IS 228 (Part 9) : 1989
(Reaffirmed 1994)

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 9 DETERMINATION OF SULPHUR BY EVOLUTION METHOD (FOR SULPHUR 0·01 TO 0·25 PERCENT)

(Third Revision)

भारतीय मानक

इस्पात के रासायनिक विश्लेषण की पद्धतियाँ

भाग 9 निष्कासन पद्धति द्वारा सल्फर ज्ञात करना (सल्फर 0·01 से 0·25 प्रतिशत के लिए)

(तीसरा पुनरीक्षण)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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Price Group 1

FOREWORD

This Indian Standard (Part 9) (Third Revision) was adopted by the Bureau of Indian Standards on 26 December 1989, 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, along with pig iron and cast iron. This standard was again revised to make it comprehensive in respect of steel analysis and to exclude pig iron and cast iron which are being covered in separate standard. 14 parts have already been issued covering only chemical analysis of steels.

This standard IS 228 (Part 9) was published in 1975. In this revision the part has been updated and reproducibility of the method incorporated.

In reporting the result of a test 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*)'.

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 9 DETERMINATION OF SULPHUR BY EVOLUTION METHOD (FOR SULPHUR 0·01 TO 0·25 PERCENT)

(Third Revision)

1 SCOPE

1.1 This standard (Part 9) covers method for determination of sulphur in plain carbon and low alloy steel by evolution method.

NOTE — This method is not suitable for steel samples containing selenium.

2 SAMPLING

2.1 Sample shall be drawn as per relevant Indian Standard.

3 QUALITY OF REAGENTS

3.1 Unless specified otherwise, analytical grade reagents and distilled water shall be employed in the tests.

4 APPARATUS

4.1 Apparatus as shown in Fig. 1 may be used with leak proof joints.

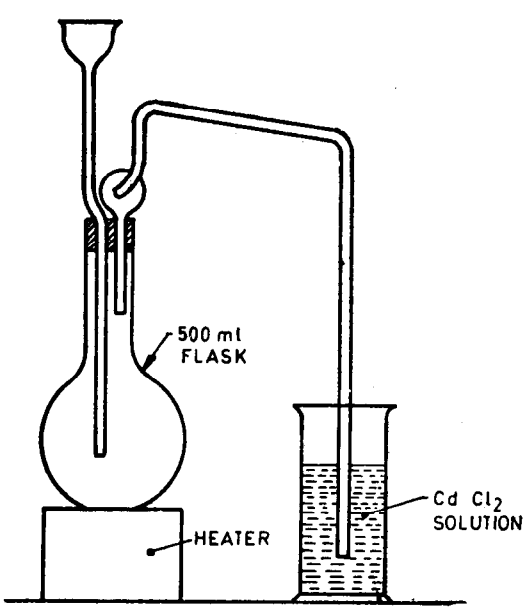

FIG. 1 APPARATUS FOR DETERMINATION OF SULPHUR BY EVOLUTION METHOD

5 DETERMINATION OF SULPHUR BY EVOLUTION METHOD

5.1 Outline of the Method

Sulphur is evolved as hydrogen sulphide and precipitated as cadmium sulphide in ammoniacal cadmium chloride solution. Solution is acidified and excess of potassium iodate-potassium iodide is added. Excesses of liberated iodine is titrated against sodium thiosulphate.

5.2 Reagents

5.2.1 *Dilute Hydrochloric Acid,* 1 : 1 (*v/v*).

5.2.2 *Ammoniacal Cadmium Chloride Solution*

Dissolve 22·8 g of cadmium chloride in 1·0 litres of water and add one litre of ammonia (rd = 0·90).

5.2.3 *Standard Iodate Solution (0·03 N)*

Dissolve 1·07 g of potassium iodate (KIO_3), 10 g of potassium iodide and 2 g of potassium hydroxide in 50 ml water and transfer to one litre volumetric flask and make up.

5.2.4 *Standard Sodium Thiosulphate Solution (0·03 N)*

Mix 8·1 g of sodium thiosulphate ($Na_2S_2O_3 \cdot 5H_2O$) with 0·5 g of sodium carbonate and dissolve in 1 litre of water. Solution is standardized against a primary standard solution of potassium iodate (**5.2.3**).

5.2.5 *Starch Solution*

Dissolve 0·1 g of soluble starch in 100 ml of hot water and boil for 2 to 3 minutes and cool.

5.3 Procedure

5.3.1 Transfer 5·00 g of sample in a 500 ml dry florence flask fitted properly with a rubber cork attached with a thistle funnel and delivery tube bend at right angle. The end of the thistle funnel should nearly touch the bottom of the flask and delivery tube remain dipped in the

measuring cylinder containing 20 ml of ammoniacal cadmium chloride solution and 80 ml of water.

5.3.2 Add dilute hydrochloric acid through thistle funnel in sufficient quantity to cover the sample completely and some quantity left in the thistle funnel also. Heat the flask gently until the sample is completely dissolved. The evolved gas (H_2S) is passed into the ammoniacal cadmium chloride solution, and cadmium sulphide is precipitated. At the completion of the reaction the delivery tube is disconnected.

5.3.3 Transfer the contents of the measuring cylinder to a 250 ml conical flask. Wash the sides of the cylinder with 50 ml of water and add the washings to the flask. Add excess of standard potassium iodate-potassium iodide solution and cool with ice water for 20-30 seconds. Swril the flask and add 20 ml cold water and 50 ml dilute hydrochloric acid. Titrate the excess of liberated iodine with standard sodium thiosulphate solution using starch as indicator till blue colour is discharged. Note the volume of standard sodium thiosulphate solution.

5.3.4 Carry out a reagent blank simultaneously.

5.3.5 *Calculation*

$$\text{Sulphur, percent by mass} = \frac{(A - B) \times C \times 1{\cdot}6}{D}$$

where

A = volume, in ml, of potassium iodate added;

B = volume, in ml, of potassium iodate unused;

C = normality of potassium iodate; and

D = mass, in g, of sample taken.

5.3.6 *Reproducibility*, $\pm 0{\cdot}005$ percent.

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Amendments Issued Since Publication

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 323 01 31, 323 33 75, 323 94 02

Telegrams: Manaksanstha
(Common to all offices)

Regional Offices:		Telephone
Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	323 76 17, 323 38 41
Eastern	: 1/14 C.I.T. Scheme VII M, V.I.P. Road, Maniktola CALCUTTA 700054	337 84 99, 337 85 61 337 86 26, 337 91 20
Northern	: SCO 335-336, Sector 34-A, CHANDIGARH 160022	60 38 43 60 20 25
Southern	: C.I.T. Campus, IV Cross Road, CHENNAI 600113	235 02 16, 235 04 42 235 15 19, 235 23 15
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