IS 15774 (2007): Sponge Iron/Directed Reduced Iron (DRI) Hot Briquetted Iron (HBI) and Cold Briquetted Iron (CBI) for Steel Making [MTD 30: Sponge Iron and Smelting Reduction]

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IS 15774 : 2007
(Amalgamating IS 10812 : 1992,
IS 13839 : 1993 and IS 13905 : 1993)

भारतीय मानक

इस्पात बनाने के लिए स्पंज लौहा/प्रत्यक्ष अपचयित लौहा (डीआरआई), तप्त और अतप्त इष्टिकाकृत लौहा — विशिष्टि

Indian Standard

SPONGE IRON/DIRECT REDUCED IRON (DRI) HOT BRIQUETTED IRON (HBI) AND COLD BRIQUETTED IRON (CBI) FOR STEEL MAKING — SPECIFICATION

ICS 73.060.10

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

November 2007

Price Group 6

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.

This standard has been formulated by amalgamating IS 10812 : 1992 'Classification of sponge iron/direct reduced iron (DRI) fines/briquettes for steel making', IS 13839 : 1993 'Specification for sponge iron/direct reduced iron (DRI) fines/briquettes for steel making' and IS13905 : 1993 'Hot briquetted sponge iron (HBI) for steel making'. With the formulation of this standard IS 10812 : 1992, IS 13839 : 1993 and IS 13905 : 1993 stand withdrawn.

In this revision, the carbon content for rotary kiln DRI/sponge iron has been modified to around 0.1 percent apart from the size range of sponge iron.

Direct reduction (DR) is a metallurgical process of producing iron from iron oxides (mainly in the form of lump ore or pellets) directly in the solid phase, that is without going through the molten stage as occurs in blast furnace or other melting processes. The product of this solid state route has a honeycomb structure with minute pores, hence it is most commonly known as 'sponge iron' or 'direct reduced iron (DRI)' or 'metallized iron'. However, for this standard the term sponge iron will only be used. In addition to the metallic iron content, sponge iron contains some unreduced iron oxides and gangue. Since there is no separation of impurities in DR process, all the gangue present in the original oxide feed goes into the product, in fact, concentration of the same increases because of removal of oxygen.

Reduction products with a metallization degree of less than 82 percent are known as 'pre-reduced iron'; and are primarily used in the smelting furnaces for the production of liquid iron.

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 value (*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.

AMENDMENT NO. 1 JUNE 2010
TO
IS 15774 : 2007 SPONGE IRON/DIRECT REDUCED IRON (DRI) HOT BRIQUETTED IRON (HBI) AND COLD BRIQUETTED IRON (CBI) FOR STEEL MAKING — SPECIFICATION

(*Page* 1, *clause* **1**, *line* 3) — Delete ‘and induction furnaces’.

(MTD 30)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 2 NOVEMBER 2012
TO
IS 15774 : 2007 SPONGE IRON/DIRECT REDUCED IRON (DRI) HOT BRIQUETTED IRON (HBI) AND COLD BRIQUETTED IRON (CBI) FOR STEEL MAKING — SPECIFICATION

(*Page* 4, *clause* **9**) — Substitute the following for the existing clause:

'**9 MARKING**

9.1 A metallic or cardboard label of appropriate size bearing the following information with suitable paint or ink shall be conspicuously displayed on the carrier or also placed inside:

a) Indication of source of manufacture;
b) Name and grade of the materials; and
c) Size.

9.2 The material shall also accompany a test certificate giving the following:

a) Indication of source of manufacture;
b) Name and grade of the materials;
c) Chemical composition; and
d) Size.'

(MTD 30)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPONGE IRON/DIRECT REDUCED IRON (DRI) HOT BRIQUETTED IRON (HBI) AND COLD BRIQUETTED IRON (CBI) FOR STEEL MAKING — SPECIFICATION

1 SCOPE

This standard covers sponge iron DRI, HBI and cold briquettes for use in steel making in electric arc furnace (EAF) and induction furnaces.

It is also used in blast furnaces and LD converter during steel making by large steel plants.

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provision 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:

IS No.	*Title*
228 (Part 1) : 1987	Method of chemical analysis of steels: Part 1 Determination of carbon by volumetric method (for carbon 0.05 to 2.50 percent) (*third revision*)
265 : 1993	Hydrochloric acid (*fourth revision*)
1387 : 1993	General requirements for the supply of metallurgical materials (*second revision*)
1405 : 1982	Methods of sampling of iron ores (*second revision*)
1493 : 1959	Methods of chemical analysis of iron ores
1493 (Part 1) : 1981	Methods of chemical analysis of iron ores: Part 1 Determination of common constituent (*first revision*)
1607 : 1977	Method for test sieving (*first revision*)
10852 : 1984	Guidelines for storage and transportation of sponge iron/direct reduced iron (DRI)
14719 : 1999	Density of hot briquetted iron (HBI) — Method for determination

3 TERMINOLOGY

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

3.1 Sponge Iron/Direct Reduced Iron — It is the resulting product (with a metallization degree greater than 82 percent) of solid state reduction of iron ores or agglomerates (generally of high grade), the principal constituents of which are metallic iron, residual iron oxides carbon and impurities such as phosphorus, sulphur and gangue (principally silica and alumina).

3.2 Hot Briquetted Iron (HBI)— HBI means briquettes made out of sponge iron/DRI produced in gas based direct reduction processes at elevated temperature by the application of external pressure in a more or less close mould.

