भारतीय मानक

इस्पात के रासायनिक विश्लेषण की प्रणाली

भाग 19 वाष्प आसवन प्रणाली द्वारा नाइट्रोजन का निर्धारण
(0.002 से 0.50 प्रतिशत नाइट्रोजन के लिए)

(दूसरा पुनरीक्षण)

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF STEELS

PART 19 DETERMINATION OF NITROGEN BY STEAM DISTILLATION METHOD (FOR NITROGEN 0.002 TO 0.50 PERCENT)

(Second Revision)

ICS 77.080.20

BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

July 1998

Price Group 1

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

FOREWORD

This Indian Standard (Part 19) (Second Revision) 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 nitrogen by steam distillation method. The other parts of this series are:

IS 228	Methods for chemical analysis of steels
Part 1 : 1987	Determination of carbon by volumetric method (for carbon 0.05 to 2.50 percent)
Part 2 : 1987	Determination of manganese in plain carbon and low alloy steels by arsenite method
Part 3 : 1987	Determination of phosphorus by alkalimetric method
Part 4 : 1987	Determination of total carbon by gravimetric method (for carbon greater than or equal to 0.1 percent)
Part 5 : 1987	Determination of nickel by dimethyl glyoxime (gravimetric) method (for nickel greater than or equal to 0.1 percent)
Part 6 : 1987	Determination of chromium by persulphate oxidation method (for chromium greater than or equal to 0.1 percent)
Part 7 : 1990	Determination of molybdenum by alpha-benzoinoxime method (for molybdenum greater than or equal to 1 percent) (*third revision*)
Part 8 : 1989	Determination of silicon by the gravimetric method (for silicon 0.05 to 5.00 percent)
Part 9 : 1989	Determination of sulphur in plain carbon steels by evolution method (for sulphur 0.01 to 0.25 percent)
Part 10 : 1989	Determination of molybdenum by thiocyanate (photometric) method in low and high alloy steels (for molybdenum 0.01 to 1.5 percent)
Part 11 : 1990	Determination of silicon by photometric method in carbon steels and low alloy steels (for silicon 0.01 to 0.05 percent)
Part 12 : 1988	Determination of manganese by periodate spectrophotometric method in low and high alloy steels (for manganese 0.01 to 2.0 percent)
Part 13 : 1982	Determination of arsenic
Part 14 : 1988	Determination of carbon by thermal conductivity method (for copper 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)
Part 17 : 1998	Determination of nitrogen by thermal conductivity method (for nitrogen up to .04 percent)
Part 18 : 1998	Determination of oxygen by instrumental method

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be same as that of the specified value in this standard.

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF STEELS

PART 19 DETERMINATION OF NITROGEN BY STEAM DISTILLATION METHOD (FOR NITROGEN 0.002 TO 0.50 PERCENT)

(Second Revision)

1 SCOPE

This standard (Part 19) describes the method for the determination of nitrogen in the range from 0.002 to 0.50 percent in steels.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
266 : 1993	Sulphuric acid (*third revision*)
1070 : 1992	Reagent grade water (*third revision*)

3 SAMPLING

The samples shall be drawn and prepared as described in the relevant Indian Standard.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and reagent grade water (*see* IS 1070) shall be employed for the test.

5 DETERMINATION OF NITROGEN

5.1 Outline of the Method

The sample is washed, thoroughly dried and decomposed with sulphuric acid in the presence of potassium sulphate and a little amount of copper sulphate. It is then steam distilled in the presence of sodium hydroxide and tartaric acid. The nitrogen present is estimated by titrating the distillate with standard sulphuric acid using mixed indicator (bromocresol green and methyl red).

5.2 Apparatus

5.2.1 Decomposition of the sample should be carried out in a 250-ml flask fitted with a ground neck carrying a 45-cm long air condenser.

5.2.2 The steam distillation is carried out in the assembly as shown in Fig. 1.

5.3 Reagents

5.3.1 *Nessler's Reagent*

To 95 ml of potassium iodide solution (25 percent), add with shaking a saturated solution of mercuric chloride until a permanent precipitate is obtained. Make small additions of the potassium iodide solution until the precipitate in almost redissolved, followed by solution of 60 g of potassium hydroxide in 60 ml of water. Add cautiously mercuric chloride solution until a slight permanent yellow precipitate is formed. Dilute to 400 ml and allow to stand overnight before filtering into a stock bottle.

