(Reaffirmed 2010)

IS : 879 - 1981

Indian Standard

SPECIFICATION FOR SODIUM NITRITE

(*First Revision*)

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

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TO

IS:879-1981 SPECIFICATION FOR SODIUM NITRITE

(First Revision)

Alterations.

[*Page 16, Table 2, col* (1), *last entry*]
Substitute '1 001 and above' *for* '1 000 and above

(Page 17, clause B-5.1, line 2) - Substitute '(R)' *for* '$\bar{R}$'.

[*Page 18, Table 3, col* (3)]:

a) *First entry* - Substitute '$\bar{X}_1$' *for* 'X_1

b) *Second entry* - Substitute '$\bar{X}_2$' *for* 'X_2'.

(Page 18, Table 3, col 5):

a) *Line 1* - Substitute "$(\bar{X}_1 - 0.6R_1)$ *for* '$(\bar{X} - 0.6R_1)$'.

b) *Line 6* - Substitute '$\bar{X}_2 + 0.6R_2$' *for* '$(\bar{X} - 0.6R_2)$'.

(CDC 3)

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Indian Standard
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Indian Standard

SPECIFICATION FOR SODIUM NITRITE

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 5 June 1981, after the draft finalized by the Inorganic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 Sodium nitrite is used in the manufacture of dyes and drugs and their intermediates, rubber blowing agents, heat transfer salts and as an analytical reagent. It is also used in electrolytic baths for the removal of excess brass from brazed bicycle frames and in explosives.

0.3 This standard was originally published in 1956 but was withdrawn as there was no indigenous production of the material. The material is now being produced in India, and hence this standard has been compiled.

0.4 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 the methods of sampling and test for sodium nitrite.

2. GRADES

2.1 The material shall be of the following two grades:

a) Technical, and

b) Analytical Reagent (AR).

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

3. REQUIREMENTS

3.1 Description

3.1.1 Sodium nitrite of technical grade shall be in the form of clear deliquescent crystals, lumps or sticks of pale yellow colour and free from foreign matter.

3.1.2 Sodium nitrite of analytical reagent grade shall be in the form of white or pale yellow deliquescent crystals.

3.2 The material, when tested according to methods prescribed in Appendix A, shall also comply with the requirements given in Table 1. Reference to relevant clauses of Appendix A is given in col 5 of the table.

TABLE 1 REQUIREMENTS FOR SODIUM NITRITE

SL NO.	CHARACTERISTIC	REQUIREMENT		METHODS OF TEST, REF TO CL No. IN APPENDIX A
		Technical Grade (on Dry Basis)	AR Grade (on Dry Basis)	
(1)	(2)	(3)	(4)	(5)
i)	Moisture and volatile matter, percent by mass, *Max*	2.0	1.0	A-2
ii)	Assay (as $NaNO_2$), percent by mass, *Min*	97.0	99.0	A-3
iii)	Matter insoluble in water, percent by mass, *Max*	0.01	0.005	A-4
iv)	Alkalinity (as Na_2CO_3), percent by mass, *Max*	0.2	0.15	A-5
v)	Chloride (as Cl), percent by mass, *Max*	*	0.002	A-6
vi)	Sulphate (as SO_2), percent by mass, *Max*	0.08	0.005	A-7

*For the following end uses, this limit, as tested by the method given in Appendix A-6.4, shall be as given below:

a) Heat transfer salt — 0.1
b) Explosives — 0.5

(*Continued*)

TABLE 1 REQUIREMENTS FOR SODIUM NITRITE - *Contd*

SL NO.	CHARACTERISTIC	REQUIREMENT		METHODS OF TEST, REF TO CL NO. IN APPENDIX A
		Technical Grade (on Dry Basis)	AR Grade (on Dry Basis)	
(1)	(2)	(3)	(4)	(5)
vii)	Heavy metals (as Pb), percent by mass, *Max*	0.005	0 001	A-8
viii)	Iron (as F , percent by mass, *Max*	0 002	0.001	A-9
ix)	Arsenic (as As), percent by mass, *Max*	—	0.000 04	A-10
x)	Calcium and magnesium (as Ca), percent by mass, *Max*	—	0.002 5	A-11
xi)	Potassium (as K), percent by mass, *Max*	—	0.005	A-12
xii)	Sodium nitrate content (as $NaNO_3$), percent by mass, *Max*	0.4	—	A-13

4. PACKING AND MARKING

4.1 Packing — The material of technical grade shall be packed in high density polyethylene bags or as agreed to between the purchaser and the supplier.

