental method
(Part 19) : 1998	Determination of nitrogen by steam distillation
(Part 20) : 1987	Determination of carbon and sulphur by infra-red absorption method
(Part 21) : 1987	Determination of copper by spectrometric method (for copper 0.02 to 0.5 percent) (*third revision*)
(Part 22) : 2003	Determination of total hydrogen in steel by thermal conductivity method (hydrogen 0.1 ppm to 50 ppm)
(Part 23) : 2003	Determination of total nitrogen in steel by optical emission spectrometer (nitrogen 0.002 to 1.0 percent)

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act,* 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc : No. MTD 2 (4272).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110 002
Telephones : 2323 0131, 2323 3375, 2323 9402

Telegrams: Manaksanstha
(Common to all offices)

Regional Offices :	Telephone
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110 002	2323 7617, 2323 3841
Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Kankurgachi, KOLKATA 700 054	2337 8499, 2337 8561, 2337 8626, 2337 9120
Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160 022	60 3843, 60 9285
Southern : C. I. T. Campus, IV Cross Road, CHENNAI 600 113	2254 1216, 2254 1442, 2254 2519, 2254 2315
Western : Manakalaya, E9 MIDC, Marol, Andheri (East), MUMBAI 400 093	2832 9295, 2832 7858, 2832 7891, 2832 7892

Branches : AHMEDABAD. BANGALORE. BHOPAL. BHUBANESHWAR. COIMBATORE. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. KANPUR. LUCKNOW. NAGPUR. NALAGARH. PATNA. PUNE. RAJKOT. THIRUVANANTHAPURAM. VISAKHAPATNAM.

Printed at New India Printing Press, Khurja, India