Doc: FAD 08 (23901) WC
October 2023

BUREAU OF INDIAN STANDARDS

DRAFT FOR COMMENTS ONLY

भारतीय मानक मसौदा

आयोडीन युक्त नमक, वैक्यूम वाष्पीकृत आयोडीन युक्त नमक और परिष्कृत आयोडीन युक्त नमक – विशिष्टि

(*आइ एस 7224 का तीसरा पुनरीक्षण*)

Draft Indian Standard

IODIZED SALT, VACUUM EVAPORATED IODIZED SALT AND REFINED IODIZED SALT – SPECIFICATION

(*Third Revision of IS 7224*)

ICS No. 67.220.20

Food Additives Sectional Committee, FAD 08 Last Date of Comments: **29 December 2023**

FOREWORD

(*Formal clauses would be added later*)

This standard was first published in 1973. The standard was first revised in 1985, to cover iodized salt with potassium iodate or any other iodate used for iodizing the salt. The limit for iodine content was revised to a minimum of 20 ppm and a new method for determination of iodine was incorporated. In the second revision issued in 2006, the specifications for refined iodized salt and vacuum evaporated iodized salt were incorporated. The market for refined salt has come into existence with the growing need for pure and non-caking salt by the consumers. Refining process involves a series of physical and chemical operations to remove the impurities (soluble and insoluble). For upgrading the quality of salt, that is sodium chloride content, it requires heavy investment in plant and machinery and for improving technology. Hence, it needs to be distinguished from ordinary (unrefined) iodized salt, for enabling consumer to take informed decision. The second revision was considered necessary because in the absence of any standard on refined iodized salt in the country, many iodized salt manufacturers are marketing powdered and dried iodized salt in attractive polypacks in guise of refined iodized salt, thus duping the general public.

In this revision, the following major changes have been made:

a) The limit of iodine is aligned with *Food Safety and Standards (Fortification of Food) Regulation*, 2018.
b) The marking clause has been updated.
c) The three amendments issued to the previous version of this standard have been incorporated.

In the preparation of this standard, due consideration has been given to the *Legal Metrology (Packaged Commodities) Rules*, 2011 and *Food Safety and Standards Act*, 2006 and Rules and Regulations framed thereunder.

The standard is however subject to restrictions imposed under these rules wherever applicable. 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.

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for iodized salt, vacuum evaporated iodized salt and refined iodized salt.

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions 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*
IS 253 : 2014	Specification for common salt (*fourth revision*)
IS 264 : 2005	Nitric acid - Specification (*third revision*)
IS 323 : 2009	Rectified spirit for industrial use – Specification (*second revision*)
IS 1070 : 2023	Reagent grade water – Specification (*fourth revision*)
IS 2088 : 2023	Methods for determination of arsenic (*third revision*)
IS 4905 : 2015	Random sampling and randomization procedures (*first revision*)

3 TYPES

Iodized salt shall be of the following three types:

a) Refined iodized salt,

b) Vacuum evaporated iodized salt, and

c) Iodized salt.

4 REQUIREMENTS

4.1 Description

4.1.1 *Refined Iodized Salt*

Refined iodized salt is solar/rock salt from which soluble and insoluble impurities are reduced by employing mechanical washing, centrifuging and iodizing, drying and sieving. It shall be free from visible contamination with clay, grit or other extraneous adulterants or impurities.

4.1.2 *Vacuum Evaporated Iodized Salt*

Vacuum evaporated iodized salt is made by evaporating brine employing forced evaporators operating under vacuum, centrifuging iodizing, drying and coating with permitted anticaking agents as permitted under the *Food Safety and Standards Act*, 2006 and Rules and Regulations framed thereunder. It shall be free from visible contamination with clay, grit or other extraneous adulterants or impurities.

4.1.3 *Iodized Salt*

Iodized salt is solar/rock salt mixed with iodizing chemical and dried if required. Material shall be a crystalline solid, white or pale pink and light grey in colour. It shall be free from visible contamination with clay, grit or other extraneous adulterants or impurities.

4.2 Particle Size

For iodized salt, minimum 95 percent by mass of the material shall pass through 4.00 mm IS sieve. For refined iodized salt, minimum 95 percent of the material shall pass through 1 mm IS sieve and not more than 10 percent by mass shall pass through 150 micron IS sieve.

4.3 The material shall also comply with the requirements given in Table 1 when dried in accordance with the method prescribed in Annex A.

4.4 The material may contain anticaking agents as permitted under the *Food Safety and Standards (Food Products Standards and Food Additives) Regulation*, 2011 subject to a maximum 2 percent by mass, either singly or in combination. Further, when calcium, potassium or sodium ferrocyanide are used as crystal modifiers and anticaking agents, their quantity should not exceed 10 mg/kg, on dry basis, either singly or in combination expressed as ferrocyanide when tested by the method prescribed in Annex B.

5 PACKING AND MARKING

5.1 Packing

5.1.1 The material shall be packed in jute bags having inside polyethylene liner or HDPE bags or in packages as agreed to between the purchaser and the supplier.