3.3 Cold Briquettes — Cold briquettes mean cold bonded sponge iron/DRI briquettes, made from sponge iron/DRI fines only.

3.4 Total Iron — It is defined as:

$$Fe(T) = Fe(M) + Fe(O)$$

where

Fe(T) = total iron,

Fe(M) = metallic iron, and

Fe(O) = iron from residual iron oxides present in sponge iron/DRI, HBI and cold briquettes.

3.5 Metallic Iron — It is the aggregate quantity of iron, either free or combined with carbon (as cementite) present in sponge iron/DRI, HBI and cold briquettes.

3.6 Residual Iron Oxides — These are remaining oxides of iron present in sponge iron either in the form of FeO, Fe_3O_4 or Fe_2O_3, though normally FeO would be the only residual oxides present.

3.7 Total Carbon — It is the total carbon present in sponge iron, and is equal to the sum of free and combined carbon (as cementite).

3.8 Impurities — These are undesirable elements/ compounds in sponge iron, sulphur and phosphorus being the most common among them.

3.8.1 *Gangue* — It is the amount of other impurities present in sponge iron (resulting from the raw materials), in the form of oxides such as Al_2O_3, SiO_2, CaO, MgO, etc.

3.8.2 *Quarternary Basicity* — It is the ratio of CaO,

MgO, and Al_2O_3, SiO_2, and is expressed as:

$$B = \frac{\text{Percent CaO + Percent MgO}}{\text{Percent } Al_2O_3 + \text{ Percent } SiO_2}$$

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

3.10 Degree of Metallization — It is used to describe the extent of conversion of iron oxide into metallic iron during reduction. It is defined as follows:

$$\text{Degree of metallization, percent} = \frac{\text{Mass of metallic iron}}{\text{Mass of total iron}} \times 100$$

3.10.1 *Equivalent Metallization* — Carbon content in sponge iron at a given degree of metallization is important. It has the capacity to remove oxygen from the remaining iron oxides during steel making. This leads to the concept of equivalent metallization, which may be defined as:

Equivalent metallization, percent = Degree of metallization (percent) + 5 times the percent of total carbon in sponge iron

3.11 Fines — It refers to cold sponge iron fines with size 0-3 mm obtained during production of sponge iron through solid/gaseous reduction route.

3.12 Binders — They refer to additives used in the briquetting process for increasing the strength of the briquette and as lubricants to decrease the roll wear.

3.12.1 *Binder Specification* — It is desirable to use a combination of solid and liquid binders for getting desired strength of cold briquettes.

3.12.1.1 *Solid binder* — Hydrated lime, $Ca(OH)_2$ should be minimum 80 percent, shall have grain size −100 mesh or 0.15 mm.

The typical composition shall be as follows:

CaO, percent	:	65-70
SiO_2, percent	:	2.5-3
MgO, percent	:	0.5-1
Al_2O_3, percent	:	0.4-0.5
LOI, percent	:	24-26

3.12.1.2 *Liquid binder*

a) *Sodium silicate* — Sodium silicate (liquid) shall have an apparent density of 1.45 t/m^3 and shall have a specific gravity of 1.40.

Typical composition of sodium silicate:

Total soluble silica	:	26 to 30 percent
Na_2O	:	8.8 to 9.4 percent
Mass ratio of total soluble silica as (SiO_2) to total Na_2O	:	– 3 to 3.4

b) *Molasses* — Specific gravity should be minimum 1.3. Sulphur and phosphorus content should be checked to ensure acceptable limits of the same in the final briquettes.

The liquid binder and solid binder shall constitute about 3-4 percent and 2-3 percent of DRI fines mix respectively.

Powdered pitch can be added to get desired strength of briquettes, if the same is not achieved by the above means. The typical specification shall be as follows:

S, percent	:	< 3
H_2O	:	O
Grain Size	:	– 0.15 mm
Softening temperature, °C	:	120 ± 25

3.12.2 *Mixing* — Mixing should take place in a suitable mixer with mixing time ranging between 2.5 and 3 min.

3.12.3 *Cold Briquetting* — Metallized fines having apparent density in the range of 1.6-1.9 t/m^3 mixed with suitable proportion of binders are to be fed into a roll type briquetting machine of suitable capacity. The briquetting force shall range from 1 200-1 500 kN at a hydraulic pressure of 180-200 bar. The briquettes obtained shall have size ranging as mentioned in **6.3**.

3.12.4 *Curing* — Natural curing of briquettes shall be done for 24 h for gaining desired strength.

4 SUPPLY OF MATERIAL

General requirements relating to supply of sponge iron/ DRI, HBI and cold briquettes shall be as laid down in IS 1387.

5 CHEMICAL COMPOSITION

5.1 Sponge Iron/DRI (Lumps) and HBI

The chemical composition of sponge iron/DRI and HBI shall conform to the requirements given in Table 1.

5.2 Sponge Iron/DRI Fines

The chemical composition of sponge iron/DRI fines is given in Table 2.

5.3 Cold Briquettes

The chemical composition of cold briquettes shall conform to the equipment given in Table 3.

