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SPECIFICATION FOR RUBBER SEALING RINGS FOR DOMESTIC FRUIT AND VEGETABLE PRESERVING JARS

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 1

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SPECIFICATION FOR RUBBER SEALING RINGS FOR DOMESTIC FRUIT AND VEGETABLE PRESERVING JARS

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SPECIFICATION FOR RUBBER SEALING RINGS FOR DOMESTIC FRUIT AND VEGETABLE PRESERVING JARS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 5 June 1969, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Chemical Division Council.

0.2 All compounding ingredients used in the rubber compound from which sealing rings are made should be free from harmful materials liable to extraction by contact with foodstuffs or which may cause the development of undesirable odour, taste, or discolouration. The types of compounding ingredients recommended for the purpose are given in Appendix A for the guidance of manufacturers.

0.3 An Indian Standard on glass container finishes covering standard neck sizes is under preparation. It is necessary that the dimensions of rubber sealing rings shall correspond to the details given in the above standard to ensure air-tight sealing. The option of selecting a particular standard neck size has been left to the purchaser of glass containers. This standard, therefore, prescribes only tolerances on various dimensions of rings.

0.4 This standard contains clauses **2.3.1**, **3.1** and **C-2.2.1**, and Table 1 which call for agreement between the purchaser and the supplier.

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 tests for rubber sealing rings to be used for domestic fruit and vegetable preserving jars.

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

2. REQUIREMENTS

2.1 Material

2.1.1 The sealing rings shall be made from rubber, compounded and vulcanized.

2.1.2 All the compounding ingredients used in the rubber shall be free from harmful materials liable to extraction by contact with foodstuffs or which may cause the development of undesirable odour, taste or discolouration.

2.1.3 For coloured rings, mercuric sulphide, lithophone and zinc sulphide shall not be used. Inorganic pigments and organic dyestuffs used shall satisfy the requirement prescribed in **2.1.2**.

2.2 Workmanship and Finish

2.2.1 The rings shall be free from visual defects in material, workmanship and finish.

2.2.2 The rings of each batch shall be of uniform colour.

2.3 Dimensions — The dimensions of the rings shall be as agreed to between the purchaser and the supplier.

2.3.1 *Tolerances* — The dimensions shall, however, be subjected to the following tolerances:

Dimension	*Tolerance*
	mm
Inside or outside diameter	$\pm$ 0·50
Width	$\pm$ 0·20
Thickness	$\begin{cases} + 0{\cdot}15 \\ - 0{\cdot}10 \end{cases}$

2.3.2 Each ring shall be uniform in diameter (inside or outside), width and thickness.

2.4 The rings shall further comply with the requirements prescribed in Table 1.

3. PACKING AND MARKING

3.1 Packing — The rubber sealing rings shall be packed as agreed to between the purchaser and the supplier.

3.2 Marking — Each package shall be marked with the name of the manufacturer or recognized trade-mark, if any; number of rings in each package; batch number; and date of packing.

TABLE 1 REQUIREMENTS FOR RUBBER SEALING RINGS

(*Clause* 2.4)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Appendix B)
(1)	(2)	(3)	(4)
i)	Nature of water extractive	No colour, odour or taste shall be developed or precipitate formed in the extracted solution	B-1
ii)	Free sulphur, percent by weight, *Max*	0·2	B-2
iii)	Total zinc oxide, percent by weight, *Max*	2·0	B-3
iv)	Heavy metals	Shall pass the test	B-4
v)	*p*H of aqueous extract	7·0 ± 0·2	B-5
vi)	Tension set before heat treatment, percent, *Max*	10	B-6
vii)	Change in tension set on heat treatment, percent, *Max*	10	
viii)	Initial hardness, IRHD*	55 to 70	B-7
ix)	Change from initial hardness on ageing, IRHD*	±3	
x)	Low temperature flexibility	To be agreed to between the purchaser and the supplier as an optional requirement depending upon the storage conditions.	

*International Rubber Hardness Degree.

3.2.1 The package 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. Presence of this 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 during production. This system, which is devised and supervised by ISI and operated by the producer, has the further safeguard that the products as actually marketed are continuously checked by ISI for conformity to the standard. 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

4.1 Scale of Sampling and Criteria for Conformity — For the purpose of ascertaining conformity of the material to this standard the scale of sampling and criteria for conformity shall be as prescribed in Appendix C.