Table 1 Requirements for Refined Iodized Salt, Vacuum Evaporated Iodized Salt and Iodized Salt

(*Clause* 4.3)

Sl. No.	**Characteristic**	**Requirements**		**Method of Test, Ref to**
(1)	(2)	Refined Iodized Salt / Vacuum Evaporated Iodized Salt (3)	Iodized Salt (4)	(5)
i)	Moisture content, percent by mass, *Max*	0.5	6.0	Annex A
ii)	Water insoluble matter, percent by mass, on dry basis, *Max*	1.0	1.0	Annex C
iii)	Chloride content (as NaCl), percent by mass, on dry basis, *Min*	98.5	96.0	A-5 of IS 253
iv)	Matter soluble in water other than sodium chloride, percent by mass, on dry basis, *Max*	1.0	3.0	Annex D
v)	Calcium (as Ca), percent by mass, on dry basis, *Max*	0.15	-	Annex E
vi)	Magnesium (as Mg), percent by mass, on dry basis, *Max*	0.10	-	Annex E
vii)	Sulphate (as SO_4), percent by mass, *Max*	0.60	-	Annex F
viii)	Iodine content, ppm	15.0-30.0	15.0-30.0	Annex G

ix)	Alkalinity (as Na_2CO_3), percent by mass, *Max*	0.15	0.15	Annex H
x)	Lead (as Pb), ppm, *Max*	2.0	2.5	Annex J
xi)	Arsenic (as As), ppm, *Max*	1.0	1.0	Annex K
xii)	Iron (as Fe), ppm, *Max*	50.0	50.0	Annex L

For consumer packing, the material shall be packed in food grade plastics (see relevant Indian Standards). The thickness of the plastic film used for packing shall be not less than 50 microns.

5.2 Marking

5.2.1 The packages shall be legibly and clearly marked with the following particulars:

a) Name and type of product;
b) Name and address of the manufacture;
c) Batch or Code number;
d) Iodizing agent used;
e) Iodine content when packed;
f) Date of manufacturing ‘month and year’;
g) Expiry date ‘month and year’;
h) Net quantity;
j) Instructions for storage ‘Store in a cool and dry place’; and
k) Any other requirements as specified under the *Legal Metrology (Packaged Commodities) Rules*, 2011 and *Food Safety and Standards (Packaging and Labelling) Regulation*, 2011 and the Rules framed thereunder.

5.2.2 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.

6 SAMPLING

Representative samples of the material for testing its conformity to this standard shall be drawn according to the method prescribed in Annex M.

7 TESTS

7.1 Tests shall be carried out as prescribed in col (5) of Table 1.

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

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

ANNEX A

[*Clause* 4.3, and *Table* 1, *S1 No.* (i)]

DETERMINATION OF MOISTURE CONTENT

A-1 PROCEDURE

A-1.1 Grind rapidly the sample material as received (say 100 g), if required, in an agate mortar approximately to a size of 2.8 mm IS Sieve, but do not actually sieve. The material which shall be in the form of powder shall be kept in an air-tight container.

A-1.2 Weigh accurately about 20 g of the material in a weighing bottle (about 30 ml capacity), preferably wide mouth squat type, previously dried and weighed. Dry in an oven at 140 to 150°C for at least 4 h. Cool in a desiccator and weigh. Repeat drying, cooling and weighing until constant mass is obtained.

A-2 CALCULATION

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

where,

M_1 = mass in g of the material taken for the test; and

M_2 = mass in g of the material after drying in the oven.

ANNEX B

[*Clause* 4.4]

DETERMINATION OF FERROCYANIDE

B-1 GENERAL

This test is designed for 10 ppm of ferrocyanide [expressed as $K_4Fe(CN)_6$].

B-2 REAGENTS

B-2.1 Dilute Sulphuric Acid — About 0.5 N solution.

B-2.2 Ferrous/Ferric Solution — Dissolve 20 g of ammonium ferrous sulphate [$(NH_4)_2SO_4.FeSO_4.6H_2O$], and 2.5 g of ammonium ferric sulphate [$(NH_4)_2 SO_4.Fe_2(SO_4).24H_2O$], in water to which 10 ml of the dilute sulphuric acid has been added. Dilute to 100 ml with water, filter and store in a dark bottle.

B-1.3 Phosphate Solution — Dissolve 70 g of potassium dihydrogen phosphate in water, add 50 ml of the dilute sulphuric acid and make up to 1 000 ml with water.

B-1.4 Sodium Chloride — Before use, heat at 500°C for 2 h in a furnace, and allow to cool.

B-1.5 Standard Potassium Ferrocyanide Solution

B-1.5.1 Ferrocyanide Stock Solution — Dissolve 2.294 g of potassium ferrocyanide trihydrate, [K, Fe(CN)c.3H,0] in water, add 5 ml of dilute potassium hydroxide solution (about 0.1 N) and dilute to 1 000 ml with freshly boiled and cooled water. Store in the dark.

B-1.5.2 Ferrocyanide Working Solution — Take 25 ml of the ferrocyanide stock solution, add 5 ml of potassium hydroxide solution (about 0.1 N) and dilute to 1 000 ml with freshly boiled and cooled water.

1 ml = 0.05 mg $K_4Fe(CN)_4$

B-2 PROCEDURE

Dissolve 10 and 0.1 g of the salt (*see* **A-1.1**) in about 40 ml of water in a Nessler cylinder graduated at 100 ml. Add 10 ml of the dilute sulphuric acid and 5 ml of the ferrous/ferric solution, mixing well after each addition. Allow to stand for about 2 min, then add 35 ml of the phosphate solution, mix, dilute to the 100 ml mark and mix again. The colour shall not be greater than the 10 ppm $K_4Fe(CN)_6$ standard which is prepared similarly using 10 g of the sodium chloride reagent to which has been added 2 ml of the ferrocyanide working solution in place of the 10 g of sample.

ANNEX C

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF MATTER INSOLUBLE IN WATER

C-1 PREPARATION OF THE SAMPLE FOR CHEMICAL TESTS

Spread 80 to 100 g of the sample (*see* **A-1.1**) in a petri dish and dry by the method given in **A-1.2**. The dried material shall be called the tiled sample and shall be used in the tests where so indicated.