4.1.1 The material of analytical reagent grade shall be packed in amber coloured airtight glass bottles.

4.2 Marking — The containers shall be marked with the following information:

a) Name and grade of the material and specific end use;

b) Name of the manufacturer and his recognized trade-mark, if any;

c) Gross and net mass;

d) Date of manufacture; and

e) Batch number.

4.2.1 In the case of analytical reagent grade, the chemical analysis of the material in respect of characteristics specified in Table 1 shall also appear on the label.

4.2.2 The containers 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 products 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.

5. SAMPLING

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

APPENDIX A

(*Clause* 3.2)

METHODS OF TEST FOR SODIUM NITRITE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be ernployed in tests.

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

A-2. MOISTURE AND VOLATILE MATTER

A-2.1 Procedure — Weigh accurately, by difference, about 50 g of the material in a petri dish from a stoppered weighing bottle and dry to constant mass at 110 ± 2°C in an oven. Cool the dried material in a desiccator containing sulphuric acid/activated silica gel.

NOTE — Use this dried material for the subsequent tests.

*Specification for water for general laboratory use (*second revision*).

A-2.2 Calculation

$$\text{Moisture and volatile matter, percent by mass} = \frac{M_1 \times 100}{M}$$

where

M = mass in g of the material taken for the test, and

M_1 = loss in mass in g of the material after drying to constant mass.

A-3. ASSAY

A-3.1 Reagents

A-3.1.1 *Sulphuric Acid* — 1 : 1 (*v/v*) (*see* IS : 266-1977*).

A-3.1.2 *Standard Potassium Permanganate Solution* — approximately 0.1 *N*.

A-3.2 Procedure — Dissolve about 2 g of the dried material (*see* **A-2.1**) accurately weighed in water and dilute it to exactly 500 ml in a volumetric flask. Take 80 ml of water in a 250-ml beaker and add 10 ml of sulphuric acid. Add from a pipette exactly 25 ml of standard potassium permanganate solution and mix thoroughly. Warm the mixture to 40°C and maintain this temperature. Titrate against solution of sodium nitrite filled in 50 ml capacity burette, keeping the tip of the burette under the surface of the permanganate solution and stirring continuously. As the end point approaches, the addition of sodium nitrite solution should be very slow since the reaction becomes sluggish. The end point will be the discharge of pink colour.

A-3.3 Calculation.

$$\text{Sodium nitrite (as } NaNO_2\text{)} = 34{\cdot}5 \times \frac{1\,250 \times N}{V \times M}$$

where

V = volume in ml of sodium nitrite solution required to discharge the pink colour of permanganate solution,

N = normality of standard potassium permanganate solution, and

M = mass in g of the dried material taken for the test (*see* **A-2.1**).

*Specification for sulphuric acid (*second revision*).

A-4. MATTER INSOLUBLE IN WATER

A-4.1 Procedure — Dissolve about 20 g of the dried material (*see* **A-2.1**) accurately weighed in 150 ml of water in a 500-ml beaker and heat on a steam bath for 1 hour. Filter through a tared Gooch crucible or a tared sintered glass crucible (G No. 4) previously washed and dried at 105 to 110°C. Wash the insoluble residue with water. Dry at 105 to 110°C. Repeat the drying till constant mass is obtained.

A-4.2 Calculation — Determine the mass of the insoluble residue and express the result as percentage of the mass of the material taken for the test.

A-5. ALKALINITY

A-5.1 Reagents

A-5.1.1 *Methyl Red Indicator* — Dissolve 0.1 g of methyl red in 100 ml of rectified spirit (*see* IS : 323-1959*).

A-5.1.2 *Standard Hydrochloric Acid* — approximately 0.1 *N*.

A-5.2 Procedure — Dissolve 5 g of the dried material (*see* **A-2.1**) accurately weighed in 25 ml of water. Add two drops of methyl red indicator and titrate with standard hydrochloric acid.

A-5.3 Calculation

$$\text{Alknlinity (as } Na_2CO_3\text{), percent by mass} = \frac{5{\cdot}3 \times V \times N}{M}$$

where

V = volume in ml of standard hydrochloric acid used for the test,

N = normality of standard hydrochloric acid, and

M = mass in g of the dried material taken for the test.