5.3.2 *Ammonia-Free Water*

Add a few pellets of caustic potash and 0.5 g of Devarda's alloy (*see* **5.3.9**) per litre of distilled water, heat on the hot plate for four hours and finally distil

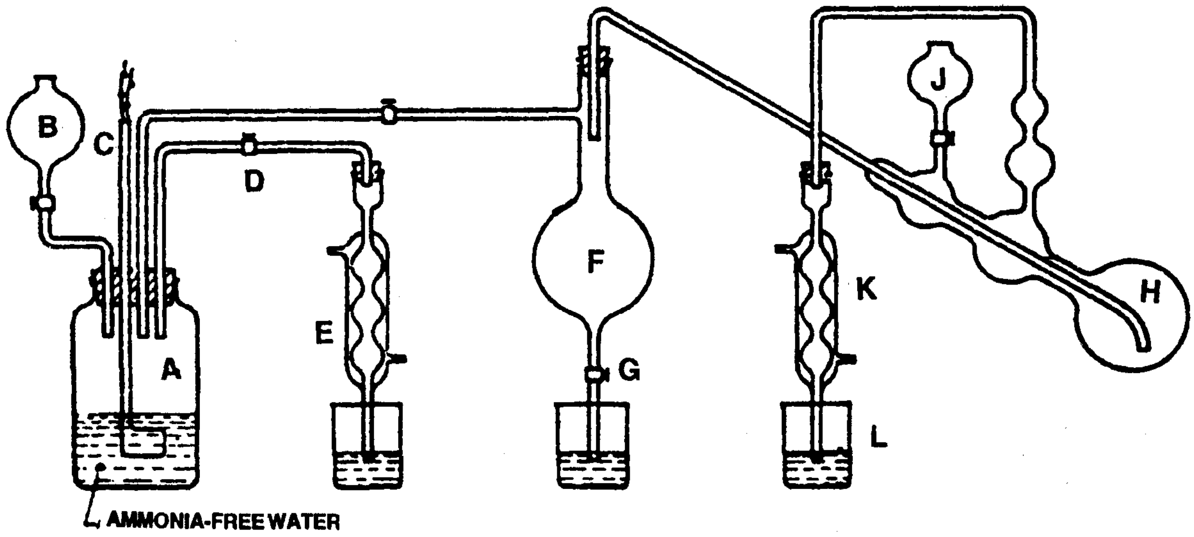

FIG. 1 APPARATUS FOR DETERMINATION OF NITROGEN BY STEAM DISTILLATION

in an all-glass unit rejecting the first 100-ml portion of the distillate. Confirm the absence of ammonia in the distillate by testing a few millilitres with Nessler's reagent before collecting it for use.

5.3.3 *Potassium Sulphate, Crystals*

5.3.4 *Copper Sulphate, Crystals*

5.3.5 *Dilute Sulphuric Acid,* 1:4 (*v/v*).

To 800 ml of ammonia-free water, add cautiously 200 ml of concentrated sulphuric acid free of nitrates.

5.3.6 *Barium Chloride Solution,* 2 percent.

5.3.7 *Sulphuric Acid,* rd=1.84 (conforming to IS 266).

5.3.8 *Mixed Indicator Solution*

Dissolve 0.075 g of bromocresol green and 0.05 g of methyl red in 100 ml of methanol.

5.3.9 *Devarda's Alloy,* 50 Cu, 5 Al, 5 Zn.

5.3.10 *Boric Acid Solution,* 0.1 percent in ammonia-free water.

5.3.11 *Sodium Hydroxide-Tartaric Acid Solution*

To one litre of ammonia-free water, add 500 g of sodium hydroxide and 133 g of tartaric acid. Warm gently to complete solution. Add one gram of Devarda's alloy, boil for 45 minutes, cool and dilute to 1 250 ml with ammonia-free water.

5.3.12 *Standard Sodium Hydroxide Solution,* (0.01 N).