C-2 PROCEDURE

Accurately weigh about 20 g of the dried sample, dissolve it in 200 ml of water in a beaker and heat to boiling and cool. Filter the solution through a weighed Gooch or sintered glass crucible (G No. 4) and wash the residue till it is free from soluble salts. Collect the filtrate and washings in a one-litre graduated flask and dilute to mark. Preserve the solution so obtained for subsequent tests. Dry the crucible along with the insoluble residue to constant mass.

C-3 CALCULATION

$$\text{Matter insoluble in water, percent by mass} = \frac{M_1}{M_2} \times 100$$

where,

M_1 = mass in g of the residue; and

M_2 = mass in g of the dried sample taken for the test.

C-3.1 Insoluble matter in water, percent by mass, is calculated on moisture-free basis. However, in the case of refined iodized salt, insoluble matter in water, percent by mass, also includes the flee-flowing agents added to the salt.

ANNEX D

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF MATTER SOLUBLE IN WATER OTHER THAN SODIUM CHLORIDE

D-1 PROCEDURE

Express the soluble carbonate as calcium carbonate (marine salt), or as sodium carbonate (Rajasthan salt, in absence of calcium and magnesium). If there is excess of calcium over the calcium carbonate, combine all the calcium with the sulphate radical and express the result as percentage of calcium sulphate. If there is excess of the sulphate over what is required for calcium, combine all the calcium with the sulphate radical and express the result as percentage of calcium sulphate. If there is excess of the sulphate over what is required for calcium, combine the excess with magnesium and express the result as percentage of magnesium sulphate. Calculate the balance of magnesium as magnesium chloride and deduct the chloride corresponding to it from the chloride content. Also, deduct from the chloride content an amount of chloride corresponding to potassium content and express it as percentage of potassium chloride.

Calculate the residual chlorine as percentage of sodium chloride.

D-2 CALCULATION

D-2.1 In the above procedure, if there is excess of sulphate over the calcium and magnesium sulphate, express it as sodium sulphate, and if there is excess of calcium over the calcium sulphate, express it as calcium chloride. This situation does not arise commonly in the analysis of common salt.

D-2.2 In the absence of calcium and magnesium, express carbonate as sodium carbonate and sulphate as sodium sulphate.

D-2.3 Calculate the matter soluble in water other than sodium chloride by subtracting from the 100 the sum of sodium chloride content and matter insoluble in water. Matter soluble in water includes the water of hydration retained-at the temperature drying.

ANNEX E

[*Table* 1, *S1 No.* (v) and (vi)]

DETERMINATION OF CALCIUM AND MAGNESIUM

E-1 REAGENTS

E-1.1 Standard Calcium Solution — Weigh 1.0 g of calcium carbonate dried at 120°C and dissolve it in the minimum quantity of dilute hydrochloric acid. Dilute the solution to one litre in a graduated flask.

One millilitre of the solution is equivalent to 0.4008 mg of calcium (as Ca).

E-1.2 Standard EDTA Solution — Dissolve 3.72 g of disodium ethylene diamine tetra acetate dehydrate in water and dilute in a graduated flask to 1 litre. The solution shall be standardized frequently against standard calcium solution following the procedure given in **E-2**.

E-1.3 Eriochrome Black T Indicator Solution — Dissolve 0.1 g of the dye in 20ml of rectified spirit

E-1.4 Dilute Sodium Hydroxide Solution — Approximately 10 percent.

E-1.5 Murexide Indicator Solution — Grind 0.2 g of murexide with 10 g of sodium chloride until the mixture is homogeneous. Thus 0.2 g of the mixture is suitable for 100 ml of the sample solution.

E-1.6 Patton and Reader's Indicator — Grind thoroughly 1 g of Patton and Reader's Indicator (calcon carboxylic acid) and 100 g AR anhydrous sodium sulphate powders in a grinder and mix the two solid powders thoroughly. Transfer the mixture to a dry air tight container. Use 1g of this solid mixture for every titration for calcium analysis.

E-1.7Ammonium Chloride-Ammonium Hydroxide Buffer Solution — Dissolve 67.5 g of ammonium chloride in a mixture of 570 ml of ammonium hydroxide (RD 0.90) and 250 ml of water. Also dissolve separately a mixture of 0.931 g of disodium ethylene diamine tetraacetatedihydrate and 0.616 of magnesium sulphate ($MgSO_4.7H_2O$) in about 50ml of water. Mix the two solutions and dilute to 1 litre.

> NOTE—Five millilitres of buffer solution added to 50ml of distilled water should not consume more than a drop of EDTA solution to change to distinct blue with eriochrome black T indicator.

E-2 PROCEDURE

E-2.1 Standardization of EDTA Solution

Transfer 25 ml of standard calcium solution into a 250 ml conical flask, add 25 ml of water, 10 ml of ammonium chloride-ammonium hydroxide buffer solution, 5 drops of the eriochrome black T indicator solution and titrate against the standard EDTA solution to a pure blue end point.

E-2.2 Titrate 23 ml of the buffer solution with EDTA solution using eriochrome black T indicator. Subtract the buffer correction for 10 ml (usually it will be 0.1 ml) from the reading obtained in **E-2.1** and note the final titre value. Calculate the calcium equivalent of 1ml of EDTA solution (say A).

E-3 DETERMINATION OF CALCIUM AND MAGNESIUM

E-3.1 Transferexactly100 ml of the solution preserved in **C-2** into a 250 ml conical flask and 10 ml of ammonium chloride-ammonium hydroxide buffer solution, 5drops of eriochrome black T indicator solution and titrate against standard EDTA solution till wine red colour of the solution changes to pure blue endpoint. Note the EDTA solution (V_1) used in the titration.

E-3.2 Transfer exactly100 ml of the solution preserved in **C-2** into 250 ml conical flask and 5ml sodium hydroxide solution and stir well. Add 0.2 g murexide (or 1g Patton and Reader's Indicator as in **E-1.6**) and titrate against standard EDTA solution till wine red colour of the solution changes to pure blue end point. Note the volume of the standard EDTA solution (V_2) used in the titration.