A-6. CHLORIDE

A-6.1 Apparatus

A-6.1.1 *Nessler Cylinders* — *See* IS : 4161-1967†.

*Specification for rectified spirit (*revised*).

†Specification for Nessler cylinders.

A-6.2 Reagents

A-6.2.1 *Glacial Acetic Acid — See* IS : 695-1975*.

A-6.2.2 *Concentrated Nitric Acid — See* IS : 264-1976†.

A-6.2.3 *Silver Nitrate Solution* — 0.1 *N.*

A-6.2.4 *Standard Chloride Solution* — One millilitre of this solution contains 0.01 mg of chloride.

A-6.3 Procedure for AR Grade

A-6.3.1 Weigh accurately 1.0 g of the dried material *(see* **A-2.1**). Dissolve it in 10 ml of chloride-free water in a 100-ml beaker. Add 1.0 ml of glacial acetic acid. Warm it for 15 minutes over water-bath to decompose the nitrous acid produced. Cool to room temperature. Transfer solution to 50 mi Nessler cylinder. Add 1.0 ml of concentrated nitric acid and 1.0 ml of silver nitrate solution. Dilute to the mark.

A-6.3.2 In another Nessler cylinder, take 10 ml of chloride-free water. Add 1.0 ml of glacial acetic acid and 1.0 ml of concentrated nitric acid. Add 2.0 ml of standard chloride solution (1 ml of which contains 0.01 mg of chloride). Add 1.0 ml of silver nitrate solution and dilute to the mark.

A-6.3.3 The sample shall be considered to pass the test if the turbidity produced in a test solution is not greater than that produced in the control test.

A-6.4 Procedure for Technical Grade

A-6.4.1 *Reagents*

A-6.4.1.1 *Standard silver nitrate solution* — approximately 0.1 *N.*

A-6.4.1.2 *Potassium chromate solution* — 5 percent (*m/v*).

A-6.4.1.3 *Nitric acid* — 70 percent (*m/m*).

A-6.4.1.4 *AR grade calcium carbonate*

A-6.5 Procedure — Dissolve 5 g of the material as prescribed under **A-2.1** and dissolved in 50 ml of chloride-free water. Add 5 ml nitric acid and heat the mixture gently to decompose the nitrous acid produce. Cool to room temperature. Neutralize acid by adding solid

*Specification for acetic acid (*second revision*).

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

calcium carbonate pinch by pinch. Add a little excess and add 1 ml of potassium chromate from the burette. Titrate against 0.1 *N* silver nitrate till the colour of the precipitate changes to buff or pink.

A-6.6 Calculation

$$\text{Chloride as (Cl), percent by mass} = \frac{3{\cdot}55 \times V \times N}{M}$$

where

V = volume in ml of standard silver nitrate used for the test,

N = normality of standard silver nitrate solution, and

M = mass in g of the dried material taken for the test.

A-7. SULPHATE

A-7.1 Apparatus

A-7.1.1 *Nessler Cylinders* — *See* IS : 4161-1967*.

A-7.2 Reagents

A-7.2.1 *Hydrochloric Acid* — 1 : 1 (*v/v*).

A-7.2.2 *Barium Chloride Solution* — Dissolve 12.5 g of barium chloride ($BaCl_2.2H_2O$) in 100 ml of water and filter through Whatman filter paper No. 40 or 42.

A-7.2.3 *Standard Sulphate Solution* — Dissolve 1.376 4 g of ammonium sulphate in water and dilute to 1 000 ml in a volumetric flask. Transfer exactly 1 ml of this solution to a volumetric flask and again dilute to 100 ml with water. One millilitre of this solution is equivalent to 0.01 mg of sulphate.

A-7.3 Procedure for Technical Grade — Weigh 1 g of the dried material (*see* **A-2.1**) and dissolve in 10 ml of water in 100-ml beaker. Add 3 ml of concentrated hydrochloric acid and evaporate to dryness on water-bath. Dissolve the residue with 10 ml of water and dilute the solution to 100 ml in volumetric flask. Pipette out 10 ml of solution in Nessler cylinder. Add 1 ml of hydrochloric acid and make up the volume to 50 ml. Take 10 ml of water in the other Nessler cylinder and add 8 ml of standard sulphate solution. Add 1 ml of hydrochloric acid. Dilute with water to 50 ml.