Table 1 Chemical Composition of Sponge Iron/DRI (Lumps) and HBI Constituents, Percent

(*Clause* 5.1)

Sl No.	Constituent	Grade		
		1	2	3
(1)	(2)	(3)	(4)	(5)
i)	Total iron, percent	89-93	88, *Min*	85 to <88
ii)	Metallic iron, percent	80, *Min*	76, *Min*	70, *Min*
iii)	Metallization, percent	88, *Min*	86, *Min*	82, *Min*
iv)	Carbon content:			
	1) Gas based sponge iron/DRI	≤1.5	≤1.5	≤1.5
	2) HBI	1.1 and above	0.97 - <1.1	0.97 - <1.1
	3) Coal based sponge iron/DRI	0.08-0.12	0.08 - 0.12	0.06 - 0.12
v)	Sulphur , *Max*:			
	1) Gas based sponge iron/DRI	0.005	0.005	0.005
	2) HBI	0.005	0.005	0.005
	3) Coal based sponge iron/DRI	0.030	0.030	0.030
vi)	Phosphorus, *Max*	0.060	0.070	0.100
vii)	$SiO_2 + Al_2O_3$, *Max*	6	7	8
viii)	Combined total of Pb, Zn, Cu, Sn, Cr, Ni, and As, *Max*	0.010	0.015	0.015

NOTES

1 The above chemical composition does not include Briquetted HBI fines.

2 Higher carbon content may also be supplied.

3 Metallic iron = $\frac{\text{Total iron} \times \text{Degree of metallization}}{100}$

4 Although FeO content of sponge iron/DRI is an important constituent, it is generally not specified because its content will depend on the original Fe content of the ore. Higher the Fe content of the ore used for direct reduction, higher will be FeO in the sponge iron/ DRI and HBI for the same degree of metallization.

5 For any given degree of metallization, the equivalent metallization would vary depending upon the total carbon content of the sponge iron/DRI and HBI, which has to be agreed mutually between the supplier and the purchaser. Therefore, the equivalent degree of metallization has not been indicated in this table.

Table 2 Chemical Composition of Sponge Iron/DRI Fines

(*Clause* 5.2)

Size	Total Iron	FeM	C	S	P
mm	Percent *Min*	Percent *Min*	Percent	Percent *Max*	Percent *Max*
0 to 3	89	80	0.10-0.15	0.03	0.06

NOTES

1 In case the fines are supplied as sponge iron fines, the composition of the fines shall be as given above.

2 +3 mm fraction shall not exceed 5 percent and –1 mm fraction should not exceed 30 percent.

5.4 Except for metallic iron and carbon the chemical analysis of sponge iron including total iron shall be determined either by the method specified in IS 1493 and IS 1493 (Part 1) or any other established instrumental/chemical method. In case of dispute the procedure in the latest edition of IS 1493 for chemical analysis shall be the 'Referee Method'.

5.5 Chemical analysis of metallic iron shall be determined by the ferric chloride method prescribed in Annex A and a correction factor of +0.8 percent metallization may be added by the manufacturer in arriving at the actual degree of metallization certified. However, in case of any dispute the purchaser shall only accept the result obtained by the bromine methanol method prescribed in Annex B for the determination of amount of metallic iron present (in this case, no correction factor need be added). Chemical analysis of carbon shall be done by the method as specified in IS 228 (Part 1).

6 SHAPE AND SIZE

6.1 The grain size of sponge iron depends on the production process and type of iron oxide used as feed. The size range of the sponge iron lumps for use in steel making shall be as follows:

Size	–	3 to 20 mm
+20 mm	–	5 percent, *Max*
–3 mm	–	5 percent, *Max*

This size range shall be valid for sponge iron at the despatch end only. The size range at the receiving point shall depend on the transport and handling during transit. Over and above, sponge iron may be obtained a maximum of one percent non-metallics which will not be considered in the size distribution nor in the chemical analysis of sponge iron. It is advisable that sponge iron should not be allowed to come in direct contact with water at any stage before usage to avoid its reoxidation.

Table 3 Chemical Composition of Cold Briquettes

(*Clause* 5.3)

Sl No.	Constituent	Grade		
		1	2	3
(1)	(2)	(3)	(4)	(5)
i)	Total iron	88 and above	87, *Min*	85-87
ii)	Metallization	90 ± 2	85, *Min*	82, *Min*
iii)	Metallic iron	78, *Min*	76, *Min*	70, *Min*
iv)	Carbon content:			
	1) Coal based, *Min*	0.3	0.3	0.3
	2) Gas based	1.3, *Min*	1.3, *Min*	1.3, *Min*
v)	S, *Max*	0.04	0.04	0.04
vi)	P, *Max*	0.06	0.06	0.06
vii)	Gangue, *Max* ($SiO_2 + Al_2O_3$)	8	9	10
viii)	Combined total of Pb, Zn, Cu, Sn, Cr, Ni, etc, *Max*	0.015	0.015	0.015

6.2 Hot Briquetted Iron (HBI)

Typical shape and size of the hot briquette follows:

Shape – Pillow

Nominal volume, mm^3 – 30 × 60 × 110 or 30 × 60 × 90

6.3 Cold Briquettes

Typical shape and size of the cold briquettes follows:

Shape – Pillow

Nominal volume, mm^3 – 64 × 30 × 20 or 38 × 44 × 19

7 DENSITY

The density of sponge iron/DRI when tested as per IS 14719, HBI and cold briquettes shall be as follows:

	Bulk Density g/cc	*Apparent Density* g/cc
Sponge iron/DRI	1.8	
HBI	2.4, *Min*	5.0, *Min*
Cold briquettes	2.2, *Min*	5-5.5

8 SAMPLING

8.1 Sponge Iron/DRI

8.1.1 Representative samples of sponge iron shall be drawn and prepared for chemical test and screen analysis as described in Annex C.