E-4 CALCULATION

Calcium (as Ca), percent by mass (on dry basis) = $\frac{A \times V_2}{M}$

Magnesium (as Mg), percent by mass (on dry basis) = $\frac{A \times (V_1 - V_2)}{M} \times 0.606\,8$

where,

A = calcium equivalent in mg of 1ml of EDTA solution determined in **E-2.2,**

V_1 = volume in ml of standard EDTA solution used in **E-3.1,**

V_2 = volume in ml of standard EDTA solution used in **E-3.2** (for Ca only); and

M = mass in g of the dried sample taken for the test in **C-2.**

ANNEX F

[*Table* 1, *Sl No.* (vii)]

DETERMINATION OF SULPHATE

F-1 GRAVIMETRIC METHOD

F-1.1 Reagents

F-1.1.1 *Dilute Hydrochloric Acid* — Approximately 4 N.

F-1.1.2 *Barium Chloride Solution* — Approximately 10 percent.

F-1.2 Procedure

Dissolve about 10 g of dried salt in about 400 ml of water, filter and wash the residue free from soluble salts. Collect the filtrate and washings. Add one drop of methyl orange and 10 ml of dilute hydrochloric acid or more till it is pink and then boil. Add to the boiling solution, 10 to 12 ml barium chloride solution drop by drop so that the addition is in slight excess and continue boiling for 4 min to obtain a granular precipitate. Allow to stand for 4 h and filter through a weighed sintered glass crucible (G No. 4) or Gooch crucible. Wash the precipitate till free from chloride and dry to constant mass at 105 to 110°C. Alternatively, filter through Whatman filter paper No. 42 and wash till it is free from chloride. Ignite and determine as barium sulphate.

F-1.3 Calculation

Sulphate (as SO_4), percent by mass = $\frac{M_1}{M_2} \times 41.13$

F-2 VOLUMETRIC METHOD

F-2.1 Reagents

F-2.1.1 *Standard Barium Chloride Solution* — 0.05 N.

Dissolve 6.108 g of barium chloride dihydrate ($BaC1_2.2H_20$) in water and make up to1litre.

F-2.1.2 *Dilute Hydrochloric Acid* — Approximately 1 N.

F-2.1.3 *Standard EDTA Solution* — Weigh 3.72 g of Disodium ethylene diamine tetra acetate dihydrate in water and dilute in a graduated flask to 1 litre. The solution shall be standardized frequently against standard barium chloride solution (**F-2.1.1**) and follow the procedure given in **E-3**. One millilitre of standard EDTA solution is equal to 0.001374 g of Ba or 0.00096 g of SO_4

F-2.1.4 *Eriochrome Black T Indicator Solution* — Same as in **E-1.3**.

F-2.1.5 *Ammonium Chloride-Ammonium Hydroxide Buffer Solution*—Same as in **E-1.7**.

F-2.2 Procedure

F-2.2.1 Pipette out 20 ml of standard barium chloride solution into a conical flask, add 2 drop of hydrochloric acid and 10 ml of ammonium chloride ammonium hydroxide buffer solution. Dilute it with water to about 50 ml, add five drops of eriochrome black T indicator solution and titrate against standard EDTA solution to pure blue end point. Note the titre value (say A).

F-2.2.2 Pipette out 100 ml of the solution preserved in **C-2** and add two drops of hydrochloric acid and heat to gentle boiling. To the hot solution, add with a pipette 20 ml standard barium chloride solution. Boil gently for about 5 min and then cool to room temperature. The solution will be neutral or slightly acid when tested with a litmus paper filtration. Add 10 ml of ammonium chloride-ammonium hydroxide buffer solution and five drops of eriochrome black T indicator solution. Titrate against standard EDTA solution to pure blue end point. Note the titre value (say B).

F-2.3 Calculation

$$\text{Sulphate (as } SO_4\text{), percent by mass (on dry basis)} = \frac{A+V_1-B}{M} \times 0.96$$

where,

A = volume in ml of standard EDTA solution used in **F-2.2.1**;

V_1 = volume in ml of standard EDTA solution used in **E-3.2** (total Ca + Mg);

B = volume in ml of standard EDTA solution used in the titration in **F-2.2.2**; and

M = mass in g of the dried sample taken for the test in **C-2**.

ANNEX G

[*Table* 1, *S1 No.* (viii)]

DETERMINATION OF IODINE

G-1 REAGENTS

G-1.1 Methyl Orange Indicator — Dissolve 0.01 g methyl orange in water and dilute to 100 ml.

G-1.2 Dilute Sulphuric Acid — Approximately 2 N.

G-1.3 Bromine Water — Saturated aqueous solution.

Determine the approximate concentration (milligram of bromine per millilitre) by adding (from a burette) measured volume to a flask containing 5 ml of 10 percent potassium iodide solution and 5 ml dilute sulphuric acid and titrating the liberated iodine with 0.1 N sodium thiosulphate solution, one millilitre of 0.1 N $Na_2S_2O_3$ = 8 mg of bromine = 12.7 mg of iodine.

G-1.4 Sodium Sulphite Solution — Approximately 1 percent (*m*/*v*).

G-1.5 Phenol Solution — Approximately 5 percent (*m*/*v*).

G-1.6 Potassium Iodide Solution — Approximately 10 percent (*m*/*v*).

G-1.7 Standard Sodium Thiosulphate Solution — 0.005 N.

Prepare 0.1 N solution by dissolving 25 g of sodium thiosulphate AR grade in 1000 ml water. Further, dilute 50 ml of this solution to 1000 ml to give 0.005 N solution.