*Specification for Nessler cylinders.

A-7.3.1 Add 1 ml of barium chloride solution each to both the solutions and allow to stand for 1 hour. The limit prescribed shall be taken as not having been exceeded if the turbidity produced in the first tube is not greater than that produced in the second tube.

A-7.4 Procedure for AR Grade

A-7.4.1 Weigh 1 g of the dried material (*see* **A-2.1**) and dissolve in 10 ml of water in a 100 ml beaker. Add 3 ml concentrated hydrochloric acid and evaporate to dryness on the water bath. After dissolving the residue with 10 ml of water, transfer the entire solution to the Nessler cylinder. Add 1 ml of hydrochloric acid and make up the volume to 50 ml. Take 10 ml of water in the other Nessler cylinder and add 5 ml of standard sulphate solution. Add 1 ml of hydrochloric acid. Dilute with water to 50 ml.

A-7.4.2 Add 1 ml of barium chloride solution each to both the solutions and allow to stand for 1 hour. The limit prescribed shall be taken as not having been exceeded if the turbidity produced in the first tube is not greater than that produced in the second tube.

A-8. HEAVY METALS

A-8.1 Apparatus

A-8.1.1 *Nessler Cylinders — See* IS : 4161-1967*.

A-8.2 Reagents

A-8.2.1 *Concentrated Hydrochloric Acid — See* IS : 265-1976†.

A-8.2.2 *Standard Lead Solution* — Dissolve 1.60 g of lead nitrate in water, and 1 ml of concentrated nitric acid and make the volume up to 1 000 ml. Transfer exactly 10 ml of this solution to a volumetric flask, again dilute with water and make up the volume to 1 000 ml mark. One millilitre of this solution is equivalent to 0.01 mg of lead (Pb).

A-8.2.3 *Acetic Acid* — approximately 1 *N*.

A-8.2.4 *Hydrogen Sulphide Solution* — Prepare a fresh, saturated aqueous solution of hydrogen sulphide gas.

A-8.3 Procedure for Technical Grade — Weigh 10.00 g of the dried material (*see* **A-2.1**) and dissolve in 50 ml of water. Add 25 ml of concentrated hydrochloric acid and evaporate to dryness on the water

*Specification for Nessler cylinders.
†Specification for hydrochloric acid (*second revision*).

bath. Dissolve the residue with water and make up the solution to exactly 500 ml (solution A). Transfer 10 ml of this solution (Solution A) into a Nessler cylinder. Dilute to 30 ml and add 1 ml of acetic acid. Take 30 ml of distilled water in another cylinders. Add the other Nessler cylinder and add 1 ml of acetic acid. Add 10 ml of hydrogen sulphide solution to each of the cylinders, make up the volume to 50 ml and compare the colour.

Take 30 ml of distilled water in another cylinder. Add 1 ml of standard lead solution and add 1 ml acetic acid.

A-8.3.1 The limit prescribed shall be taken as not having been exceeded if the intensity of colour produced in the first cylinder is not greater than that produced in the second cylinder.

A-8.4 Procedure for AR Grade

A-8.4.1 Weigh 10.00 g of the dried material (*see* **A-2.1**) and dissolve in 50 ml of water, add 25 ml of concentrated hydrochloric acid and evaporate to dryness on the water bath. Dissolve the residue with water and make up the solution to exactly 100 ml (solution A). Transfer 10 ml of this solution (solution A) into a Nessler cylinder. Dilute to 30 ml and add 1 ml of acetic acid. Take 30 ml of distilled in the other Nessler cylinder and add 1 ml of standard lead solution and add 1 ml of acetic acid. Add 10 ml of hydrogen sulphide solution to each of the cylinders make up the volume to 50 ml and compare the colour.

A-8.4.2 The limit prescribed shall be taken as not having been exceeded if the intensity of colour produced in the first tube is not greater than that produced in the second tube.

A-9. IRON

A-9.1 Apparatus

A-9.1.1 *Nessler Cylinder — See* IS : 4161-1967*.

A-9.2 Reagents

A-9.2.1 *Concentrated Hydrochloric Acid— See* IS : 265-1976†.