5. TESTS

5.1 All tests shall be carried out within three months of the delivery.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

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

APPENDIX A

(*Clause* 0.2)

RECOMMENDED COMPOUNDING INGREDIENTS

A-1. GENERAL

A-1.1 In giving the following data it is not intended to imply that the use of alternative materials may not give rings of a suitable quality. The recommendations are, in every case, intended mainly to indicate types of materials which have been found, by practical experience, to be suitable for producing rings to be used in the domesic glass jars meant for preservation of fruits and vegetables.

A-2. COMPOUNDING

A-2.1 The rings may be made from natural rubber, or some suitable synthetic non-toxic elastomer or a blend of the two together with the necessary compounding ingredients. The ingredients should be free from the prohibited materials specified in **2.1.**

NOTE — In case of natural rubber rings, light coloured crepe, of natural rubber that has been rendered suitable for extrusion (superior processing rubber), air-dried plantation rubber that is free from *p*-nitro-phenol (so called air-dried sheets) and in case of synthetic rubber rings, cis-1, 4-polyisoprene, polybutadiene, styrene-butadiene copolymers, or nitrile-butadiene copolymers, and chloroprene rubber (polychloroprene) or a blend of any of these may be used as raw material. Divinyl benzene may be used as a third polymerization component.

A-3. ACCELERATORS

A-3.1 Recommended accelerators of the tasteless type to suit the requirements specified in **2.1.2** are thiuram disulphides or monosulphides, dithiocarbamates and suitable polyamines.

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

A-4. ANTIOXIDANTS

A-4.1 Where it is considered that an antioxidant be employed, the following materials are recommended to suit the requirements prescribed in **2.1.2:**

a) Condensation products of acetone and aniline, and

b) Di-β naphthyl-*p*-phenylenediamine (symmetric).

A-5. FILLERS

A-5.1 The following are recommended.

A-5.1.1 *China Clay — see* IS : 505-1968*.

A-5.1.2 *Barytes — see* IS : 1683-1960†.

A-5.1.3 *Blanc fixe*

A-5.1.4 *Kieselguhr*

A-5.1.5 *Silica*

A-5.1.6 *Whiting — see* IS : 1685-1960‡.

A-5.1.7 *Carbon black*

A-6. SOFTENERS

A-6.1 The following are recommended.

A-6.1.1 *Stearic Acid — see* IS : 1675-1960§.

A-6.1.2 *Petroleum Jelly*

A-6.1.3 *Light Coloured Mineral Oil* — for example transformer oil (*see* IS : 335-1963‖).

APPENDIX B

(*Table* 1)

TEST METHODS FOR RUBBER SEALING RINGS

B-1. TEST FOR COLOUR, ODOUR AND TASTE OF WATER EXTRACTIVE

B-1.1 Procedure — Using clean forceps, transfer sufficient number of rings weighing about 20 g to a beaker. Autoclave them with 100 ml of

*Specification for light kaolin (*first revision*).

†Specification for barytes for rubber industry.

‡Specification for whiting for rubber industry.

§Specification for stearic acid, technical.

‖Specification for insulating oil for transformers and switchgear (*revised*).

water under a steam pressure of 1·05 to 1·40 kgf/cm² at a temperature of 120° to 125°C for 30 minutes. Cool and examine the extracted solution.

B-2. DETERMINATION OF FREE SULPHUR

B-2.1 Outline of the Method — A weighed test portion is extracted with acetone in an all-glass soxhlet type apparatus for 8 to 16 hours. When extraction is complete, the solvent is evaporated. Free sulphur is determined gravimetrically from the extract.

B-2.2 Apparatus

B-2.2.1 *Extraction Apparatus* — The extraction apparatus is of the reflux type with the condenser placed immediately above the cup which holds the rubber. The cup is situated in the vapours of the boiling solvent and is emptied by a siphon. The apparatus is of glass except in patterns where an extraction cup is suspended from the end of the condenser, in which case platinum wire is used for the suspension. The apparatus fits together without the use of cork, rubber or metal and in such a manner that loss of vapour during extraction does not exceed 20 percent of the extracting liquid.

B-2.3 Reagents

B-2.3.1 *Acetone* — conforming to IS : 170-1966*.

B-2.3.2 *Bromine*

B-2.3.3 *Barium Chloride Solution* — Dissolve 10 g of barium chloride in 100 ml of water.

B-2.3.4 *Hydrochloric Acid* — conforming to IS : 265-1962†.