G-1.8 Starch Solution — One percent, freshly prepared (*m*/*v*).

G-1.9 Sodium Chloride Solution — Dissolve 50 g AR grade sodium chloride (free from iodine) in water and make up the volume to 250 ml.

G-1.10 Potassium Iodide for Control – Determination — Dissolve 0.3270 g potassium iodide in water and makeup the volume to 250 ml. Dilute 50 ml of this solution to 250 ml, and use 5 ml (1.0 mg iodine and 1.308 mg potassium iodide) for control determination.

G-2 PREPARATION OF SAMPLE SOLUTION

Dissolve 100 g accurately weighed material in water and make up the volume to the mark in a 500 ml one mark graduated flask. Take 100 ml of this solution for test under **G-3**.

G-3 PROCE-DURE

G-3.1 Method A (Referee Method)

G-3.1.1 Pipette 100 ml of the prepared sample solution (20 g NaCl content) (*see* **G-2**) into 200 ml conical flask. Neutralize to methyl orange indicator with dilute 2 N sulphuric acid. Add bromine water dropwise from burette in a quantity equivalent to 20 mg of bromine. After a few minutes, destroy most of the remaining free bromine by adding sodium sulphite solution dropwise while mixing to pale yellow colour. Wash down the neck and sides of the flask with water and completely-remove free bromine by addition of 1or 2 drops of phenol solution.

G-3.1.2 Add 1 ml of dilute sulphuric acid and 5 ml of potassium iodide solution and titrate the liberated iodine against standard sodium thiosulphate solution, adding 1 ml starch solution near the end of titration. Carry out a blank determination on reagents make one or more control determinations, using 100 ml sodium chloride solution to which has been added appropriate quantity of potassium iodide control solution.

G-3.1.3 Calculation

$$\text{Iodine (I), ppm (on dry basis)} = \frac{528.75 \times (V_1 - V_2) \times F}{M \times (100 - m)} \times 100$$

where,

V_1 = volume in ml of standard sodium thiosulphate solution required for the test with the prepared sample solution;

V_2 = volume in ml of standard sodium thiosulphate solution required for the blank determination;

$F = \frac{actual\ normality\ of\ the\ sodium\ thiosulphate}{normal\ normality\ of\ sodium\ thiosulphate}$;

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

m = percentage moisture content calculated as per Annex A.

G-3.2 Method B (Alternate Method when Iodate is Used for Iodizing the Salt)

G-3.2.1 Accurately weigh about 20 g of salt sample. Dissolve it in about 100 ml water. Acidify with 10 ml of dilute 2 N sulphuric acid. Add 10 ml of 10 percent potassium iodide solution. Titrate the liberated iodine against standard 0.005 N sodium thiosulphate solution adding 2 ml of starch solution near the end of titration.

The solution will turn yellow if iodine is present. Stopper the flask and place it in the dark for 10 min (This is to avoid side reaction from occurring which can generate more iodine from iodide by exposure to light). Remove the flask from dark place.

$$\text{Iodine (I), ppm (on dry basis)} = \frac{105.7 \times V_1 \times F}{M \times (100 - m)} \times 100$$

where,

V_1= volume in ml of 0.005N sodium thiosulphate solution required for titration;

M = mass, in g, of the salt sample taken for analysis;

m = percentage moisture content calculated as per Annex A; and

$\text{F} = \frac{\text{actual normality of sodium thiosulphate}}{\text{nominal normality of sodium thiosulphate}}$

ANNEX H

[*Table* 1, *Sl No.* (ix)]

DETERMINATION OF ALKALINITY

H-1 REAGENTS

H-1.1 Standard Hydrochloric Acid — 0.1 N.

H-1.2 Methyl Orange Indicator — Dissolve 0.1 g of methyl orange in 100 ml of water.

H-2 PROCEDURE

H-2.1 Procedure for Common Salt

Pipette out 100 ml of the solution, preserved in **C-2** and titrate against standard hydrochloric acid using and titrate against standard hydrochloric acid using methyl orange as indicator.

H-3 CALCULATION

Alkalinity (as Na_2CO_3), percent by mass = $5.3\,\frac{V}{M}$

where,

V = volume in ml of standard hydrochloric acid used in the titration, and;

M = mass in g of the prepared sample taken for the test.

ANNEX J

[*Table* 1, *Sl No.* (x)]

DETERMINATION OF LEAD

J-1 Two methods have been specified for determination of lead - the chemical method and the atomic absorption spectrophotometric method. The atomic absorption spectrophotometric method be taken as referee method.

J-2 CHEMICAL METHOD

J-2.1 Apparatus

J-2.1.1 *Nessler Cylinder*, 50 ml capacity**.**

J-2.2 Reagents

J-2.2.1 *Acetic Acid***,** approximately 33 percent (*v*/*v*)

J-2.2.2 *Dilute ammonium hydroxide*, approximately 4 N.

J-2.2.3 *Potassium cyanide solution* – Dissolve 10 g of potassium cyanide in 90 ml of water, add 2 ml of hydrogen peroxide (20 volume strength), allow to stand for 24 h and make up to 100 ml with water.

J-2.2.4 *Sodium Sulphide Solution* – Dissolve 10 g of sodium sulphide ($Na_2S.9H_2O$) in 100 ml of water.

J-2.2.5 *Standard Lead Solution* – Dissolve 0.160 g of lead nitrate in 5 ml of concentrated nitric acid (confirming to IS 264) and dilute to 100 ml in a graduated flask. Again dilute 10 ml of the solution to 1 000 ml. One millilitre of the solution finally obtained contains 0.01 mg of lead (as Pb).

