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"पुराने को छोड़ नये के तरफ"
Jawaharlal Nehru
"Step Out From the Old to the New"

Indian Standard

METHOD FOR DETERMINATION OF SULPHUR PRESENT IN SPONGE IRON AFTER SEPARATION OF NON-MAGNETIC MATERIALS

ICS 77.080.10
FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Sponge Iron and Smelting Reduction Sectional Committee had been approved by the Metallurgical Engineering Division Council.

Sulphur is an important element which affects the properties of the sponge iron. The test methods specified in this standard will help in evaluation of the performance of sponge iron considered for various application for further processing.

The composition of Committee responsible for the formulation of this standard is given at Annex A.

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 the same as that of the specified value in this standard.
1 SCOPE
This standard prescribes the methods for analyzing sulphur in sponge iron after separation of non-magnetic material.

2 REFERENCES
The following standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<table>
<thead>
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<th>IS No.</th>
<th>Title</th>
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<tr>
<td>10812:1992</td>
<td>Classification of sponge iron/direct reduced iron (DRI) fines/briquettes for steel making (first revision)</td>
</tr>
<tr>
<td>13839:1993</td>
<td>Sponge iron/direct reduced iron (DRI) fines/briquettes for steel making — Specification</td>
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</table>

3 TERMINOLOGY
For the purpose of this standard, sponge iron would mean as defined in IS 10812 and IS 13839.

4 SAMPLE
Sample shall be drawn as specified in IS 10812 for chemical analysis.

5 TEST METHOD
Chemical analysis of sulphur present in sponge iron after separation of non-magnetic material can be determined by the following methods:

a) Evolution and titration method, and
b) Combustion method.

5.1 Evolution Method
When HCl is added to the sponge iron sample, the sulphides of iron are decomposed and an equivalent amount of H₂S gas is evolved. The gas is absorbed in a solution of ammonium cadmium chloride which forms a precipitate of yellow cadmium sulphate. This is treated with an excess solution of iodine of known strength in the presence of HCl. The excess of iodine is treated back with a standard sodium thiosulphate solution. The amount of the iodine solution consumed is a measure of the sulphur in the sample.

5.1.1 Solution Required

5.1.1.1 Stock ammonical cadmium chloride solution
Dissolve 6.25 g of cadmium chloride in 100 ml water and add 500 ml ammonia solution of 0.66 sp gr.

5.1.1.2 Standard sodium thiosulphate solution N/32
Dissolve 7.742 g of sodium thiosulphate in water. Add 2.0 g of sodium bicarbonate and make the solution upto 1 litre.

5.1.1.3 Standard iodine solution N/32
The solution is prepared in such a way that 1 ml of it corresponds to 0.0005 g of sulphur. Dissolve exactly 1.529 g of pure potassium dichromate. Add 15 ml 1:2 H₂SO₄. Cool and add to it a solution containing 15 g of potassium iodide and make upto 1 litre 1 ml of this solution will be equal to 0.01 percent sulphur on a 5 g sample. Dissolve 1.114 g of KIO₃ (potassium iodate) and 10 g of KI (potassium iodide) and 2 g of sodium bicarbonate in 1 litre of distilled water. One ml of this solution will be equal to 0.01 percent sulphur on a 5 g sample.

5.2 Procedure

5.2.1 Weigh 5 g of the crushed sponge iron sample and introduce it in to a 500 ml flat bottom flask fitted with a rubber bung which holds a thistle funnel and a delivery tube. The funnel should almost reach to the bottom of the flask.

Immerse the end of the delivery tube in 200 ml of dilute ammonical cadmium chloride which is made by mixing 40 ml of the stock solution with 160 ml water. Ammonical cadmium chloride solution is used because ammonia neutralizes the HCl, which is produced due to the reaction:

\[
\text{CdCl}_2 + \text{H}_2\text{S} = \text{CdS} + 2 \text{HCl}
\]

This prevents the precipitation of cadmium sulphide in the solution. Now add 100 ml of boiling hot 50 percent HCl solution through the thistle funnel and keep it hot until all vigorous effervescence ceases. Be careful not to boil the solution until the sample is thoroughly dissolved, otherwise ferric chloride will pass into the
cadmium chloride solution and will give a high percentage for sulphur. Also the HCl used should be free from Cl₂ and FeCl₃, which are usually present in it. They oxidize the H₂S formed and lead to low results.

When the sample is completely dissolved, raise the solution to boil to expel the remaining H₂S gas. The evolution of H₂S should be rapid, otherwise a compound of C, H and S may form in the flask. This compound is difficult to volatilize and even if it does, it is not absorbed by the CdCl₂ solution.