B-2.4 Procedure — Using clean forceps, transfer sufficient number of rings to weigh about 10 g, to a round bottom flask. Add 100 ml of acetone to the flask and allow to stand overnight. Connect the flask to an efficient reflux condenser, boil and reflux for eight hours. Evaporate carefully to dryness on a steam bath and dry at 100° ± 2°C to constant weight. Add to the dried acetone extract 50 ml of water and 1 to 3 ml of bromine and cover with a watch glass. Allow the vessel to stand in a water bath at 70°C for at least 30 minutes, then remove the watch glass and heat continuously without boiling till the solution is almost colourless. Add 1 ml of hydrochloric acid, filter the solution and dilute it to 250 ml with water. Heat the solution to boiling, add slowly a slight excess of hot barium chloride solution, continue to boil the liquid for 5 to 10 minutes and then allow to stand for one hour at 90° to 100°C. Filter the liquid through a cintered glass or Gooch crucible which has been previously washed, dried

*Specification for acetone (*first revision*).

†Specification for hydrochloric acid (*revised*).

at 110°C and weighed. After the filtration has been completed, wash the crucible and the precipitate with hot water till the washings are free from chlorides, dry at 110° for one hour, cool in a desiccator and weigh.

B-2.4.1 Make a blank determination with the reagents using the same quantities and under the same conditions of test and apply the correction, if any, to the weight obtained in **B-2.4.**

B-2.5 Calculation

$$\text{Free sulphur content, percent by weight} = \frac{13{\cdot}73\,B}{W}$$

where

B = corrected weight in grams of the precipitate, and

W = weight in grams of the rubber rings taken for the test.

B-3. DETERMINATION OF TOTAL ZINC OXIDE

B-3.1 Apparatus

B-3.1.1 *Muffle Furnace* — suitable for operation between temperatures of 500° and 600°C with an accuracy of ±25°C.

B-3.2 Reagents

B-3.2.1 *Demineralized Water* — prepared either by distillation or by the use of ion-exchange materials.

NOTE — Experience has indicated that the purity of the water may influence the stability of the blue colour obtained in the final titrations. As such special care shall be taken to ensure freedom from traces of iron and other metals. Instability of colour or failure to obtain a clear blue colour when titrating the blank may be an indication that further purification of the water used is nceessary.

B-3.2.2 *Hydrochloric Acid* — concentrated, conforming to IS : 265-1962*.

B-3.2.3 *Hydrogen Sulphide*

B-3.2.4 *Nitric Acid* — concentrated, conforming to IS : 264-1968†.

B-3.2.5 *Ammonium Chloride* — solid.

B-3.2.6 *Ammonium Hydroxide* — 20 percent (*w*/*w*).

B-3.2.7 *Ammonium Nitrate* — 2 percent aqueous solution.

B-3.2.8 *Ammonium Chloride Buffer Solution* — Dissolve 67·5 g of ammonium chloride in water, add 600 ml of 0·88 N ammonia solution and dilute to

*Specification for hydrochloric acid (*revised*).

†Specification for nitric acid (*first revision*).

950 ml with water. Add to this solution 0·616 g of magnesium sulphate ($MgSO_4.7H_2O$) and 0·93 g of solid EDTA dissolved in 50 ml of water.

B-3.2.9 *Indicator Solution* — Dissolve by warming 0·5 g of eriochrome black T* in 100 ml of methylated spirit and add 4·5 g of hydroxylamine hydrochloride. Allow to stand over night and centrifuge or filter. This indicator solution should not be kept for more than one month.

B-3.2.10 *EDTA Solution — 0·01 M* — Dissolve 3·72 g of disodium dihydrogen ethylene diamine tetra-acetate dihydrate in water and make up to 1 litre. Standardize this solution as prescribed below.

B-3.2.10.1 Prepare a standard zinc solution by dissolving 0·814 g of ignited zinc oxide in the minimum quantity of hydrochloric acid (1 volume concentrated acid + 1 volume water) and making up to 1 litre with water. Pipette 10 ml of this standard zinc solution into a 250-ml conical flask. Add 100 ml of water, 5 ml of ammonium chloride buffer solution and 6 to 8 drops of indicator solution. Titrate with EDTA solution until the colour changes from wine-red to clear blue. Carry out a blank determination on the reagents omitting the standard zinc solution, and from the corrected titration calculate the weight of zinc oxide equivalent to 1 ml of the EDTA solution.

B-3.2.11 *Potassium Cyanide* — 5 percent aqueous solution.