J-2.3 Procedure

J-2.3.1 Dissolve 4 g of the dried sample (*see* **C-1**) in water in a Nessler cylinder and add 5 ml of acetic acid. Make the mixture alkaline with dilute ammonium hydroxide and add 1 ml of potassium hydroxide and add 1 ml of potassium cyanide solution. If turbid, filter. Add two drops of sodium sulphide and mix well. Carry out a control test in another Nessler Cylinder in exactly the same manner but using 1 ml of standard lead solution in place of the dried sample. Dilute the solution in both the cylinders to 50 ml mark. Compare the colour produced in the two cylinders against a white background.

J-3 ATOMIC ABSORPTION SPECTROPHOTOMTRIC METHOD

J-3.1 Principle

Test portions are dried and then ashed at 450°C under a gradual increase (about 50°C/ hr) in temperature, 6 N HCl (1+1) is added and the solution is evaporated to dryness. The residue is dissolved in 0.1 N HNO_3 and the analyte is determined by flame and graphite procedures.

J-3.2 Apparatus

J-3.2.1 *Atomic Absorption Spectrophotometer*, with an air acetylene burner or nitrous oxide-acetylene burner for flame and a graphite furnace for electro-thermal determinations with appropriate background (non-atomic correction) Instrument parameters are usually given by the manufacturer in the manual provided with the instrument.

J-3.2.2 *Hollow cathode or electrodeless discharge lamps for all elements.*

J-3.2.3 *Furnace*, programmable or muffle furnace with thermostat maintaining 450 ± 25°C.

J-3.2.4 *Hot Plate*, with heating control to heat up to 300°C

J-3.2.5 *Quartz or Platinum Dishes*

J-3.2.6 *Polystyrene Bottles, with leak proof closures* – 100 ml.

> Note – Carefully clean and rinse all glassware and plasticware with HNO_3 or HCL to avoid metal contamination. First wash with water and detergent, rinse with tap water, followed by distilled water, then with acid (1+9) and finally 3-4 times with distilled water.

J-3.3 Reagents

J-3.3.1 *Water*, redistilled or distilled.

J-3.3.2 *20 percent Sulphuric Acid.*

J-3.3.3 *Hydrochloric Acid A.R (6N)* – Dilute 500 ml hydrochloric acid to one litre with water.

J-3.3.4 *Nitric Acid A.R 0.1 M* – Dilute 7 ml concentrated acid to one litre.

J-3.3.5 *Nitric Acid Concentrated (Sp. Grade 1.40)*

J-3.3.6 *Lead Standard Solution -1 mg/ml* – Dissolve 1 000 g Pb in 7 ml concentrated HNO_3 in one litre volumetric flask. Dilute to volume with water. Commercially available standard solutions for AAS may be used for all metal standards.

J-3.3.7 *Working Standard Solution* – For graphite furnace analysis dilute standard solutions with 0.1 M HNO_3 to a range of standards that cover the linear range of the elements to be determined. For flame analysis dilute standard solutions with 0.1 M HNO_3 to arrange of standards that covers the concentration of the elements to be determined.

J-3.4 Procedure

J-3.4.1 *Preparation of the Sample*

Weigh accurately about 25 g of well homogenized sample into a clean silica dish. Add 25 ml of 20 percent sulphuric acid. Mix thoroughly with a glass stirring and ensuring all sample material is wetted by the acid. Rinse stirring rod with water and silica dish. Dry the contents of the dish thoroughly on a steam bath or in an oven around 110°C. When the sample is thoroughly dried, heat the contents of the dish with a soft flame (such as that of a Bunsen Burner) until all volatility or readily combustible matter has been removed. Transfer the dish to a furnace set at 250°C. Slowly raise the temperature to 500°C. Ash at this temperature for about 6 to 8 hours. Remove the dish and cool. Ash should now be white or brownish red and essentially be carbon free. If ash contains carbon particles, wash down sides of dish with water and add 2 ml of HNO_3 until white/brownish red, carbon free ash is obtained. When clean ash is obtained, remove the dish from furnace, cool and add 1 ml of HNO_3 and 10 ml of water. Heat on hot plate till sample ash is dissolved. Quantitatively transfer the contents of the dish to a 10 ml of HCl (1+1) and transfer the solution again to the same volumetric flask to volume with water.

Prepare sample blank by following the same procedure as described for sample. Use same quantities of reagents including water for both sample and blank. Subject both sample and blank to identical treatment (even the length of the time kept in the furnace etc.)

Notes

1. Do not ash HNO_3 in furnace. Always dry HNO_3 (in the dish) on steam bath or on hot plate and then ash in furnace.
2. Do not allow sample to ignite during any stage of heating.
3. If the calcium content of the sample is high, then avoid the use of sulphuric acid and ash at temperature not exceeding 470°C.

J-3.4.2 *Determination*

Atomic Absorption Spectrophotometry – Lead in foods generally requires graphite furnace AAS (GFAAS) for determination.

a) Set the instrument as per the previously established optimum conditions as per the guidelines given in the Instrument Manual (provided along with the instrument). The standard conditions for Atomic Absorption Spectrophotometer are wavelength of 217 nm and air-acetylene as flame grades.
b) Determine absorbance of sample solutions(s) and blank.
c) Calculate the heavy metal content from standard curve.

J-3.4.3 *Preparation of Standard Curve*

Read the absorbance of a series of standard metal solutions in the Atomic Absorption Spectrophotometer after setting the instrument as per optimum conditions. Plot absorbance against µg of metal/ml solution.

ANNEX K

[*Table* 1, *Sl No.* (xi)]

DETERMINATION OF ARSENIC

K-1 METHOD I (GUTZEIT METHOD)

Weigh accurately 1.00 g of the material and dissolve in 10 ml of water and carry out the test for arsenic as prescribed in IS 2088 using 0.001 32 mg of arsenic trioxide (As_2O_3) for preparing the comparison stain.