After the expulsion of H₂S gas, detach the delivery tube from the flask. Cool the solution in the absorption vessel and add 30 ml of 1.16 sp gr HCl. Run from a burette the standard iodine solution until a faint yellow colour appears, showing the presence of excess iodine. Titrate excess of iodine with the standard sodium thiosulphate solution using 2 ml of freshly prepared starch solution as an indicator. A faint blue colour of the solution marks the end point.

The reactions are:

\[
\begin{align*}
\text{FeS} + 2 \text{HCl} & \rightarrow \text{FeCl}_2 + \text{H}_2\text{S} \\
\text{H}_2\text{S} + \text{CdCl}_2 & \rightarrow \text{CdS} + 2 \text{HCl} \\
\text{CdS} + 2 \text{HCl} + \text{I}_2 & \rightarrow \text{CdCl}_2 + 2 \text{HI} + \text{S}
\end{align*}
\]

5.2.2 Calculation

Sulphur (percent) = \( V \times 0.01 \) (on 5 g sample)

where

\[ V = \text{volume of iodine solution consumed in ml.} \]

5.3 Combustion Method

When a current of pure dried oxygen is passed over the sample heated to a temperature of about 1 200°C, the sulphur is oxidized to SO₂, which is absorbed in silver nitrate solution. Due to this, silver sulphite is formed and an equivalent amount of HNO₃ is liberated. This is titrated against a standard solution of sodium hydroxide. From the amount of NaOH used, the percentage of sulphur in the sample is calculated.

This is a rapid method for determining sulphur in sponge iron. The combustion furnace assembly is very similar to that used for the determination of carbon in steels. It consists of silicon carbide rods as heating elements to attain high temperature in the tube furnace necessary to evolve sulphur as SO₂. Tin granules can be added to the sample to speed up oxidation of the sulphides present.

5.3.1 Solution Required

a) **Methyl red indicator** — One g of methyl red salt in a mixture of 60 ml ethyl alcohol and 40 ml water. Keep for 12 h and filter.

b) **Silver nitrate solution** — Dissolve 4 g of AgNO₃ in 1 litre of water. For absorption purpose, add 10 ml of this solution and 5 drops of methyl red indicator in 100 ml of distilled water.

c) **N/10 sodium hydroxide solution** — Dissolve 1 g of NaOH in 1 000 ml water.

5.4 Procedure

Weigh 1 g sample in a porcelain boat and spread it evenly. Cover the sample with a few tin shots. Place the boat with the sample into the combustion furnace which should be maintained at 1 200°C. Pass dried oxygen at the rate of 1 to 3 l/min for about 3 to 4 min until the combustion is completed. Take the evolved SO₂ into a 250 ml conical flask containing the silver nitrate absorption solution, wait until the absorption solution turns pink. To avoid sucking of the solution, supply a fairly fast stream of oxygen. Seven minutes after the commencement of the experiment, remove the absorption flask and titrate with 0.1 N/40 NaOH until pink colour just disappears.

The value of the standard NaOH solution in terms of sulphur is determined by running a duplicate experiment with 1 g standard sample of known sulphur content. By knowing the value of the NaOH solution per ml, the sulphur in the unknown sample can be calculated.

5.5 Calculation

\[
\text{Sulphur, percent} = \frac{A \times B}{C}
\]

where

\[ A = \text{sulphur burette reading for unknown test sample}, \]
\[ B = \text{percentage of sulphur in known standard sample}, \] and
\[ C = \text{sulphur burette reading for known standard sample}. \]
ANNEX A

(Foreword)

COMMITTEE COMPOSITION

Sponge Iron and Smelting Reduction Sectional Committee, MTD 30

Organization

Tata Iron & Steel Co Ltd, Jamshedpur
Mukand Ltd, Thane
M. N. Dastur & Co (P) Ltd, Kolkata
National Metallurgical Laboratory, Jamshedpur
Kudremukh Iron Ore Co Ltd, Chikmaglur
Sponge Iron India Ltd, Khammam
SAIL, R&D Centre, Ranchi
Central Fuel Research Institute, Dhanbad
MECON (India) Ltd, Ranchi
Orissa Sponge Iron Ltd, Distt Keonjhar
Sunflag India Ltd, Bhandara
Gas Authority of India, New Delhi
Essar Steels, Mumbai
National Mineral Development Corporation Ltd, Hyderabad
Tata Sponge Iron, Distt Keonjhor
Vikram Ispat, Mumbai
Jindal Steel & Power Ltd, Raigarh
Ministry of Steel, New Delhi
Steel Furnace Association of India, New Delhi
GSDAI Ltd, Hyderabad
HEG Ltd, Durg
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Prakash Industries, New Delhi
Raipur Alloys, Raipur
Ispat Industries Ltd (Nippo Denro), Raigad
Monnet Ispat Ltd, Raipur
Usha (India) Ltd, New Delhi
Sponge Iron Manufacturers’ Association, New Delhi
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Amendments Issued Since Publication

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