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Indian Standard

SPECIFICATION FOR BENZOTHIAZYL-2-CYCLOHEXYL SULPHENAMIDE

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Indian Standard

SPECIFICATION FOR BENZOTHIAZYL-2-CYCLOHEXYL SULPHENAMIDE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 27 September 1973, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Chemical Division Council.

0.2 Benzothiazyl-2-cyclohexyl sulphenamide is a well known accelerator with a delayed onset of cure, primarily used in the tyre industries.

0.3 It is soluble in benzene, carbon tetrachloride, acetone, slightly soluble in ethanol and insoluble in water. In order to facilitate proper application of this member of the sulphenamide class of accelerators in the rubber industry, this standard indicating chemical requirement and technological evaluation procedure has been brought out.

0.4 The material being a reactive chemical, all persons concerned with handling of this chemical should be thoroughly informed about the nature of the material, possible effect on contact, inhalation and accidental ingestion. Further, since it is unstable compound it should be stored in a cool place.

0.5 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*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for benzothiazyl-2-cyclohexyl sulphenamide intended for use in rubber compounding as a delayed action accelerator. This standard is only for the powder form (without being treated with dedusting or binding agents).

*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — The material shall be in the form of light creamy grey powder and shall be free from visible impurities.

2.2 The material shall also comply with the requirements given in Table I when tested according to the procedures given in col 4 of the table.

TABLE 1 REQUIREMENTS OF BENZOTHIAZYL-2-CYCLOHEXYL SULPHENAMIDE

Sl No.	Characteristic	Requirement	Method of Test, Ref to Cl No.
(1)	(2)	(3)	(4)
i)	Melting point:		A-3 of IS : 6918-1972*
	a) Appearance of first droplet	Not below 92°C	
	b) Completion of melting	Not below 98°C and not above 105°C	
ii)	Residue on 150-microns IS Sieve, percent by mass, *Max*	0·075	A-4 of IS : 6918-1972*
iii)	Sulphated ash, percent by mass, *Max*	0·5	A-6 of IS : 6918-1972*
iv)	Copper, ppm, *Max*	20	A-7 of IS : 6918-1972*
v)	Relative density at 27°C/27°C	1·28 to 1·35	4 of IS : 7086-1973†
vi)	Volatile matter, percent by mass, *Max*	1·0	A-2 of Appendix A
vii)	Free amine, percent by mass, *Max*	1·0	A-3 of Appendix A
viii)	Assay, percent by mass, *Min*	97·0	A-4 of Appendix A
ix)	Ethanol insolubles, percent by mass, *Max*	1·0	A-5 of Appendix A

*Specification for mercaptobenzothiazole.
†Methods of sampling and test for rubber compounding ingredients, Part I.

2.2.1 All tests shall be carried out within 15 days of the receipt of the material by the purchaser.

2.3 Compounding — The material when compounded and tested according to the procedure given in Appendix B shall have performance characteristics comparable to standard material satisfying the requirements of **2.1** and **2.2**.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed securely so as to avoid exposure to air, moisture and acidic atmosphere. The exact mode of packing shall be as agreed to between the purchaser and the supplier.

3.2 Marking — The packages shall have the following information marked on it:

a) Manufacturer's name and registered trade-mark, if any;
b) Chemical name or trade name of the material;
c) Net mass of the material; and
d) Lot and batch number.

3.2.1 The packages may also be marked with the ISI Certification Mark.

NOTE — *The use of the ISI Certification Mark is governed by the provisions of the* Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked porducts are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING AND CRITERIA FOR CONFORMITY

4.1 Sampling — For the purpose of ascertaining the conformity of benzothiazyl-2-cyclohexyl sulphenamide in a consignment to this specification, sampling as prescribed in **15** of IS : 7086-1973* shall be followed.

4.2 Number of Tests

4.2.1 Test for copper (*see* Table 1) shall be conducted on individual samples.

4.2.2 Tests for all other characteristics shall be conducted on a composite sample.

4.3 Criteria for Conformity

4.3.1 *Copper* — The mean and range of test results for copper shall be calculated as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{The sum of test results}}{\text{Number of test results}}$$

$$\text{Range } (R) = \text{The difference between the maximum and minimum value of the test results.}$$

The lot shall be deemed to have satisfied the requirement of the specification if:

$$\bar{X} + 0{\cdot}6\,R \leqslant 20$$

*Methods of sampling and test for rubber compounding ingredients, Part I.

4.3.2 *Composite Sample* — In respect of all other characteristics the lot shall be considered as conforming to the specification if the composite sample satisfies each one of these requirements.

APPENDIX A

(*Table* 1)

TEST METHODS FOR BENZOTHIAZYL-2-CYCLOHEXYL SULPHENAMIDE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in the tests.

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

A-2. VOLATILE MATTER

A-2.1 Apparatus

A-2.1.1 *Glass Petri Dish* — 12 cm in diameter having 40 ml capacity.

