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“जानने का अधिकार, जीने का अधिकार”
Mazdoor Kisan Shakti Sangathan
“The Right to Information, The Right to Live”

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


“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”
Bhartrhari—Nitisatakam
“Knowledge is such a treasure which cannot be stolen”
Indian Standard

FEEDSTOCK (IRON OXIDES AND NATURAL GAS) FOR GAS-BASED DIRECT REDUCTION PROCESSES — GUIDELINES

( First Revision )

ICS 73.060:77.020

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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FOREWORD

This Indian Standard (First Revision) 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.

In recent years, sponge iron has gained prominence as a feedstock, for steel making. Quality of raw materials for gas-based direct reduction processes plays a significant role for the production or sponge iron. To ensure the supply of suitable quality raw materials, it has been considered desirable to formulate this standard. In this revision, the method of determination of sticking of iron ores (lumps and pellets) used in direct reduction by gaseous reducing agents and determination of disintegration of DR Feedstock (Iron ore and pellets) using gaseous reductants in a shaft covered in IS 14373 : 1996 ‘Determination of sticking of iron ores used in direct reduction by gaseous reducing agent — Specification’ and IS 14405 : 1996 ‘Determination of disintegration of DR feedstock (iron ore, pellets) using gaseous reductants in a shaft reactor — Specification’ have been incorporated. Subsequent to the publication of this revision, IS 14373 and IS 14405 shall be withdrawn.

In the formulation of this standard, assistance have been derived from the ISO document in order to keep harmony with the test method proposed by ISO/11257 : 1998 ‘Iron ores — Determination of disintegration and metallization of feed stock for direct reduction by gas reforming process’ and ISO/TC 102/SC 5N89 ‘Method for determination of sticking of iron ores used in direct reduction done by gaseous reducing agents’.

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 founded 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.
Indian Standard

FEEDSTOCK (IRON OXIDES AND NATURAL GAS) FOR GAS-BASED DIRECT REDUCTION PROCESSES — GUIDELINES

(First Revision)

1 SCOPE
1.1 This standard covers the requirements of iron oxides feedstock (both lump ore and pellets) and natural gas for gas-based direct reduction, as desired by the established processes in commercial operation, for the production of steel making grade direct reduced iron (DRI).

1.2 Requirements covered in this standard shall be met at the point of receipt, unless it is stated otherwise.

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>
<tr>
<th>IS No.</th>
<th>Title</th>
</tr>
</thead>
<tbody>
<tr>
<td>460 (Part 2) : 1985</td>
<td>Specification for test sieves: Part 2 Perforated plate test sieves (third revision)</td>
</tr>
<tr>
<td>1405 : 1982</td>
<td>Method of sampling iron ores (second revision)</td>
</tr>
<tr>
<td>11092 : 2001</td>
<td>Iron ore pellets for direct reduction processes — Specification (second revision)</td>
</tr>
<tr>
<td>11093 : 2001</td>
<td>Iron ore lumps for direct reduction processes — Specification (second revision)</td>
</tr>
</tbody>
</table>

3 TERMINOLOGY
For the purpose of this standard, the following definitions shall apply.

3.1 Load — It is a force applied to the surface of the test portion in order to simulate that of furnace burden; it is expressed as a 'mechanical pressure', that is the force per unit surface.

3.2 Sticking or Clustering Behaviour — It is a tendency of reduced iron ores to agglomerate when exposed to the temperature, the gas and the load existing in a reducing furnace.

3.3 Cluster — It is two or more particles of reduced iron ore sticking together.

3.4 Sticking Temperature — It is the temperature of the direct reduction process at which the reduced iron sticks together and the clusters so formed do not disintegrate due to their movement in the furnace.

3.5 Test Sticking Temperature — It is a temperature at which the procedure described in this standard gives the sticking index equal to a certain value.

3.6 Sticking Index — It is the relative measure expressing the results of the sticking test.

3.7 Metallization — It is a measure of the conversion of iron oxides into metallic iron (either free or in combination with carbon as a cementite) by removal of oxygen due to the action of the reductant used.

