

भारतीय मानक मसौदा
बेरियम सल्फाइड, तकनीकी (काली राख) — विशिष्टि
(IS 5877 का पहला पुनरीक्षण)

Draft Indian Standard
Barium Sulphide, Technical (Black Ash) — Specification
(*First Revision of IS 5877*)

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ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01

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FOREWORD

(*formal clauses will be added later*)

This standard was first published in 1971. In this first revision, Reference clause has been incorporated. Also, Packing and Marking clause has been updated.

Barium sulphide (black ash) is the most important raw material for the manufacture of other barium compounds like blanc fixe lithophone, barium carbonate, barium nitrate, barium chloride and barium hydroxide. It is also used for the removal of hair from hides; for the preparation of hydrogen sulphide free from arsenic; for the manufacture of barium polysulphide for use as a pesticide; and in vulcanizing rubber.

Barium sulphide is evaluated in trade on the basis of its water-soluble barium sulphide and acid soluble barium compounds only. Hence in this standard, only the requirements of these characteristics have been specified and the limits of impurities, like silica, iron, carbon and unreacted barium sulphate, have not been incorporated. The presence of silica, carbon and unreacted barium sulphate is, in fact, immaterial since they dissolve neither in water nor in the acid. The iron compounds are not water soluble and in acid extraction, they are eliminated at the neutral point and, therefore, also do not matter in actual practice.

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 2:2022 Rules for rounding off numerical values (*second revision*). The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Draft Indian Standard
BARIUM SULPHIDE, TECHNICAL (BLACK ASH) —
SPECIFICATION
(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for barium sulphide, technical, known in the trade as 'black ash'.

2 REFERENCES

The standards given below 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 edition of these standards:

<i>IS No.</i>	<i>Title</i>
IS 265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth Revision</i>)

3 REQUIREMENTS

3.1 Description

Barium sulphide is a powdery, black grey, porous mass, which absorbs carbon dioxide and moisture from air, and is oxidized.

3.2 The material shall comply with the requirements given in Table 1 when tested in accordance with the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 4 of the table.

Table 1 Requirements for Barium Sulphide, Technical

(Clause 3.2, B-4.1 and B-5.1)

Sl. No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Annex A)
(1)	(2)	(3)	(4)
i)	Water solubles (as barium sulphide), percent by weight, <i>Min</i>	60.0	A-2
ii)	Acid solubles (as barium sulphide), percent by weight, <i>Max</i>	25.0	A-3

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in clean and dry jute bags lined with polyethylene or mild steel drum lined with polyethylene, so as to avoid contact with air.

4.2 Marking

Each container shall be legibly and indelibly marked with the following information:

- a) Name and grade (technical) of the material;
- b) Net weight of the contents;
- c) Year of manufacture;
- d) Manufacturer's name and/or recognized trade-mark, if any; and
- e) Lot number in code or otherwise to enable the batch of manufacture to be traced from records.

4.2.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.

5 SAMPLING

The method of drawing representative samples of the material, number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Annex B.

ANNEX A

(Clause 3.2)

METHODS OF TEST FOR BARIUM SULPHIDE, TECHNICAL

A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF WATER SOLUBLES

A-2.1 Reagents

A-2.1.1 *Iodine Solution* — 0.1 N.

A-2.1.2 *Dilute Hydrochloric Acid* — 5 N approximately.

A-2.1.3 *Standard Sodium Thiosulphate Solution* — 0.1 N.

A-2.1.4 *Starch Indicator Solution*

Triturate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of water in a mortar. Pour the resulting paste into 1 litre of boiling water and continue boiling for 3 min, allow the solution to cool and decant off the clear liquid.

A-2.2 Procedure

Weigh accurately about 3 g of finely powdered sample in a beaker. Add about 50 ml of hot water and boil for about a minute. Filter on a Buchner funnel using filter paper Whatman No. 40 or equivalent. Extract four times with hot water and preserve the residue for the estimation of acid soluble barium sulphide in **A-3**. Make up the volume of the filtrate to 500 ml in a volumetric flask.