*Specification for Nessler cylinders.

†Specification for hydrochloric acid (*second revision*).

A-9.2.2 *Butanolic Potassium Thiocyanate Solution* — Dissolve 10 g of potassium thiocyanate in 10 ml of water at 27 ± 2°C. Add sufficient *n*-butanol to make up to 100 ml, and shake vigorously until the solution is clear.

A-9.2.3 *Standard Iron Solution* — Dissolve 0.702 2 g of ferrous ammonium sulphate in 10 ml of dilute sulphuric acid (10 percent by volume). From the burette add 0.1 *N* potassium permanganate solution till pink colour persists. Dilute it to 1 000 ml in a volumetric flask. Take 10 ml of this solution and dilute to 100 ml. One millilitre of the final solution is equivalent to 0.01 mg of iron (Fe^{2+}).

A-9.2.4 *Ammonium Persulphate* — solid.

A-9.3 Procedure — Dissolve 10.0 g of the dried material (*see* **A-2.1**) in 50 ml of water, add 25 ml of concentrated hydrochloric acid and evaporate to dryness on the water bath. Dissolve the residue in water and make up to 100 ml in a volumetric flask. For technical grade, take 10 ml and for AR grade take 20 ml of the made up solution. Add 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Shake vigorously for 30 seconds and allow to separate. Carry out a control test in the other Nessler cylinder using 2 ml of standard iron solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour produced in the tubes after 5 minutes.

A-9.3.1 The limit prescribed shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

A-10. ARSENIC

A-10.1 Dissolve 5 g of the material in 25 ml of dilute sulphuric acid and evaporate until copious white fumes are evolved. Allow to cool, add 50 ml of water and 5 ml of standard hydrochloric acid and test as prescribed in **5.1** of IS : 2088-1971*.

A-11. CALCIUM AND MAGNESIUM

A-11.1 Reagents

A-11.1.1 *Ammonia Ammonium Chloride Buffer Solution* — Dissolve 67.5 g of ammonium chloride in 300 ml of water, add 570 ml of ammonium hydroxide solution (18 *N*) and make up to 1 litre with water.

A-11.1.2 *Ammonium Hydroxide Solution* — 18 *N*.

*Methods for determination of arsenic (*first revision*).

A-11.1.3 *Sodium Sulphide Solution*

A-11.1.4 *EDTA Solution* – 0.01 *M.*

A-11.1.5 *Methyl Thymol Blue Indicator Solution* — 0.1 percent solution in 50 ml of rectified spirit.

A-11.2 Procedure

A-11.2.1 Dissolve 13 g of the material in 100 ml of water, add 20 ml of ammonium chloride solution, 25 ml of ammonium hydroxide solution and 5 drops of sodium sulphide solution. Titrate with EDTA solution using methyl thymol blue as indicator until the blue solution becomes colourless or grey. No more than 0.8 ml of EDTA solution is required.

A-12. POTASSIUM

A-12.1 Determine the potassium by flame photometer according to the instructions of the manufacturer.

A-13. SODIUM NITRATE

A-13.1 Reagents

A-13.1.1 *Sulphric Acid* — 0.2 *N.*

A-13.1.2 *Sodium Hydroxide Solution*— 0.2 *N.*

A-13.1.3 *Sodium Hydroxide Solution* — 42 percent (*m/v*).

A-13.1.4 *Devardas Alloy*

A-13.1.5 *Methyl Red Indicator Solution*

A-13.2 Procedure — Weigh accurately about 0.3 g of the material and transfer it into a Kjeldahl flask. Dilute it to about 250 ml with water. Mount the Kjeldahl flask heater and connect it through a condenser to a beaker or conical flask (600-ml capacity containing 25 ml of sulphuric acid and 2 drops of methyl red indicator). Now add 2 to 3 g of devardas alloy to the flask and tighten all connections. Add 10 ml of sodium hydroxide (42 percent) solution slowly through dropping funnel or by the sides of the flask. Allow the reaction to proceed in cold for 15 minutes. Heat the flask slowly, gradually increasing the temperature. Collect 250 to 300 ml of distillate. Remove the beaker and switch off the heater. Titrate excess sulphuric acid in the beaker against sodium hydroxide (0.2 *N)* using metnyl red indicator to a neutral end point.

