भारतीय मानक

इस्पात के रासायनिक विश्लेषण की पद्धतियाँ

भाग 22 तापीय चालकता पद्धति द्वारा इस्पात में कुल हाइड्रोजन का निर्धारण (हाइड्रोजन 0.1 पीपीएम से 50 पीपीएम)

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF STEELS

PART 22 DETERMINATION OF TOTAL HYDROGEN IN STEEL BY THERMAL CONDUCTIVITY METHOD (HYDROGEN 0.1 ppm TO 50 ppm)

ICS 77.080.20

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Price Group 1

FOREWORD

This Indian Standard (Part 22) 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.

This part covers the method for determination of total hydrogen in steel by 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)
(Part 16) : 1992	Determination of tungsten by spectrophotometric method (for tungsten 0.1 to 2 percent)

(*Continued on third cover*)

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF STEELS

PART 22 DETERMINATION OF TOTAL HYDROGEN IN STEEL BY THERMAL CONDUCTIVITY METHOD (HYDROGEN 0.1 ppm TO 50 ppm)

1 SCOPE

This standard (Part 22) covers the determination of total (diffusible and residual) hydrogen in steel (0.1 ppm to 50 ppm).

2 SAMPLING

2.1 The sample should be collected in a suitable metal sampler as prescribed by the instrument manufacturer.

2.2 Immerse the sampler in the molten metal for 2 to 4 s to allow the sample to be drawn into the inner chamber. Excessive immersion time may cause misleading results. Immersion depth must be a minimum of 51 mm into the molten metal.

2.3 Immediately after removing the sampler from the molten metal, plunge the end of the sampler into cold water. Agitate it to hasten cooling.

3 PRINCIPLE

In steel the total hydrogen is present in two forms as diffusible hydrogen and the residual hydrogen. The diffusible hydrogen is the hydrogen which diffuses out during the solidification of the sample. This is contained within the sample tube. During the analysis for the diffusible hydrogen, the sample tube is pierced and the diffused hydrogen is swept out with nitrogen and measured. The residual hydrogen is the hydrogen which is remaining after the solidification of sample. This hydrogen is released by heating the sample in a nitrogen atmosphere. Carbon dioxide and moisture released along with hydrogen are removed by suitable absorbent. Hydrogen is then measured by thermal conductivity method.

4 APPARATUS

Any commercial analyzer can be used consisting of essentially furnace and a measurement unit.

5 REAGENTS

5.1 Nitrogen Gas (Above 99.99 Percent Purity)

5.2 Hydrogen Gas (Above 99.99 Percent Purity)

5.3 Anhydrous Magnesium Perchlorate (Commonly known as Anhydrone)

5.4 Sodium Hydroxide on Asbestos (Commonly known as Ascarite)

5.5 Copper Turnings

6 CALIBRATION

6.1 Calibration by Standard

6.1.1 Prepare and stabilize the instrument as per the instructions laid down by the manufacturer.

6.1.2 Use certified reference materials of the desired concentration range for calibration.

6.1.3 Weigh the sample and follow the calibration procedure as laid down in the operation manual of the instrument and establish the instrument response.

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

NOTE — Repeat the calibration when the carrier gas supply has been changed or when the system has not been used for long time.

6.2 Calibration by Gas Dosing

Some instruments have provision for calibration by hydrogen gas dosing. Follow the procedure suggested by the manufacturer of the instrument for calibration by analyzing a certified reference standard sample.

7 ANALYSIS

7.1 Analyze the samples collected using the patented sampler for diffusible and residual hydrogen content.

7.2 Diffusible Hydrogen

7.2.1 Insert the sample tube into the piercing unit of the instrument. The analysis cycle starts automatically. The diffused hydrogen contained in the sample tube is carried by the carrier gas to the detector and displayed as diffused hydrogen.

7.3 Residual Hydrogen

7.3.1 After the completion of the diffusible hydrogen analysis, take out the sample tube from the piercing unit and cut it to get out the pin sample, following

the manufacturer's recommendations.

NOTE — The residual hydrogen is determined by heating the sample in a furnace in nitrogen atmosphere. The furnace normally is a tubular furnace capable of generating a temperature of 1 100°C. It shall be provided with proper temperature measurement and control devices.

7.3.2 Weigh the pin sample and enter the weight in the system. Put the sample in the reaction tube and start the analysis. The sample is heated to a temperature of 1 000°C to 1 050°C and the residual hydrogen liberated is carried by the carrier gas and measured.

8 REPRODUCIBILITY

The reproducibility/precision of analysis should be within ± 10percent.

(*Continued from second cover*)

(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 23) : 2003	Determination of nitrogen in steel by optical emission spectrometer (nitrogen 0.002 to 1.0 percent)
(Part 24) : 2003	Determination of nitrogen in steel by inert gas fusion — Thermal conductivity method (nitrogen 0.001 to 0.2 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'.

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Amendments Issued Since Publication

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