3.8 Degree of Metallization — It is used to describe the efficiency of removal of oxygen from iron oxide during reduction. It is defined as follows:

\[
\text{Degree of metallization, } = \frac{\text{Mass of metallic iron}}{\text{Mass of total iron}} \times 100 \%
\]

3.9 Metallic Iron — It is the iron chemically uncombined and combined as cementite (Fe₃C).

3.10 Disintegration — The size degradation that occurs during the reduction of iron ore and pellets in a direct reduction shaft reactor.

3.11 Disintegration Index — It is relative measure of the susceptibility to the disintegration of iron bearing materials expressed by the mass percentage less than 3.15 mm of the reduced sample.

4 REQUIREMENTS FOR IRON OXIDE FEEDSTOCK
4.1 The physical and chemical requirements for iron ore lump and pellets for direct reduction process shall be as given in IS 11092 and IS 11093.
4.1.1 Disintegration of DR Feedstock Using Gaseous Reductant

The method for evaluating disintegration of iron bearing materials (ore and pellets) under conditions similar to those prevailing in direct reduction process carried out in a shaft furnace using gaseous reducing agents shall be as given in Annex A.

4.1.2 Sticking or Clustering of Iron Oxide Feedstock

The method for determination of clustering characteristics of iron ore lumps and pellets during reduction under condition similar to those prevailing in direct reduction process carried out in a shaft furnace using gaseous reducing agents shall as given in Annex B.

5 NATURAL GAS

5.1 Quality Requirements

5.1.1 Natural gas is used for the generation of the reducing gas mixture of carbon monoxide and hydrogen, by adopting a suitable natural gas reforming process.

5.1.2 The major quality parameters of natural gas are the methane CH₄, sulphur contents and some portion of hydrocarbon heavier than methane. A high methane content is desirable in the natural gas for efficient conversion to the reformed gas. Unsaturated hydrocarbons may cause coking (carbon deposition on the catalyst) which decreases the catalyst activity and, therefore, their levels in the natural gas should be low. The sulphur content should be as low as possible in order to minimize poisoning of the reformer.

5.1.3 The typical analysis of the off-shore natural gas for gas-based direct reduction processes is given below for guidance.

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Mol. Percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitrogen (N₂)</td>
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</tr>
<tr>
<td>Carbon dioxide (CO₂)</td>
<td>4.98</td>
</tr>
<tr>
<td>Methane (CH₄)</td>
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</tr>
<tr>
<td>Ethane (C₂H₆)</td>
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<tr>
<td>Propane (C₃H₈)</td>
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</tr>
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<td>Butane (C₄H₁₀)</td>
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<td>Pentane (C₅H₁₂)</td>
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</tr>
<tr>
<td>H₂O</td>
<td>0.01</td>
</tr>
<tr>
<td>H₂S</td>
<td>&lt;25 ppm</td>
</tr>
</tbody>
</table>

ANNEX A

(Clause 4.1.1)

DETERMINATION OF DISINTEGRATION OF DR FEEDSTOCK (IRON ORE LUMPS AND PELLETS) USING GASEOUS REDUCTANTS IN A SHAFT REACTOR

A-1 This Annex prescribes a method for evaluating the disintegration of iron bearing materials (ore and pellets) under conditions similar to those prevailing in direct reduction process carried out in a shaft furnace using gaseous reducing agent.

A-2 PRINCIPLE

The test portion is subjected to isothermal reduction in a rotating tube at 760°C or at 850°C for 4 h by reducing gas consisting of H₂, CO, CO₂ and CH₄. Heating and cooling of the sample is done in an inert nitrogen atmosphere followed by sieving to calculate the disintegration index. After testing, the sample is also chemically/instrumentally (utilizing magnetizing principle) analyzed to determine the metallization.

A-3 TEST SAMPLE

For 500 g test portions in the size range 10.0 to 12.5 mm for pellets and 10 to 20 mm (being 50 percent between 10 to 16 mm and 50 percent between 16 to 20 mm) for iron ore lumps.

A-4 APPARATUS

A-4.1 The apparatus shall consists of the following (see Fig.1).

A-4.1.1 Gas Supply System — It shall be capable of supplying the gases at the specified gas flow rates.

A-4.1.2 Reduction Tube — It shall be made of non-scaling, heat resisting metal to withstand a temperature of 760°C. The inside diameter of the reduction tube shall be 130 mm and the inside length 200 mm. Four
equally spaced lifters 200 mm long, 20 mm wide and 2 mm thick shall be mounted longitudinally inside the reduction tube.