K-2 METHOD II (SILVER DIETHYL DITHIOCARBAMATE RED COMPLEX B ABSORPTIOMETRIC METHOD)

K-2.1 Principle

Arsenic reacts with a solution of silver diethyl dithiocarbamate, $[(C_2H_5)_2NCS_2Ag]$ in pyridine to form a soluble red complex which has an absorption maximum at 540 (nm) nanometre. The arsenic shall be in the trivalent state in the sample which is secured by reducing the arsenate with potassium iodide and stannous chloride in acid media. The arsenic is converted into arsine by the treatment of hydrochloric acid and zinc and evolved arsine is absorbed in the reagent to form a red complex. 'Using any standard photoelectric absorption meter, absorption measurement is done at 540 nm, with coloured red complex solution against blank reagent solution for total transmittance. From the transmittance or optical density obtained with known arsenic content covering the 0-10 µg, standard calibration graph is prepared by plotting the percent transmittance or optical density or logarithm of percent transmittance (log T) against known concentration. As it obeys Beer's Law, log T or the optical density are directly proportional to the concentration and only a few points are required to establish the graph for the determination of arsenic under the experimental condition.

K-2.2 Apparatus

The evolution and absorption apparatus as shown in Fig 1 shall consist of the following:

K-2.2.1 *Conical Flask* — 100 ml for the evolution of arsine (*A*).

K-2.2.2 *Connection Tube* —To trap hydrogen sulphide (B).

K-2.2.3 *Absorption Tube (C)*

K-2.2.4 *Spring Clip* — To-secure the joint connecting B to C. (It may be either ground cone-socket joint or ball joint with hooks).

K-2.3 Spectrophotometer or Photoelectric Absorptiometer

K-2.4 Reagents

K-2.4.1 *Potassium Iodide* — 150 g/l solution. Store in a dark place.

K-2.4.2 *Stannous Chloride Solution*

K-2.4.3 *Zinc Shots* — Arsenic-free.

K-2.4.4 *Silver Diethyl Dithiocarbamate* — 5g/l solution in pyridine. Dissolve 1 g of silver diethyl dithiocarbamate (SDDC) in pyridine (relative density 0.980 approximately) and dilute to 200ml with pyridine. Store in a well stoppered glass bottle protected from light. This solution is stable for two months (*see* Note).

NOTE — If suitable reagent is not available, it may be prepared from sodium diethyl dithiocarbamate by the method given below:

a) *Purification of sodium diethyl dithiocarbamate* — Dissolve 10g sodium diethyl dithiocarbamate [$(C_2H_5)_2NCS_2Na.3H_2O$] in 35 ml of ethanol (95 percent *v*/*v*) and filter. Add to this solution with continual stirring, 100 ml of diethyl ether. Filter with suction, wash the precipitate with ether and dry in air.

b) *Preparation of the reagent* — Dissolve 2.25 g sodium diethyl dithiocarbamate in 100 ml water. Dissolve 1.7 g of silver nitrate in 100 ml water. Mix the two solutions slowly with continuous agitation. Keep the mixture at a temperature below 10°C. Filter with the aid of suction pump and dry the product in vacuum at room temperature. Preserve in a cool place protected from light. The solution is unsatisfactory if the optical density is less than 0.03 kg of arsenic using 5 ml of this solution.

K-2.5 Procedure

K-2.5.1 Transfer 10 ml of the standard arsenic solution containing 10 kg of arsenic and 10 ml of the concentrated hydrochloric acid into a 100ml conical flask-4 and dilute it approximately to 40 ml with water. Add 2 ml of the potassium iodide solution (**K-2.4.1**) and 0.5 ml of the stannous chloride solution (**K-2.4.2**). Mix and allow to stand for 15 min. Place some dry lead acetate paper in the lower portion of the connection tube B and glass wool (or cotton) moistened with lead acetate solution in its upper portion. Assemble the apparatus. Transfer 5.0 ml of silver diethyl dithiocarbamate solution to the absorption tube. After 15 min standing period, introduce 5 g of the zinc shots (**K-2.4.3**) into the conical flask A and rapidly replace the cone into the neck of the flask. Allow the reaction to continue for 45 min. Disconnect the absorption tube C and tilt the absorber tubes of that the reagent solution flows back and forth between the absorber and bulb to dissolve any red complex so as to thoroughly mix the solution. Transfer the solution to the photometric cell. Absorption measurement is done at 540 nm with 5ml coloured red complex solution in a cell of 1cm thickness against blank reagent solution for total transmittance. Volume and optical path of the comparison cell shall be the same for both the measurements and may be adjusted to suit the instrument. Alternatively, record its optical density at 540 nm as both are calibrated on the scale.

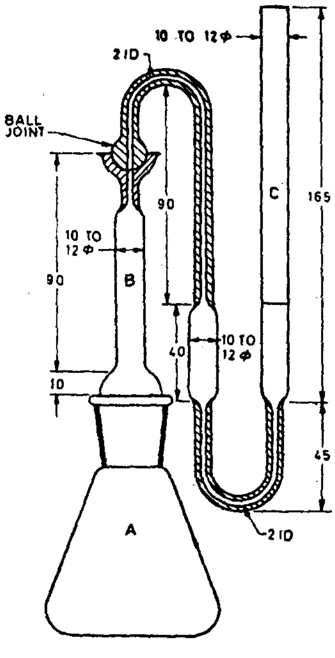
K-2.5.2 Transfer 10 g of the dried sample into the conical flask and carry out the procedure as described above. With the absorbed solution, measure its percent transmittance or optical density at 540 nm against total transmittance for the reagent. Since the colour is not stable, measurement of optical density or percent transmittance shall be done immediately.