A-13.3 Calculation

A-13.3.1 Total nitrogen, percent by mass $= \frac{(V \times N - V_1 N_1) \times 1{\cdot}408}{M}$

where

V = volume in ml of sulphuric acid used,
N = normality of sulphuric acid solution,
V_1 = volume in ml of sodium hydroxide solution used,
N_1 = normality of sodium hydroxide solution, and
M = mass in g of the material taken for the test.

A-13.3.2 Sodium nitrate, percent by mass = 6.068 (A — B)

where

A = total nitrogen, percent by mass (*see* **A-13.3.1**), and
B = nitrogen due to sodium nitrite percent by mass (*see* **A-13.3.2**).

APPENDIX B

(*Clause* 5.1)

SAMPLING OF SODIUM NITRITE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, storing, preparing and handling test samples, the following precautions shall be observed.

B-1.1 Samples shall not be taken at a place exposed to weather.

B-1.2 Precautions shall be taken to protect the samples, the sampling instrument and the containers for samples from adventitious contamination.

B-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

B-1.4 The samples shall be placed in suitable, clean, dry and airtight glass containers.

B-1.5 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and year of manufacture.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of sodium nitrite of the same grade and drawn from a single batch of manufacture shall constitute a lot. If the consignment is declared to consist of different batches, the batches shall be marked separately and the group of containers in each such group constitute separate lots.

B-2.2 Samples shall be tested for each lot separately, for ascertaining conformity of the material to the requirements of this specification.

B-2.3 The number of containers *(n)* to be selected from the lot shall depend upon the size of the lot (*N*) and shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

LOT SIZE (*N*)	No. OF CONTAINERS TO BE SELECTED (*n*)
(1)	(2)
Up to 15	2
16 to 25	3
26 to 50	4
51 to 100	5
101 to 150	6
151 to 300	7
301 to 500	8
501 to 1 000	9
1 000 and above	10

B-2.3.1 These containers shall be selected at random from the lot and in order to ensure the randomness of selection, procedures given in IS : 4905-1968* may by followed.

B-3. PREPARATION OF TEST SAMPLES

B-3.1 From each of the containers selected according to **B-2.3.1**, a representative portion of the material, about 200 g, shall be drawn with the help of a suitable sampling instrument.

*Methods for random sampling.

B-3.1.1 Out of these portions, equal quantities of the material shall be taken and mixed thoroughly to form a composite sample of about 500 g. The composite samples shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.1.2 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the containers sampled shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.2 All the individual samples and the composite samples shall be transferred to separate sample containers. All the containers shall be sealed and labelled with full identification particulars.

B-3.3 The referee test sample consisting of a composite sample and a set of individual samples shall bear the seals of both the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of a dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for determination of assay content, moisture and volatile matter content shall be performed on each of the individual samples.

B-4.2 Tests for the determination of the remaining characteristics given in **3** of the specification shall be performed on the composite sample (*see* **B-3.1.1**).

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples — From the test results, the average ($\overline{X}$) and the range ($\overline{R}$) shall be computed for each of the characteristics tested on individual samples (the range being defined as the difference between the maximum and minimum values of the test results). The Appropriate expression as shown in col 5 of Table 3 shall be calculated for these characteristics. If the values of the expressions satisfy the conditions as given in col 5 of Table 3, the lot shall be declared to have satisfied the requirements for these characteristics.

B-5.2 For Composite Sample — The lot shall be considered to have passed in respect of the characteristics tested the composite test sample, if the test results satisfy the corresponding requirements given in Table 1.

B-5.3 The lot shall be considered as conforming to the requirements of specification if it satisfies all the criteria given in **B-5.1** and **B-5.2.**

TABLE 3 CRITERIA FOR CONFORMITY BASED ON INDIVIDUAL SAMPLES

(*Clause* B-5.1)

SL No.	CHARACTERISTIC	AVERAGE OF TEST RESULTS 1, 2, 3 *(n)*	RANGE	CRITERIA FOR CONFORMITY
(1)	(2)	(3)	(4)	(5)
i)	Assay content	X_1	R_1	($\bar{X}\theta.6R_1$) shall be greater than or equal to the minimum specified value given in Table 1.
ii)	Moisture and volatile matter content	X_2	R_2	($\bar{X}\theta.6R_2$) shall be less than or equal to the maximum value specified in Table 1.

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