A-4.1.3 Furnace — A suitable furnace, having a heating capacity sufficient to arrive at the test temperature within 60 min and to maintain the entire test portion at 760±5°C.

A-4.1.4 Weighing Balance — A suitable balance capable of weighing with a precision of ±0.5 g.

A-4.1.5 Test Sieves — These should conform to IS 460 (Part 2) and have square apertures of nominal sizes 16.0 mm, 12.5 mm, 10.0 mm, 6.3 mm, 3.15 mm and 500 μm.

A-4.1.6 Dust Collection Unit — This is to trap any solid particles carried in the gas stream out of the tube during the test.

A-5 TEST CONDITIONS

Volumes and flow rate of gases in this standard are measured at NTP.

A-5.1 Composition of Reducing Gas

The required gases shall consist of:

- \( \text{H}_2 \) : 55 ± 0.5 percent (v/v)
- \( \text{CO} \) : 35 ± 0.5 percent (v/v)
- \( \text{CO}_2 \) : 5 ± 0.5 percent (v/v)
- \( \text{CH}_4 \) : 5 ± 0.5 percent (v/v)

A-5.2 Purity of Reducing Gases

Impurities in the reducing gas shall not exceed:

- \( \text{O}_2 \) : 0.1 percent (v/v)
- \( \text{H}_2\text{O} \) : 0.2 percent (v/v)

A-5.3 Flow Rate of Reducing Gas

The reducing gas flow rate shall, during the test period, be maintained at 15 nl/min ± 0.5 nl/min.

A-5.4 Reducing Temperature

The reducing gas shall be preheated while entering the reduction tube to maintain the temperature of the reduction tube and hence the test portion at 760±5°C or 850°C during the entire period of testing.

A-6 TEST PROCEDURE

The test portion of 500 g, in the size range of 10.0 to 12.5 mm for pellets and 10 mm to 20 mm (being 50 percent between 10 to 16 mm and 50 percent between 16 to 20 mm) for iron ore lumps is weighed with a precision of ±1 g and is then placed in the reduction tube. The reduction tube is placed in the furnace and the gas flow system connected (see Fig. 1) shows an example of the arrangements of the reduction test unit.

Flow of nitrogen gas through the reduction tube is started at the rate of 10 l/min. Reduction tube is rotated at 10 ± 0.2 rpm and then the furnace is switched on. The heating rate is regulated to achieve a temperature of 760°C in 1 h. When the temperature approaches
760°C, the flow of nitrogen is increased to 15 l/min. These conditions are maintained for 15 min. Then the inert gas is replaced by the reducing gas, at a flow rate of 15 l/min, and reduction is allowed to take place for 4 h while maintaining the temperature at 760°C or 850°C. At the end of this period, the rotation and heating is stopped and the sample is cooled to room temperature in a flow of inert (nitrogen) gas. The sample is then removed from the reduction tube by tilting the reactor and hand sieved for 2 min through 6.3 mm, 3.15 mm, and 500 μm sieves. In many cases, some sticking of material inside the tube will take place and some scrapping will be necessary to remove all the material from the reduction tube.

The dry weight of dust trapped in a dust collector must be added to the weight of the 0.5 mm fraction. The reduced sample is pulverized and subjected to chemical/instrumental analysis for determination of total iron, ferrous iron and metallic iron according to relevant Indian Standards.

### A-7 EXPRESSION OF RESULTS

The DR disintegration index (DR – RDI), as a percentage by mass, is calculated as follows:

\[
(DR - \text{Strength}) + 6.3 = \frac{m_1}{m_0} \times 100
\]

\[
(DR + \text{RDI}) - 6.3 = \frac{m_0 - m_1}{m_0} \times 100
\]

\[
(DR - \text{RDI}) - 3.15 = \frac{m_0 - (m_1 + m_2)}{m_0} \times 100
\]

where

- \(m_0\) = mass of test portion after reduction, in g;
- \(m_1\) = mass of oversize fraction retained on the 6.30 mm sieving, in g;
- \(m_2\) = mass of oversize fraction retained on 3.15 mm sieving, in g; and
- \(m_3\) = mass of oversize fraction retained on the 500 μm sieving, in g.