8.1.2 The screen analysis of sponge iron may be carried out in accordance with IS 1607.

8.1.3 The magnetic portion shall only be considered for the purpose of chemical analysis.

8.2 Hot Briquetted Iron (HBI)

8.2.1 Representative samples of HBI shall be drawn and prepared for chemical analysis and screen analysis as described in Annex D.

8.2.2 The screen analysis of HBI may be carried out in accordance with IS 1607.

8.3 Cold Briquettes

8.3.1 In case of fines, sampling procedures shall be as per IS 1405.

8.3.2 Representative samples of cold briquettes shall be drawn and prepared for chemical analysis and physical test as described in Annex E.

9 MARKING

The material shall accompany a test certificate giving the following:

a) Grade of the material,
b) Chemical composition,
c) Size specification, and
d) Manufacturer's name.

10 STORAGE AND TRANSPORTATION

10.1 Sponge iron/DRI needs special care during storage and transportation, guidelines for which have been covered under IS 10852.

10.2 During transportation of HBI and cold briquettes, the under size (below 5 mm) shall not be more than 5 percent.

ANNEX A

(*Clause* 5.5)

METHOD FOR DETERMINATION OF METALLIC IRON IN SPONGE IRON/DIRECT REDUCED IRON (DRI) BY THE FERRIC CHLORIDE METHOD

A-1 FERRIC CHLORIDE METHOD

A-1.1 Outline of the Method

The reaction between ferric chloride and metallic iron proceeds as follows:

$2FeCl_3 + Fe = 3\ FeCl_2$

The technique involves digestion of the sample in a ferric chloride solution followed by filtration and titration of the ferrous iron in the filtrate. Precautions should be taken to prevent atmospheric oxidation of the ferrous chloride formed.

A-1.2 Reagents

A-1.2.1 *Ferric Chloride [10 Percent (m/v)]* — Dissolve 100 g of ferric chloride ($FeCl_3$), $6H_2O$) in water and dilute to one litre.

A-1.2.2 *Hydrochloric Acid*, rd = 1.16, conforming to IS 265.

A-1.2.3 *Phosphoric Acid*, rd = 1.75

A-1.2.4 *Sodium Diphenylamine Sulphonate Indicator* – Dissolve exactly 0.32 of barium diphenylamine sulphonate in 100 ml of hot water. Add 0.5 g of sodium sulphate, stir and filter off the precipitate of barium sulphate. Store the filtrate in a dark-coloured bottle or 0.2 percent solution of sodium diphenylamine sulphonate indicator in water.

A-1.2.5 *Potassium Dichromate Solution* (0.1N) — Dissolve exactly 4.903 g of potassium dichromate (dried at 100 to 110°C) in water in 1 000-ml volumetric flask and make up the volume up to the mark. Mix thoroughly and use as a standard solution.

A-1.3 Procedure

Transfer 1 g of sample in a 500-ml conical flask and add 200 ml of ferric chloride solution. Create an inert atmosphere in the flask obtained through displacement of air by nitrogen, stopper the flask and agitate the solution with polypropylene coated magnetic stirrer for 1 h. Filter the solution through medium texture filter paper. Wash the residue with ferric chloride solution. Transfer the filtrate to one litre beaker containing 400 ml water, 25 ml sulphuric acid, 25 ml phosphoric acid, 3 to 4 drops of sodium diphenylamine sulphonate and titrate with potassium dichromate solution (0.1N).

$$1\ ml\ 0.1N K_2Cr_2O_7 = 0.001\ 862\ g\ of\ metallic\ iron\ Fe(M).$$

A-1.3.1 The above relationship of 1 ml (0.1N) $K_2Cr_2O_7$ = 0.001 862 Fe(M) is derived as follows:

a) 1 000 ml of $K_2Cr_2O_7$ (1N) = 55.85 g of Fe (T)
   or 1 000 ml of $K_2Cr_2O_7$ (0.1N) = 5.585 g of Fe (T)
   or 1 ml of $K_2Cr_2O_7$ (0.1N) = 0.005 585 g of Fe (T)

b) $Fe + 2FeCl_3 = 3FeCl_2$

Therefore three parts of $FeCl_2$ correspond to one part of Fe(M) or one part of $FeCl_2$ corresponds to 1/3 part of Fe(M).

$$\text{or 1 ml of } K_2Cr_2O_7\ (0.1N) = \frac{0.005\ 585}{3}$$
$$= 0.001\ 862\ g\ of\ Fe(M)$$

ANNEX B

(*Clause* 5.5)

METHOD FOR DETERMINATION OF METALLIC IRON IN SPONGE IRON/DIRECT REDUCED IRON (DRI) BY THE BROMINE METHANOL METHOD (REFEREE METHOD)

B-1 BROMINE METHANOL METHOD

B-1.1 Outline of the Method

The bromine and methanol mixture is added to the sample of sponge iron resulting in the dissolution of the metallic iron. The residue is separated by filtration, hydrochloric acid is added to the residue which contains oxides of Fe^{2+} and Fe^{3+} and it dissolves the FeO present. The addition of stannous chloride, reduces all the Fe^{2+} present in the residue Fe^{3+}. The excess stannous chloride is eliminated by reaction with mercuric chloride ($HgCl_2$). The amount of combined iron is determined. The difference between total iron and the combined iron is equal to the amount of metallic iron.

$$2Fe + 3Br_2 = 2Fe^{3+} + 6Br^-$$

$$2Fe^{3+} + Sn^{2+} = 2Fe^{2+} + Sn^{4+}$$

$$Sn^{2+} \text{ (excess)} + 2\,Hg^{2+} = Sn^{4+} + 2\,Hg^{+}$$

$$6Fe^{2+} + Cr_2O_7^{2-} + 14\,H^{+} = 6Fe^{3+} + 2Cr^{3+} + 7H_2O$$

B-1.2 Reagents

B-1.2.1 *Bromine Methanol Solution (50 ml/l)* — Dissolve 50 ml of bromine in 950 ml of water-free methanol. The solution shall be freshly prepared and used immediately.