A-2.2.1 Take 20 ml of iodine solution into a 250 ml iodine flask and add a little dilute hydrochloric acid. Add 20 ml aliquot of the filtrate in the same flask and keep it for 5 min in a dark place. Titrate the excess of iodine with standard sodium thiosulphate solution using starch indicator solution. Conduct a blank simultaneously with the same reagents.

A-2.3 Calculation

$$\text{Water solubles (as barium sulphide), percent by weight} = \frac{211.78 \times (V_1 - V_2) \times N}{W}$$

where

V_1 = volume in ml of sodium thiosulphate solution consumed in the blank titration;

V_2 = volume in ml of sodium thiosulphate solution consumed in titration with the sample solution;

N = normality of sodium thiosulphate solution; and

W = weight in g of the material taken for the test.

A-3 DETERMINATION OF ACID SOLUBLES

A-3.1 Reagents

A-3.1.1 *Concentrated Hydrochloric Acid* — *see* IS 265.

A-3.1.2 *Dilute Sulphuric Acid* — approximately 1:2 and 1:99 (v/v).

A-3.2 Procedure

Transfer the residue reserved in **A-2.2** with filter paper to a 400 ml beaker, add 20 ml of concentrated hydrochloric acid and boil; filter and wash with hot water. Heat the solution to boiling, add 5 ml of hot dilute sulphuric acid (1:2) slowly with constant stirring. Cool, dilute to about 200 ml and allow to stand for 4 h at 60 °C. Filter the precipitate through Gooch crucible or filter paper Whatman No. 42 or equivalent. Wash the precipitate twice with dilute sulphuric acid (1:99) and then with hot water. Dry the precipitate and ignite slowly at low flame until the filter paper is thoroughly ashed. Cool, moisten with a few drops of dilute sulphuric acid (1:2). Heat on the burner till the excess acid is fumed off and finally heat at 800 °C in a furnace till constant weight is obtained.

A-3.3 Calculation

$$\text{Acid solubles (as barium sulphide), percent by weight} = \frac{100 \times 0.725 \times W_1}{W}$$

where

W_1 = weight in g of the barium sulphate precipitate obtained after ignition; and

W = weight in g of the material taken for the test.

NOTE — In actual practice, the acid soluble part of the residue is converted to barium chloride. The acid soluble part consists of barium carbonate, silicate, etc. Only barium carbonate is determined and expressed as barium sulphide.

ANNEX B

(Clause 5)

SAMPLING OF BARIUM SULPHIDE, TECHNICAL (BLACK ASH)**B-1 GENERAL REQUIREMENTS OF SAMPLING**

B-1.1 In drawing, preparing, storing and handling the samples, the following precautions shall be observed.

B-1.2 Samples shall not be taken at a place exposed to the adverse effects of weather.

B-1.3 The sampling instrument and sample containers shall be clean and dry.

B-1.4 Before taking the sample, the contents of the selected packages shall be thoroughly mixed.

B-1.5 After filling, the sample containers shall be sealed and marked with relevant particulars.

B-1.6 The material being caustic, particular care shall be taken in its handling.

B-2 SCALE OF SAMPLING**B-2.1 Lot**

All the packages in a consignment of the material drawn from a single batch of manufacture shall constitute a lot. Each lot shall be tested separately for all the requirements of this specification.

B-2.2 The number of packages to be sampled at random from a lot depends on the lot size and shall be as given in Table 2.

Table 2 Number of Packages to be Selected for Sampling*(Clause B-2.2)*

Lot Size	Number of Packages to be Selected
<i>N</i>	<i>n</i>
(1)	(2)
Up to 25	3
26 to 50	4
51 to 100	5
101 to 200	6
201 and above	8

B-3 PREPARATION OF TEST SAMPLES

Draw with an appropriate sampling implement about 20 g of barium sulphide from different parts of each of the selected packages. Equal quantities from each of these samples shall be taken and mixed together to form a composite sample weighing 50 g.

B-4 NUMBER OF TESTS

Tests for the determination of characteristics laid down in Table 1 shall be conducted on composite sample.

B-5 CRITERIA FOR CONFORMITY

For declaring the conformity of the lot to the requirements of all the characteristics, the composite sample shall satisfy the relevant requirements in col 4 of Table 1.