Dissolve 0.5 g of sodium hydroxide pellets in one litre of freshly boiled and cooled distilled water. Dissolve 0.400 0 g of potassium acid phthalate in one litre of freshly boiled and cooled distilled water. Standardize the sodium hydroxide solution against the potassium acid phthalate solution, using phenolphthalein indicator, and render it equivalent to the phthalate solution.

5.3.13 *Standard Sulphuric Acid,* 0.01 N.

Add 3 ml of dilute sulphuric acid (1:9) to 500 ml of water and dilute to one litre. Standardize the dilute sulphuric acid against standard sodium hydroxide solution (0.01 N), using phenolphthalein as indicator. Render it equivalent to the standard alkali solution by dilution with water.

5.3.14 *Standard Sulphuric Acid,* 1 ml =0.000 7 g of nitrogen (0.005 N)

Dilute 50 ml of the standard sulphuric acid (0.01 N) to 100 ml in a volumetric flask.

5.4 Procedure

5.4.1 Wash the sample with ammonia-free water, alcohol and finally with ether. Dry it thoroughly and transfer one to five grams, depending on the nitrogen content of the sample, to a 250-ml round bottom flask. Add five grams of potassium sulphate crystals and one small crystal of copper sulphate and 50 ml of dilute sulphuric acid. Attach an air condenser and digest gently over a broad flame until the decomposition is complete. Evaporate to fumes, cool, dilute with 35 ml of water, warm to redissolve separated salts. If a dark-coloured insoluble residue remains, allow to settle or centrifuge after the addition of five millilitres of barium chloride solution (two percent). Decant off the clear solution into a beaker and treat the residue in the flask with 5 ml of sulphuric acid and 2 g of potassium sulphate. Heat strongly and fume until all the dark-coloured particles are decomposed. Dilute and mix with first portion of the solution. Preserve the solution.

5.4.2 Charge the steam generator *A* with ammonia-free water through the dropping funnel *B*, switch on the immersion heater *C* and allow steam to issue freely from the flask *F* by keeping the stopcock *G* open. Add 20 ml of sodium hydroxide-tartaric acid mixture to the flask *H* through the funnel *J*, close the stopcock *G*, allow steam to bubble through the mixture for 5 minutes. Regulate the flow of steam through the mixture by means of stopcock *D* which provides for the escape of the excess of steam. This can be condensed by the condenser *E* and returned to the steam generator at regular intervals. At the end of the steaming period, switch off the heater and allow the contents of the flask *H* to syphon into the flask *F* and drain out through the tap *G*.

5.4.3 Place 10 ml of boric acid solution in beaker *L* and raise it until the lower end of the condenser *K* dips into the solution. Pour the solution of the sample, reserved in accordance with **5.4.1** into the distillation flask *H* through the funnel *J*. Rinse the beaker with a little ammonia-free water, in the flask *H*, and then add 100 ml of sodium hydroxide-tartaric acid mixture. Stopper the top of the funnel *J*, switch on the heater *C* and close the tap *G*, as soon as the steam starts escaping through it. Adjust a proper flow of cooling water through the condenser *K* and distil as rapidly as possible, collecting 60 to 70 ml of the distillate, indicated by mark on the receiver.

5.4.4 Add three drops of the mixed indicator to the distillate and titrate against standard sulphuric acid (0.005 N). Note the volume of the acid consumed by the solution.

5.4.5 Make a blank determination following the same procedure and using the same amounts of all reagents. The blank should not exceed the equivalent of 0.5 ml of standard sulphuric acid (0.005 N). If higher blanks are recorded, their origin should be traced and eliminated.

5.5 Calculation

$$\text{Nitrogen, percent by mass} = \frac{(V_1 - V_2) \times 0.000\,07}{m} \times 100$$

where

V_1 = volume in ml, of the standard sulphuric acid (0.005 N) consumed by the sample,

V_2 = volume in ml, of the standard sulphuric acid (0.005 N) consumed by the blank run, and

m = mass in g, of the sample taken.

5.6 Reproducibility, ± 0.001 percent.

Bureau of Indian Standards

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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 Handbook' and 'Standards Monthly Additions'.

This Indian Standard has been developed from Doc: No. MTD 2 (4121).

Amendments Issued Since Publication

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