NOTE — Proper care should be taken while preparation of solution and during analysis.

B-1.2.2 *Stannous Chloride Solution* — Dissolve by heating 60 g of pure stannous chloride in a mixture of 400 ml of concentrated hydrochloric acid and 600 ml of water until dissolution is completed. Cool and add a few pieces of granulated tin and preserve the solution in air-tight amber-coloured bottle to prevent oxidation.

B-1.2.3 *Mercuric Chloride Solution* — Prepare a saturated solution of mercuric chloride in water.

B-1.2.4 *Standard Potassium Dichromate Solution (0.1N)* — Dissolve exactly 4.903 g of potassium dichromate (dried at 100 to 110°C) in water in a 1 000-ml volumetric flask and make up the volume to the mark. Mix thoroughly and use as a standard solution or 0.2 percent solution of sodium diphenylamine sulphonate indicator in water.

B-1.2.5 *Hydrochloric Acid, rd* = 1.16, conforming to IS 265.

B-1.2.6 *Sodium Diphenylamine Sulphonate Indicator, See* **A-1.2.4**.

B-1.2.7 *Sulphuric Acid-Phosphoric Acid Mixture* — Add slowly and with stirring 150 ml of sulphuric acid (rd = 1.84) and 150 ml of phosphoric acid (rd = 1.71) to 700 ml of water. Destroy any oxidizable impurities by adding potassium permanganate solution (0.1N) drop by drop until the pink colour of permanganate persists and cool the solution.

B-1.3 Procedure

B-1.3.1 *Combined Iron*

Weigh 0.5 g of sample into a 500-ml ground glass joint flask. Add 100 ml of bromine methanol solution and fit the flask to reflux condenser. Cool, filter and wash the residue with distilled water, then with methanol and again with distilled water.

B-1.3.2 Carefully remove the residue and wash with hydrochloric acid (1:1). Add 45 ml hydrochloric acid and allow to dissolve covering the flask with a watch-glass. Add stannous chloride to the solution, until the solution becomes colourless. Add 2 drops of stannous chloride in excess. Allow the solution to cool and add 15 ml of mercuric chloride (5 percent). Allow to stand for two minutes and add 25 ml of acid mixture. Add 3-4 drops of indicator and titrate with potassium dichromate solution (0.1N). The solution changes its colour from colourless to yellow, green and end point is purple.

B-1.4 Calculation

$$\text{Combined iron, percent} = \frac{A \times B \times 0.055\,85 \times 100}{C}$$

A = volume of potassium dichromate solution consumed, in ml;

B = strength of the standard dichromate solution; and

C = mass of the sample taken, in g.

B-1.4.1 *Total Iron*

Total iron shall be determined in accordance with the procedure laid down in IS 1493 (Part 1).

B-1.4.2 *Metallic Iron*

Metallic iron shall be determined by the difference of total iron and combined iron that is,

Metallic iron = Total iron – Combined iron

ANNEX C

(*Clause* 8.1.1)

SAMPLING OF SPONGE IRON

C-0 The methods for sampling are applicable to the taking of samples of DRI from conveyors, railways wagons or containers (including trucks) and stockpiles, during the loading or discharging of a lot in cases where manual sampling can be carried out safely.

C-1 GENERAL PROCEDURES FOR MANUAL SAMPLING

Sampling shall be carried out while a lot is being transferred. The general sampling procedure shall be as follows:

a) Identify the lot to be sampled,
b) Ascertain the nominal lot size, and
c) Determine the mass of increment considering the nominal lot size.

C-2 INCREMENTS

C-2.1 Minimum Mass of Increment

C-2.1.1 The mass of each increment shall be as specified in Table 4 according to the nominal lot size of the DRI being sampled.

Table 4 Minimum Mass of Increment

Sl No.	Nominal Top Size		Minimum Mass of Increment
	Over	Up to and Including	kg
	mm	mm	
(1)	(2)	(3)	(4)
i)	50	–	12
ii)	22.4	50	4
		22.4	0.8

C-2.1.2 Increments shall be taken in such a manner as to ensure that they are of almost uniform mass.

C-2.2 Number of increments to be taken from a lot shall be as given in Table 5.

Table 5 Minimum Number of Increments

Sl No.	Mass of Lot, mt		Number of Increments
	Over	Up to and including	
(1)	(2)	(3)	(4)
i)	30 000	–	35
ii)	15 000	30 000	30
iii)	5 000	15 000	25
iv)	2 000	5 000	20
v)	1 000	2 000	15
vi)	500	1 000	10
vii)	–	500	6

C-2.3 While taking increments, special care shall be taken to obtain representative increments because of the segregation of fine and coarse particles.