B-3.3 Procedure — Weigh to an accuracy of 0·01 g about 1 g of the sample into a previously ignited and weighed crucible and place in the hole in the asbestos board. Heat gently over a small bunsen flame so that the rubber does not ignite and no spurting occurs. When the rubber is completely decomposed to a charred mass, transfer the crucible to the muffle furnace at a temperature of 550° ± 25°C. Continue the heating until the ash is free from specks of carbon, cool in a desiccator and weigh. Repeat heating, cooling and weighing until the change in weight on further heating for 20 minutes does not exceed 1 mg. Add 3 ml of hydrochloric acid to the ash and evaporate to dryness on a boiling water bath. Carry out, simultaneously, a blank determination in a second crucible using the same reagents and giving identical treatment to both test and blank determinations. Add a further 2 ml of concentrated hydrochloric acid, warm and wash the contents of the crucible into a 250-ml beaker. Dilute with water to about 50 ml, boil for 10 minutes and filter while still hot. Test a small portion of the filtrate with hydrogen sulphide. If a precipitate forms, pass the gas through the whole of the hot filtrate, filter off the precipitate, wash with water saturated with hydrogen sulphide and boil the filtrate and washings to expel dissolved gas. If there is no precipitate, boil a small test portion until it is free from hydrogen sulphide and return it to the bulk solution. Add 2 drops of nitric acid and boil the solution for a few minutes. To the boiling solution add 2 g of

*1-(1-hydroxy-2 naphthylazo)-6-nitro-2-naphthol-4 — sulphonic acid.

ammonium chloride and then ammonia solution until the solution is alkaline. Continue to boil for two or three minutes, then filter off any iron and aluminium hydroxides precipitated. If more than a trace of iron and aluminium is present, re-dissolve the precipitate in hydrochloric acid and re-precipitate by addition of ammonium chloride and ammonium hydroxide. Filter the hot solution through filter paper, wash with hot ammonium nitrate and add the filtrate and washings to those from the first precipitation. If necessary, boil the solution in order to reduce its volume to 80 to 85 ml, and then dilute to 100 ml in a volumetric flask. Pipette 10 ml of the test solution into a 250-ml conical flask. Add 100 ml of water, 5 ml of ammonium chloride buffer solution and 6 to 8 drops of indicator solution. Heat to about 60°C. Titrate with EDTA solution until the colour changes from wine-red to clear blue. Carry out also a titration on 10 ml of the blank solution. Denote the corrected titration by T_1. Pipette a further 10 ml of the test solution into a 250-ml conical flask. Add 100 ml of water, 5 ml of ammonium chloride buffer solution, 6 to 8 drops of indicator solution, and 10 ml of potassium cyanide solution; titrate with EDTA solution until the colour changes from wine-red to clear blue. Carry out also a titration on 10 ml of the blank solution. Denote the corrected titration by T_2.

B-3.4 Expression of Results — Calculate the zinc oxide content as follows:

$$\text{Zinc oxide (ZnO), percent by weight} = \frac{T_1 - T_2 \times 81{\cdot}4 \times M}{W}$$

where

T_1, T_2 = titration readings in millilitres as indicated in **B-3.3**,
M = molarity of the EDTA solution, and
W = weight in grams of the test portion.

B-4. DETERMINATION OF HEAVY METALS

B-4.0 Outline of the Method — Presence of heavy metals is tested by treating the material with hydrogen sulphide. Appearance of black colour, or turbidity or both indicates the presence of heavy metals.

B-4.1 Reagents

B-4.1.1 *Concentrated Hydrochloric Acid* — conforming to IS : 265-1962*.

B-4.1.2 *Ammonium Chloride* — solid.

B-4.1.3 *Dilute Hydrochloric Acid* — 1 : 1 (*see* IS : 265-1962*).

*Specification for hydrochloric acid (*revised*).

B-4.1.4 *Dilute Acetic Acid* — 1 N.

B-4.1.5 *Hydrogen Sulphide Solution* — freshly prepared saturated solution.

B-4.2 Procedure — Ash the material as described under **B-3.3.** Treat the ash with 3 drops of concentrated hydrochloric acid. Evaporate to dryness over a low flame and return to the muffle furnace for 20 to 30 minutes. A clean white ash shall result; otherwise, the hydrochloric acid treatment may be repeated. Dissolve the ash in 1 ml of dilute hydrochloric acid and wash with small quantity of water to an evaporating dish. Repeat washing to ensure complete transfer of the dissolved ash. Evaporate to dryness on a steam-bath and dissolve the residue in about 20 ml of water. Take 10 ml of this solution in a test tube, add 0·5 g of ammonium chloride and 1 ml of acetic acid. Add 5 ml of hydrogen sulphide solution to it.

B-4.3 The material shall pass the test if the solution does not develop any black colour or turbidity.