### A-8 NUMBER OF TESTS AND PERMISSIBLE TOLERANCES

The test shall generally be carried out in duplicate and individual as well as average values reported. The difference between the paired results should remain within the tolerance of ± 2 percent for disintegration test and ± 0.5 percent for parameters evaluated in chemical analysis.

### A-9 TEST REPORT

The test report shall include the following information:

a) Name and address of the testing laboratory,

b) Date of issue of the test results,

c) Reference to this Indian Standard,

d) Identification of the sample and its chemical analysis,

e) Results of the test rounded up to one decimal place, and

f) Test conditions other than the standard specified shall be indicated.
ANNEX B
(Clauses 4.1.2)

DETERMINATION OF STICKING OR CLUSTERING OF IRON ORES (LUMPS AND PILLETS)
USED IN DIRECT REDUCTION BY GASEOUS REDUCING AGENTS

B-1 This Annex specifies two methods, namely Method A and Method B for determining the sticking index of iron ores. These methods prescribe determination of sticking characteristics of iron ore lumps and pellets during reduction under conditions similar to those prevailing in gas based direct reduction process carried out by shaft furnace using gaseous reducing agent. The result of this test are expressed in relative terms.

B-2 TEST SAMPLE

B-2.1 The test sample shall be taken in accordance with the procedure specified in IS 1405. A quantity of sample sufficient to provide at least seven test portions shall be prepared. The test portions shall be oven dried at 105 ± 5°C and cooled to room temperature before the preparation of proper test portion.

B-2.2 The sample weight shall be 2 000 g and 500 g for Method A and Method B respectively.

NOTE — If the method of expressing the results, described in B-7.1 (b) is used, the number of individual particles in the test portion should be counted.

B-2.3 Lump ore size shall be + 6 mm –15 mm and pellet size shall be 10 to 12.5 mm for the purpose of test sample.

B-3 REDUCING GAS

Reducing gas composition shall be as follows:

H₂ : 55 ± 0.5 percent (v/v)
CO : 35 ± 0.5 percent (v/v)
CO₂ : 5 ± 0.5 percent (v/v)
H₂O : 5 ± 0.5 percent (v/v)
N₂ : 0.1 ± 0.05 percent (v/v)

B-4 TEST CONDITIONS

B-4.1 Gas Flow-Rate

a) Method A — 40 ± 2 lpm at STP
b) Method B — 30 ± 1 lpm at STP

B-4.2 Reduction Temperature

Each sample shall be reduced at two of these temperatures 750°C, 800°C and 850°C. The first test portions should be measured at 800°C. If the sticking index is higher than 10 percent, the second test should be done at 750°C. If the sticking index measured at 800°C is less than 10 percent the second test should be done at 850°C.

B-4.3 Time of Reduction

180 min.

B-4.4 Load

a) Method A — The load of 1 800 N is applied to the surface of the test portion of 122.7 cm² (125 mm inside diameter reduction tube).

b) Method B — The load of 650 N (66 kg-force) is applied to the surface of the test portion of 44.2 cm² (75 mm inside diameter reduction tube).

The mechanical pressure applied to the surface of the portion is 14.7 N/cm².

B-5 APPARATUS

B-5.1 Reduction Tube — A tube assembly made of heat-resistant steel capable of withstanding the test conditions without any significant permanent deformation. The reduction tube should have the internal diameter of 125 mm or 75 mm and it should be long enough to assure preheating the gas flow to a test temperature.

B-5.2 Furnace — A suitable furnace capable of keeping a test temperature constant over the entire height of the test portion with an accuracy of ± 5°C.

B-5.3 Gas Supply System — It shall be capable of supplying the gases at specified gas flow rates and connected to the tube in such a manner so that weighing is not affected.

B-5.4 Loading System — A pneumatically operated pressure ram capable of applying load to the surface of the test portion. The accuracy of the pressure indication should be ± 0.2 N/cm².

B-5.5 Test Sieves — Test sieves shall have square mesh opening of 10, 12, 16 and 25 mm nominal size and conform to IS 460 (Part 2).