A-2.2 Procedure — Weigh 25 g accurate to 1 mg of the material and spread it evenly on the dish. The dish is placed in an oven maintained at 65°C for one hour. The loss in mass is recorded.

A-2.3 Calculation

$$\text{Volatile matter, percent by mass} = \frac{M_1 - M_2}{M_1} \times 100$$

where

M_1 = mass of the sample taken, and

M_2 = mass of the sample after heating.

A-3. DETERMINATION OF FREE AMINE

A-3.1 Procedure — Weigh approximately 5 g of the material accurately to 1 mg and put in a 300-ml Erlenmeyer flask. Add 150 ml of isopropanol-toluene mixture (5 : 3) to dissolve the material. The solvent mixture of isopropanol-toluene should be neutralized with respect to bromophenol

*Specification for water, distilled quality (*revised*).

blue to a greenish yellow end point before use. After adding a few drops of bromophenol blue indicator the solution is titrated with 0·1 N hydrochloric acid till the appearance of yellow colouration.

A-3.2 Calculation

$$\text{Free amine, percent by mass} = \frac{0{\cdot}99\,V}{M}$$

where

V = volume in ml of 0·1 N hydrochloric acid used, and
M = mass in g of the material taken for the test.

A-4. ASSAY

A-4.1 Procedure — Proceed as in **A-3.1**. Pass hydrogen sulphide gas for 15 to 20 minutes from the solution in which the free amine has been neutralized with 0·1 N hydrochloric acid. Titrate the liberated amine with 0·5 N hydrochloric acid till yellow colouration persists for one minute.

A-4.2 Calculation

$$\text{Assay, percent by mass} = \frac{13{\cdot}2\,V}{M}$$

where

V = volume in ml of 0·5 N hydrochloric acid used, and
M = mass in g of the material taken for the test.

A-4.3 If agreed to between the purchaser and the supplier ultra-violet spectrophotometric method may also be used.

A-5. DETERMINATION OF ETHANOL INSOLUBLES

A-5.1 Procedure — Weigh 4 g of the material to the nearest milligram and put in a 400-ml beaker. Add 250 ml of ethanol into it. Stir it at room temperature for 30 minutes with the help of a magnetic stirrer. Allow the residue to settle. Decant the bulk of the supernatant liquid. Transfer the residue as thick slurry on to a previously weighed sintered glass crucible (porosity No. 3). Wash the cake two or three times with 25 ml of ethanol and then suck it dry. Dry the crucible at 100°C and weigh.

A-5.2 Calculation

Ethanol insolubles, percent by mass = mass of the residue × 20

APPENDIX B

(*Clause* 2.3)

METHOD FOR COMPOUNDING AND TESTING OF BENZOTHIAZYL-2-CYCLOHEXYL SULPHENAMIDE

B-1. TEST COMPOUND

B-1.1 As a guidance, the following test compound may be used for testing performance properties of benzothiazyl-2-cyclohexyl sulphenamide in rubber compound:

	Parts by mass
Natural rubber, Grade A (conforming to IS : 4588 - 1968*)	100
Carbon black, HAF	45
Zinc oxide (conforming to IS : 3399-1973†)	5
Mineral oil (aromatic) plasticizer	5
Stearic acid (conforming to IS : 1675 - 1971‡)	3
Sulphur	2·3
Benzothiazyl-2-cyclohexyl sulphenamide	0·5

B-2. COMPOUNDING

B-2.1 Follow the procedure prescribed in NR : 9 of IS : 3660 (Part I) 1966§.

B-3. TESTS

B-3.1. The tests given below are recommended for each test sample. The approved sample shall also be tested side by side using the same master batch, that is, compound excluding sulphur and accelerator only.

B-3.1.1 Mooney scorch test shall be done at 120°C in accordance with the method prescribed in NR : 8 of IS : 3660 (Part I)-1966§.

*Specification for raw natural rubber.
†Specification for zinc oxide for rubber industry (*first revision*).
‡Specification for stearic acid, technical (*first revision*).
§Methods of test for natural rubber, Part I.

B-3.1.2 Tensile strength, modulus at 300 percent elongation, elongation at break at different cures at 141°C (from below to above the expected optimum cure) shall be tested in accordance with the method prescribed in IS : 3400 (Part I)-1965*.

B-3.1.3 Hardness on optimum cure at 141°C shall be tested in accordance with the method prescribed in IS : 3400 (Part II)-1965†.

B-4. RESULT

B-4.1 The values obtained with the test sample shall not vary by more than ± 20 percent for Mooney scorch and ± 10 percent for all the other characteristics from those obtained with the approved sample.

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*Mehods of test for vulcanized rubbers: Part I Tensile stress-strain properties.
†Methods of test for vulcanized rubbers: Part II Hardness.

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