B-5. DETERMINATION OF *p*H OF AQUEOUS EXTRACT

B-5.1 From each lot, cut 2 rings to about 2 mm pieces. Autoclave the pieces for 5 minutes at a pressure of 0·4 to 0·5 kgf/cm² with 200 ml of water. Discard the first extract and repeat the process with another 500 ml of water for 40 minutes. Decant the extract, cool and determine the *p*H with a *p*H-meter equipped with glass electrodes.

B-6. TEST FOR TENSION SET

B-6.1 Measure internal diameter of the ring. Mount the ring evenly on a cylinder with diameter twice that of the internal diameter of the ring and keep it for 10 minutes at 27° ± 2°C. Remove the ring and allow to rest for 10 minutes at 27° ± 2°C. Then measure the internal diameter.

B-6.1.1 Immerse a ring in boiling water for a period of 4 h, remove and immediately put in cold water for at least 1 h but not longer than 2 h. Determine the tension set on the heat treated ring according to **B-6.1.**

B-7. DETERMINATION OF HARDNESS

B-7.1 Perform this test on a press cured rubber slab of the same compound and cured under the same conditions as for the rings. The minimum dimensions of the slab shall be 7·5 × 7·5 × 6·5 mm. Determine the degree of hardness in accordance with the method prescribed in IS : 3400 (Part II)-1965*.

B-7.2 The test pieces tested for hardness according to **B-7.1** shall be aged for a period of 168 h at 70° ± 1°C in an air-oven according to the method prescribed in IS : 3400 (Part IV)-1965†. After ageing they shall be tested for hardness in accordance with the method prescribed in IS : 3400 (Part II)-1965*.

*Methods of test for vulcanized rubbers: Part II Hardness.

†Methods of test for vulcanized rubbers: Part IV Accelerated ageing.

APPENDIX C

(*Clause* 4.1)

SCALE OF SAMPLING AND CRITERIA FOR CONFORMITY

C-1. SCALE OF SAMPLING

C-1.1 Lot — In a consignment, all the sealing rings of the same type, dimension, design and manufactured from the same type of rubber and belonging to the same batch of production shall constitute a lot.

C-1.2 Samples shall be selected from each lot separately for ascertaining its conformity or otherwise to the requirements of this specification.

C-2. NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

C-2.1 The number of sealing rings to be selected at random from a lot for different tests shall depend upon the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

TABLE 2 SCALE OF SAMPLING AND PERMISSIBLE NUMBER OF DEFECTIVES

No. of Sealing Rings in the Lot	Dimensions, Workmanship and Finish		No. of Tests Each for Tension Set and Hardness [Items (vi) to (ix) of Table 1]
	Sample Size	Permissible No. of Defectives	
(1)	(2)	(3)	(4)
Up to 100	5	0	3
101 „ 150	8	0	
151 „ 300	13	0	
301 „ 500	20	0	5
501 „ 1 000	32	1	
1 001 and above	50	2	8

C-2.1.1 The rings to be selected from the lot shall be chosen at random. For this purpose at least 10 percent of the packages shall be opened and required number of rings shall be selected by taking approximately equal number at random from each of the package.

C-2.2 All the sealing rings selected according to **C-2.1** shall be examined for workmanship, finish and dimensions. Any ring failing in one or more of these characteristics shall be considered as defective. If the number of defectives found in the sample is less than or equal to the corresponding permissible number given in column 3 of Table 2, the lot shall be declared as conforming to these requirements, otherwise not.

C-2.2.1 In the case of those lots which have been found unsatisfactory according to **C-2.2**, all the sealing rings may, depending upon the agreement between the purchaser and the supplier, be inspected for these characteristics and the defective ones be removed.

C-2.3 The lot having been found satisfactory for workmanship, finish and dimensions shall then be tested for tension set before and after heat treatment and for hardness before and after ageing. The number of independent tests to be conducted for each of the characteristics, hardness and tension, is given in column 4 of Table 2. For this purpose required number of rings shall be selected at random from those already chosen under **C-2.1**. The lot shall be declared satisfactory with respect to these characteristics if none of the tests yield unsatisfactory results.

C-2.4 The lot which is found satisfactory under **C-2.2** and **C-2.3** shall be examined for chemical characteristics (items i to v of Table 1). For this purpose, one test shall be conducted for each of the above characteristics. For this, sufficient number of rings shall be chosen from those already selected. A lot shall be deemed to be conforming to the requirements for chemical characteristics, and hence to the requirements of this specification, if all the test results meet the corresponding requirements given in col 3 of Table 1.