IS 228 (Part 23) : 2003

भारतीय मानक

इस्पात के रासायनिक विश्लेषण की पद्धतियाँ

भाग 23 प्रकाशिक उत्सर्जन स्पेकट्रमी मापी द्वारा इस्पात में कुल नाइट्रोजन का निर्धारण (नाइट्रोजन 0.002 से 1.0 प्रतिशत)

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF STEELS

PART 23 DETERMINATION OF TOTAL NITROGEN IN STEEL BY OPTICAL EMISSION SPECTROMETER (NITROGEN 0.002 TO 1.0 PERCENT)

ICS 77.080.20

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Price Group 1

Methods of Chemical Analysis of Ferrous Metals Sectional Committee, MTD 2

FOREWORD

This Indian Standard (Part 23) 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 inert gas fusion — Thermal conductivity method 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 total nitrogen (0.002-1.0 percent) in steel by optical emission spectrometer. 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 23 DETERMINATION OF TOTAL NITROGEN IN STEEL BY OPTICAL EMISSION SPECTROMETER (NITROGEN 0.002 TO 1.0 PERCENT)

1 SCOPE

This standard (Part 23) covers the determination of nitrogen (0.002 - 1.0 percent) in steel by optical emission spectrometer.

2 SAMPLING

2.1 The sample is collected in a mould cavity that provides a quickly chilled surface through rapid solidification. It should be homogeneous and free from fractures and voids. Alternatively sampling probes may be used to collect the sample.

2.2 Typical sample sizes are as follows:

a) Diameter : 20-50 mm, and
b) Thickness : 10-20 mm.

2.3 The sample is prepared on an abrasive belt or disc grinder, taking extreme care that the sample does not become too hot. It is desirable to use automatic polishing machine to ensure consistency of polish. The most commonly used abrasives are alumina, silicon carbide or zirconia of 60 grit.

3 PRINCIPLE OF THE TEST METHOD

A capacitor discharge is produced between polished surface of the sample and a conical shaped electrode. The discharge is terminated at a predetermined time. The radiant energy of the nitrogen line and an internal standard line are converted into electrical energy by means of photomultiplier tubes. The concentration of nitrogen in steel is obtained by reference to standard.

4 APPARATUS

4.1 Spectrometer

Any commercially available optical emission spectrometer with a facility to measure the nitrogen emission intensity can be used.

4.2 Argon Flushing System

The system consists of high purity argon cylinders, with argon content above 99.995 percent, a two stage leak proof regulator, a gas flow meter and connecting copper/stainless steel tubing of 4-6 mm diameter and rare gas purifier to provide inert gas atmosphere during sparking.

NOTE — The argon gas purifier system uses heated titanium, copper oxide and molecular sieves to absorb the impurities like nitrogen, oxygen, hydrocarbons, etc.

4.3 UV Optics

The UV optic, situated in a chamber located directly behind the spark stand, is filled with nitrogen at atmospheric pressure. The nitrogen is circulated through the tank and the gas purifier by a membrane pump. The isolated gas system provides an atmosphere capable of transmitting wave lengths down to 140 nm. Alternatively, any optics in a spectrometer capable for measuring the nitrogen emission intensity may be used.

NOTE — The most sensitive line of nitrogen having wavelength 149 nm lies in the vacuum ultraviolet region. Any typical iron line is used as the internal standard.

4.4 Exposure Conditions

The preflush, preburn and integration period during the spark may be followed as per the instructions in the operation manual of the manufacturer of the instrument.

4.5 Environment Control

The optical emission spectrometer should be housed in an air conditioned room maintained at a temperature specified by the manufacturer with a relative humidity of 50 ± 5 percent. The temperature should be kept within ± 2°C of the nominal, to reduce the drift in spectral lines due to temperature variation. The room should be free from vibrations, dust and oil vapours.

4.6 Counter Electrode

The pointed thoriated tungsten electrode used as counter electrode may be reshaped, when necessary. The electrode tip is cleaned with a brush after every spark.

NOTE — The spark stand, lens or quartz windows are to be cleaned regularly to minimize the transmission losses. Frequency of cleaning is to be established by individual laboratories depending upon the volume of work and the deposits.

5 CALIBRATION

5.1 Optimize the position of the entrance window by carrying out profiling with a high nitrogen bearing steel sample.

5.2 Select 5 to 10 reference steel standards that cover the required concentration range of nitrogen. Spark these samples and construct the analytical curve as per instructions in the operation manual of the manufacturer of the instrument.

6 STANDARDIZATION

6.1 Standardize the instrument as per the operation manual of the manufacturer of the instrument. The frequency of standardization will depend on the long term stability of the instrument.

6.2 Confirm that the standardization is correct by analyzing a standard sample. The value should be within the allowable limits of the certified value of the standards. If not, repeat the standardization.

NOTE — The standardization should be repeated if the argon cylinder has been changed, the spectrometer is not in use for very long time or when an unusual drift is noticed when a standard has been sparked for checking.

7 ANALYSIS OF THE SAMPLE

7.1 Place the prepared sample on the spark stand. Spark the sample at two different spots. If the two values differ more than 3 standard deviations established for procedure, then give a third spark. Repolish and repeat the sparking at 2 or 3 spots. Take average of nearly matching values.

7.2 Spark a standard sample of known concentration and make correction in the unknown sample result, if necessary.

NOTE — It is suggested that the standard sample used for verification should have similar matrix and nearly the same nitrogen content 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 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*)'.

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