C-2.4 The sampling device used should have width 6 times of the nominal lot size.

The volume of the device in the effective collection area should be sufficient to hold at least twice the minimum mass of increment.

C-3 METHODS OF MANUAL SAMPLING

C-3.1 Sampling from Conveyors

C-3.1.1 When the increment is taken from a stopped conveyor belt, a section of adequate length in the direction of the stream and of the full width and thickness of the DRI stream, should be taken.

C-3.1.2 When the increment is taken from a moving conveyor, the full width and thickness of the DRI stream shall be taken by a mechanically assisted device from the failing stream.

C-3.2 Sampling from Wagons or Containers

C-3.2.1 The increment shall be taken at random from the new surface of DRI exposed during the loading or unloading of the wagons or trucks.

C-3.2.2 When it is suspected that there is some bias between strata (between the top and bottom, the front and the rear, or the left and the right) in the DRI in the wagon or truck, it is advisable to take increments from each such stratum.

C-3.3 Sampling from Bunker Discharge

The sampling of DRI from bunker discharge shall be conducted in accordance with the method specified in **C-3.1**.

C-3.4 Sampling from Stockpiles

The sampling of DRI from stockpiles shall be performed from conveyors either by stopped belt sampling or from a transfer point in accordance with the method specified in **C-3.1** while the stockpile is being formed or reclaimed.

The sponge iron should be stacked in geometric shapes preferably 100 mt in quantity. The quantity of sponge iron shall be assessed from bulk density if it is not weighed. The height of the stack should not be more than 1.5 m. A bulk sample from central portion of 1 m to 1.5 m diameter on the top shall be made to expose

the bottom of the stack. The trenches of 0.5 m, *Min* depth shall be made longitudinally on four sides to expose the materials. Samples with the help of double scoops shall be collected in such a manner that the size distribution of the sample shall be proportionate to the size distribution of the stockpile. The scoop should be moved from bottom to the top of the trench.

C-4 PROCEDURE FOR SAMPLE PREPARATION

The preparation of samples of DRI shall be conducted with extreme care to minimize the chance of reoxidation due to dampness, overheating or both. All equipments should be thoroughly cleaned to remove remnants of deleterious material and it is desirable to flush the equipment just prior to use with a small quantity of the same DRI.

C-4.1 Crushing and Grinding

The crushing and grinding shall be conducted with a crusher and a grinder suitable for the size and mechanical strength of the DRI particles. The crusher and grinder should be purged just before use with DRI from the same source. Precautions shall be taken to minimize overheating and reoxidation and to avoid the production of flakes of metal.

C-4.1.1 From the gross sample +10 mm size shall be crushed to –10 mm with the help of jaw crusher and mixed along with –10 mm fractions already screened. The mixed material shall be split up for reduction of volume. When the sample quantity attains around 5 kg, then the material is further crushed to –6 mm, mixed thoroughly and divided by splitter till the final quantity is around 1 kg. The sample thus obtained shall be ground in disc grinder to –150 µm size. Special precautions should be taken to ensure that grinding process does not generate excessive heat which could significantly change the chemical composition.

C-4.2 Mixing

By mixing the sample thoroughly it can be made homogeneous and consequently the errors in sample division can be lessened. The mixing may be conducted either by a mechanical mixer or by hand. The mixer shall be selected to suit the sample and its particle size.

C-5 APPARATUS FOR SAMPLE PREPARATION

The following apparatus which shall be thoroughly cleaned and examined before and after use, shall be provided for sample preparation:

a) Crusher and grinder, for example, jaw crusher, cone crusher, vertical mill ring grinder and agate pestle and mortar;
b) Mixers, for example, double cone mixer;
c) Riffles;
d) Scoop crusher; and
e) Disc pulverizer.

C-6 PREPARATION OF TEST SAMPLES

C-6.1 Preparation of Test Samples for Size Analysis

The test sample for size analysis should be prepared from each increment, each partial sample or the gross sample without mixing.

C-6.2 Preparation of Test Sample for Chemical Analysis

The test sample for chemical analysis may be prepared from each increment, each partial sample of the gross sample.

C-6.2.1 *Mass and Size of Test Sample for Chemical Analysis*

A test sample of 100 g, *Min* at –150 µm in particle size shall be prepared.

C-6.2.2 *Preparation of Test Sample for Chemical Analysis*

C-6.2.2.1 Prepare a sample of 500 g min at –250 µm in particle size from each increment, each partial sample of the gross sample according to the division methods.

C-6.2.2.2 Grind the above sample to –150 µm in particle size and from this sample, prepare a set of not less than four samples each of 100 g, *Min* by an appropriate division method. Seal the samples and distribute them to the laboratories concerned 5 um from –250 µm in particle size, special precautions should be taken to ensure that the grinding process does not generate excessive heat which could significantly change the chemical composition. Precautions may include the following:

a) Reducing the grinding time by grinding smaller charges,
b) Use a single pass straight through type of grinder,
c) In the grinding of samples, usually some material remain sticking to the surface of the pot specially when the material is of high metallization. Care should be taken to include this sticky material also while entire lot of powder is taken out after grinding, and
d) Some material sticking to the surface of pot should also be included while the entire lot of powder is taken out after grinding.