B-5.6 Weighing System — Scales shall be capable of weighing the sample portion of 2 000 g with an accuracy of ± 1 g.

B-5.7 Disintegration System — A device for the disintegration of the agglomerated material by dropping. The clusters shall be dropped on the clean steel plate of thickness of 25 mm and a weight of 15 kg.

B-6 TEST PROCEDURE

B-6.1 The predried sample portion along with two layers
of ceramic pellets are carefully introduced to the reduction tube assembly which consists of a steel sleeve, a ceramic tube sleeve and the main reduction tube. While introducing the test sample surfaces of bottom layer of ceramic pellets, test samples and the upper layer of ceramic pellets are leveled carefully. Gas supply, thermocouple and loading device, etc., are connected to the system. The reduction tube assembly is inserted into the furnace and then preheated to the test temperature.

B-6.2 Nitrogen circulation is started (20 lpm or 15 lpm) and increased when approaching test temperature to 40 lpm or 30 lpm. After temperature stabilization the nitrogen gas is replaced with reducing gas keeping the gas flow rate the same. After 1 h load of 1 800 N or 650 N is applied respectively.

B-6.3 After 3 h of reduction, reducing gas is replaced with nitrogen, load is withdrawn and sample is cooled to below 50°C. After cooling the test sample is carefully taken out. The non-adhered particles are separated from the agglomerated material. The agglomerated material is subject to drop test from a height of 40 cm or 80 cm on steel plate. The dropped material is removed from the steel plate after each drop in order to prepare a clean surface for the next drop. Calculate the sticking index either as per (a) or (b) given below (see Tables 1 and 2 respectively):

a) Separate and weigh the clusters remaining after each drop procedure ($m_i$),

b) Separate and count the individual particles ($N_i$) liberated by each drop and clusters remained after each drop, and

c) Repeat the dropping procedures 20 times ($i = 1$ to 20).

B-7 EXPRESSION OF RESULTS

B-7.1 Calculation of the Sticking Index

a) *Method A* — Sticking index based on the mass of the agglomerated material.

$$S.I.(i) = \frac{100}{\sum_{i=1}^{n} m_i/m_r}$$

where

$m_i = $ mass of agglomerated material (one or more clusters) after each drop, and

$m_r = $ mass of the reduced test portion.

An example of the calculation of the sticking index after 1 to 20 drop procedures is given in Table 1.

b) *Method B* — Sticking index based on the number of individual pieces and clusters.

The ratio of sticking, ($T_i$) is calculated as follows:

$$T_i = \frac{i}{N_o} \left[ N_o - \left( N_i + \sum_{k=1}^{i} N_k \right) \right]$$

where

$N_o = $ number of individual particulars in the test portion,

<table>
<thead>
<tr>
<th>No. Drop Procedure</th>
<th>$m_i$</th>
<th>$m_i/m_r$</th>
<th>$\sum_{i=1}^{n} m_i/m_r$</th>
<th>S.I.(i) Percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>706</td>
<td>0.490</td>
<td>0.490</td>
<td>49</td>
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<td>0.000</td>
<td>1.362</td>
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</tbody>
</table>
\( N_c = \) number of clusters in the agglomerated material after the dropping procedure,
\( N_k = \) number of individual particulars liberated from the agglomerated material by one dropping procedure, and
\( i = \) number of dropping procedures.

Sticking index S.I.(i) for a dropping procedure is calculated as follows:

\[
\text{S.I.}(i) = \frac{100}{i} \sum_{k=1}^{i} \frac{N_c}{N_k}
\]

An example of the calculation is given in Table 2.

**B-7.2 Metallization**

\[
\text{Fe (met)} = \left( \frac{\text{Percent Fe (total)}}{100} \right) \times 100
\]

The disintegrated portion is analyzed for the metallization and carbon content.

Table 2 Example of the Calculation of the Sticking Index Based on the Number of Individual Particulars and Clusters (\( N_c = 600 \) Pellets)

<table>
<thead>
<tr>
<th>( I )</th>
<th>( N_k )</th>
<th>( \sum_{k=i}^{i} N_k )</th>
<th>( N_c )</th>
<th>( \sum_{k=i}^{i} N_k )</th>
<th>( T )</th>
<th>S.I.(i) Percent</th>
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