K-2.5.3 The material shall be taken to have not exceeded the limit prescribed in Table 1 if the percent transmittance is more than the percent transmittance obtained for 10 ml of standard arsenic solution (10 µg). If the optical density with the sample is less than the optical density obtained with 10 ml of standard solution, it passes the limit test of 1 ppm arsenic.

K-2.5.4 *Plotting and Determination of Arsenic*

If it is desired to know the exact amount of arsenic, determine the percent transmittance or optical density for another standard solution containing 5 µg of arsenic.

Since it obeys Beer's Law, draw a graph plotting the logarithm of the percent transmittance (log T) or optical density determined for the standard solution against their arsenic content. Straight line is obtained passing the points obtained for 0, 5 and 10 µg of arsenic. From the graph, read the amounts of arsenic corresponding to the respective percent transmittance or optical density of the sample and blank solution.



All dimensions in millimetres.

FIG. 1 APPARATUS FOR ARSENIC DETERMINATION

K-2.5.5 *Calculation*

Arsenic content in the sample in µg/g (ppm) = $0.1(M_1 - M_2)$

where,

M_1 = mass in µg in the sample; and

M_2 = mass in µg in the blank.

ANNEX L

[*Table* 1, *S1 No.* (xii)]

DETERMINATION OF IRON

L-1 APPARATUS

L-1.1 Nessler Cylinders —50 ml capacity.

L-2 REAGENTS

L-2.1 Thioglycollic Acid — Containing not less than 97 percent of thioglycollic acid by mass.

L-2.2 Concentrated Ammonium Hydroxide — Relative density 0.90.

L-2.3 Standard Iron Solution — Dissolve 2.81 g of ferrous ammonium sulphate [$FeSO_4(NH_4)2SO_4.6H_2O$] in water, add a few millilitres of dilute sulphuric acid and dilute to 1 000 ml with water.

L-2.4 Dilute Standard Iron Solution — Dilute 10 ml of the standard iron solution (**L-2.3**) to 1 000 ml with water. The solution should be prepared freshly as required. This solution contains 0.000 004 g of iron per millilitre (1 ml = 0.004 mg Fe).

L-3 Procedure

L-3.1 Pipette out 20 ml of the solution prescribed in **C-2** into a Nessler cylinder. Dilute to 20 ml with water and add 4 drops of thioglycollic acid followed by 1 ml concentrated ammonium hydroxide. Carry out a control test in another Nessler cylinder using 5 ml of dilute standard iron solution and the same quantities of other reagents. Dilute the solution in both the cylinders to 50 ml and mix well.

L-3.2 The requirement specified in Table 1 shall be taken as not having been exceeded, if the intensity of colour produced with the material is not greater than that obtained in the control test.

ANNEX M

(*Clause* 6)

METHODS OF SAMPLING OF IODIZED SALT AND REFINED IODIZED SALT

M-1 GENERAL REQUIREMENTS OF SAMPLING

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

M-1.2 Samples shall not be taken at an exposed place.

M-1.3 Precautions shall be taken to protect the samples, the sampling instrument and the containers for samples from adventitious contamination.

M-1.4 To draw a representative sample, the contents of each container elected for sampling shall be mixed thoroughly by suitable means.

M-1.5 The sample shall be placed in suitable, clean, dry and air-tight glass containers.

M-1.6 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.

M-2 SCALE OF SAMPLING

M-2.1 Lot

All the packages in a single consignment of iodized salt drawn from a single batch of manufacture shall constitute a lot. If the consignment consists of different batches, the batches shall be marked separately and the group of containers in each batch shall constitute separate lots.

M-2.1.1 The number of packages (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 Packages to be Selected

For Sampling

Lot Size (N) (1)	No. of Packages to be Selected (n) (2)
4 to 50	3
51 to 100	4
101 to 150	5
151 to 300	7
301 to above	10

M-2.1.2 These packages shall be selected at random from the lot, and to ensure randomness of selection, a random number table (*see* IS 4905) may be used.

M-3 PREPARATION OF TEST SAMPLES

M-3.1 Packages

M-3.1.1 From each of the packages selected in accordance with **M-2.1.2**, a portion of the material (about 200 g) shall be drawn with the help of a suitable sampling instrument.

M-3.1.2 Out of these portions, equal quantities of the material shall be taken and mixed thoroughly to form a composite sample of about 400 g. The composite samples shall be divided into three equal parts, namely, one for the purchaser, one for the supplier and the third to be used as free-sample.

M-3.1.3 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 sample representing the container samples shall be marked for the purchaser, another for the supplier and the third to be as free-sample.

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

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

M-4 NUMBER OF TESTS

M-4.1 Tests for the determination of moisture content, chloride content (as NaCl) and level 1 of iodine shall be performed on each of the individual samples.

M-4.2 Tests for the determination of all other characteristics given under 4 shall be performed on the composite sample (*see* **M-3.1.2**).

M-5 CRITERIA FOR CONFORMITY

M-5.1 For Individual Samples

From the test results the average (x) and the range (R) or ($\bar{R}$) shall be computed for each of the characteristics mentioned in **M-4.1**. Range (R) is defined as the difference between the maximum and the minimum values of the individual test results. Range ($\bar{R}$) is the average of two ranges calculated on each of the two sets of five consecutive test results when the total number of test results on a characteristic is 10. The criteria for conformity of the lot in respect of each of these characteristics is that (x–0.6R) or (x–0.6$\bar{R}$) should be greater than or equal to the corresponding minimum requirements specified under **4**.

M-5.2 For Composite Samples

The lot shall be considered to have passed in respect of the characteristics tested on the composite test sample, if the test results satisfy the corresponding requirements given under **4**.

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