C-7 PACKING AND MARKING OF SAMPLE FOR CHEMICAL ANALYSIS

The chemical analysis sample for distribution shall be

tightly sealed in air-tight containers. The label and a card placed in the container shall contain the following particulars:

a) Type of DRI and name of lot (Name of ship, train, etc);
b) Mass of lot;
c) Sample number;
d) Place and date of sample;
e) Place and date of sample preparation; and
f) Any other items, if necessary.

Of the samples prepared, one sample shall be provided for the supplier, one for the purchaser and one for the arbitrator and if required one held in reserve. The reserve samples shall be retained for six months.

ANNEX D

(*Clause* 8.2.1)

REPRESENTATIVE SAMPLES OF HBI

D-1 SAMPLING

The sampling of the hot briquetted sponge iron will be done at the following points:

a) On piles,
b) On trucks or wagons, and
c) On conveyor.

D-2 SAMPLING FROM PILES

From every 500 mt or less hot briquetted sponge iron, a minimum of one gross sample shall be collected. The gross sample shall consist of 10 increments of 10 kg each. This 100 kg briquettes shall be used for both chemical as well as screen analysis as shown in Fig.1.

D-3 SAMPLING ON TRUCKS OR WAGONS

From each truck or wagon, one increment of suitable weight shall be drawn. All increments thus drawn shall constitute a gross sample. The total mass of the gross sample shall be 50 kg which shall be used for both chemical as well as screen analysis as shown in Fig. 1.

D-4 SAMPLING ON CONVEYOR

From conveyor increments of suitable weight shall be drawn at regular intervals. All increments thus drawn shall constitute a gross sample. The total mass of the gross sample shall be 50 kg which shall be used for both chemical as well as screen analysis as shown in Fig. 1.

D-5 PREPARATION OF SAMPLE FOR CHEMICAL TESTS AND SCREEN ANALYSIS

The laboratory samples shall be obtained by the procedure given in Fig. 1. The sample shall be stored with the following identification:

a) Lot number,
b) Sample number, and
c) Sampling date.

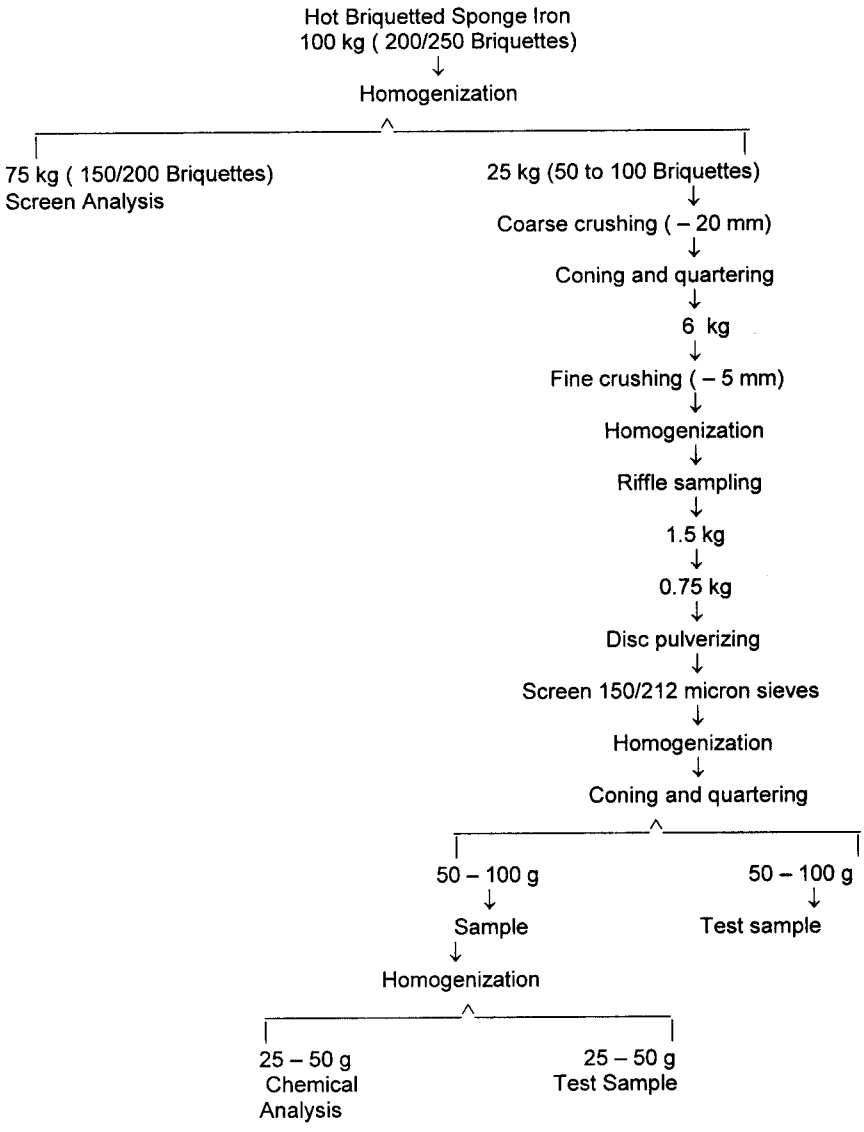

FIG. 1 REDUCTION OF SAMPLE TO FINAL STAGE FOR CHEMICAL/SCREEN ANALYSIS

ANNEX E

(*Clause* 8.3.2)

REPRESENTATIVE SAMPLES OF COLD BRIQUETTES

E-1 SAMPLING

The sampling of the cold briquetted sponge iron shall be done at the following points:

a) On piles,
b) On trucks or wagons, and
c) On conveyers.

Sampling of briquettes shall be done preferably in dry weather and the following procedure shall be adopted at different points.

E-2 SAMPLING FROM PILES

For every 50 tonnes or less of cold briquetted sponge iron, a minimum of one gross sample shall be collected. The gross sample shall consist of 10 increments of 2.5 kg each. This 25 kg briquettes shall be used for both chemical as well as screen analysis as shown in Fig. 2.

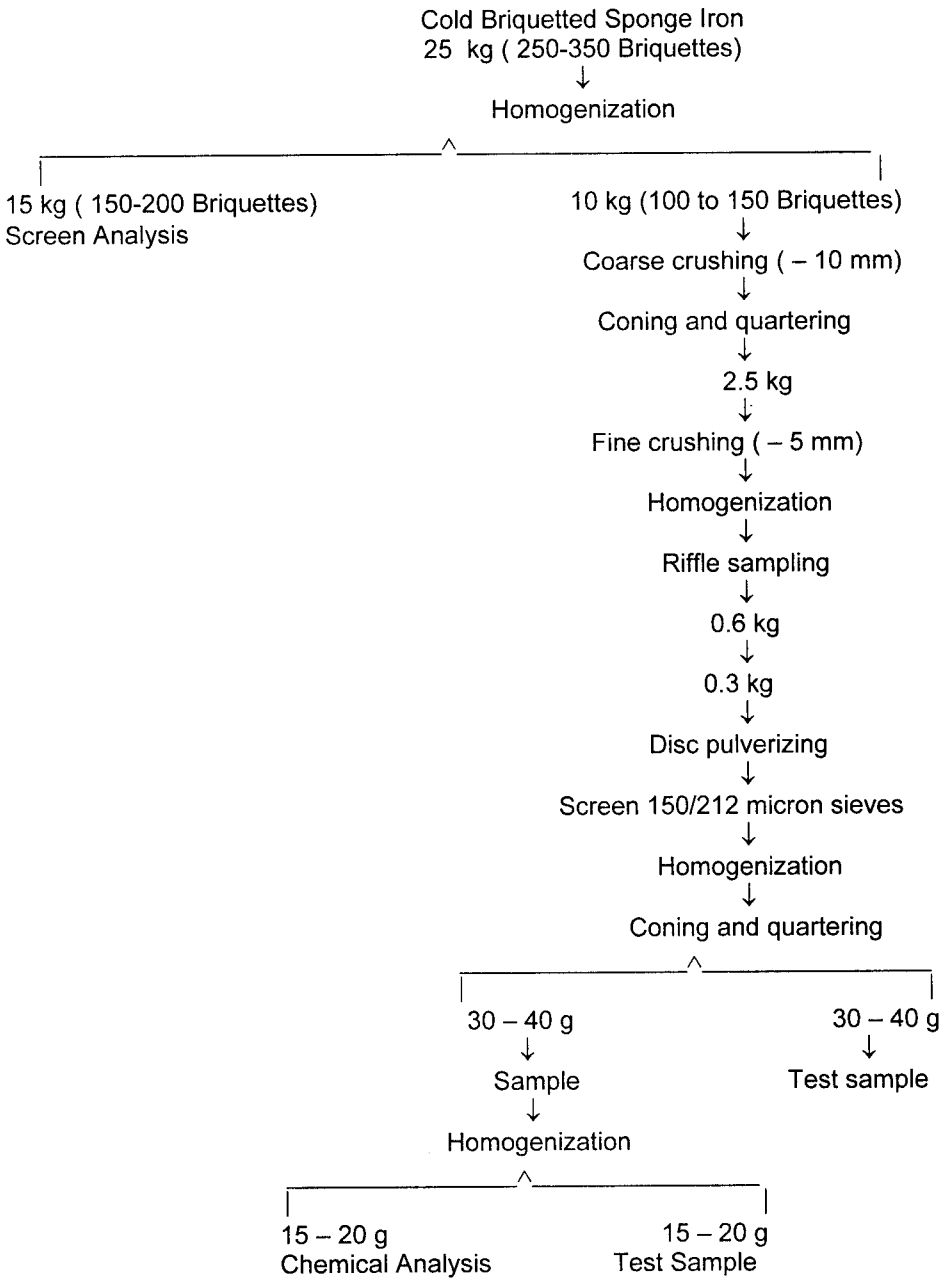

FIG. 2 REDUCTION OF SAMPLE TO FINAL STAGE FOR CHEMICAL/SCREEN ANALYSIS

E-3 SAMPLING ON TRUCKS OR WAGONS

From each truck or wagon, one increment of suitable weight shall be drawn. All increments thus drawn shall constitute a gross sample with total mass of the gross sample shall be 25 kg which shall be used for both chemical as well as screen analysis as shown in Fig. 2.

E-4 SAMPLING ON CONVEYOR

From conveyor, increments of suitable weight shall be drawn at regular intervals. All increments thus drawn shall constitute a gross sample. The total mass of the gross sample shall be 25 kg which shall be used for both chemical as well as screen analysis as shown in Fig. 2.

E-5 PREPARATION OF SAMPLE FOR CHEMICAL TESTS AND SCREEN ANALYSIS

The laboratory samples shall be obtained by the procedure given in Fig. 2. The samples shall be stored with the following identifications:

a) Lot number,
b) Sample number, and
c) Sampling date.

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