BELIZE NATIONAL STANDARD

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BELIZE NATIONAL STANDARD
SPECIFICATION FOR SALT

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Government Complex Building
Mahogany Street Extension
P.O. Box 1647
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CENTRAL AMERICA

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BELIZE NATIONAL STANDARD
SPECIFICATION FOR SALT

Committee Representation

The preparation of this standard for the Standards Advisory Council established under the Standards Act of 1992, was carried out under the supervision of the Bureau's Technical Committee for Food and Food Related Products, which at the time comprised the following members:

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TECHNICAL SECRETARY
Mrs. Helen Reynolds Arana
Belize Bureau of Standards

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0 FOREWORD

0.1 This standard was prepared with the intention of providing requirements for salt for usage in food processing as well as for general household use. Details of relevant test methods are also included.

Metric units are used throughout the standard.

0.2 In the preparation of this standard considerable assistance was derived from the JS 87:1985 – Specification for Salt – Jamaica Bureau of Standards and the Guatemala Regulations for the Fortification of Salt for Direct Human Consumption.

0.3 This standard shall be read in conjunction with the Belize Agricultural Health Authority (Food Safety) Regulations, 2001.

1 SCOPE

1.1 This standard prescribes requirements for salt which is intended for general household use as well as for food processing. Salt which is a by-product of the fertilizer or chemical industries is not covered by the standard.

2 PRODUCT DESCRIPTION

The product shall be white crystalline sodium chloride free from visible impurities and shall be either rock salt, sea salt or vacuum evaporated salt.

3 DETAILED REQUIREMENTS

3.1 **Additives**. All additives used shall be of food grade quality.

3.1.1 'Free flowing' or free running' salt shall contain an approved anticaking agent.

3.1.2 Salt labelled 'iodized' shall contain a minimum of 20 milligrams of iodine and a maximum of 60 milligrams of iodine per kilogram of salt when tested in accordance with the requirements of Appendix C.

3.1.3 Salt labelled "flouridized" shall contain a minimum of 175 milligrams of fluoride and a maximum of 225 milligrams of fluoride per kilogram of salt.

3.1.4 Not more than 13 ppm of potassium hexacyanoferrate II (ferrocyanide) [or hexacyanoferrate II (ferrocyanide) salts of sodium, calcium and manganese] shall be present when tested in accordance with the requirements of Appendix D.

3.2 **Moisture**. The dried product shall contain not more than 0.5% by weight of moisture when tested by the method stipulated in Appendix B. The moisture content of undried salt shall not he greater than 4% by weight.

3.3 **Particle size**

3.3.1 ***Recrystallized table salt***. Nor less than 95% shall pass through a U.S. standard No. 25 (710μ) sieve and not more than 10% shall pass through a U.S. Standard No. 70(212μ) sieve.

3.3.2 ***Solar evaporated salt.*** One hundred per cent shall pass through a U.S. Standard No.16 (1.18μ) sieve and not more than 20% shall pass through a U.S. Standard No. 100 (150μ) sieve.

3.3.3 ***Special products***. The particle size is subject to agreement between manufacturer and purchaser.

3.4 **Purity**. The limits of impurities, when determined by the methods described in appendices E to K shall be as follows:

(a) Not more than 10 ppm of iron shall be present (see Appendix E).

(b) The sulphate content shall not exceed 0.5% (see Appendix F).

(c) The calcium and magnesium content shall not exceed 0.2% by weight of each ion (see Appendix F).

(d) Matter insoluble in water shall not exceed 1% (see Appendix G).

(e) Not more than 2 ppm of arsenic shall be present (see Appendix H).

(f) Not more than 2 ppm of copper shall be present (see Appendix J).

(g) Not more than 2 ppm of lead shall be present (see Appendix K).

3.5 **Sodium chloride**. Salt shall contain not less than 98% by weight of Sodium chloride when tested according to the method described in Appendix A.

4 PACKAGING

4.1 Only packaging materials which are not likely to impair the organoleptic or chemical characteristics of salt or make them harmful to health may be used. The materials used for packaging should be mutually compatible with salt.

5 LABELLING

5.1 Labelling of this commodity shall be in conformance with BZS 1: Part 2: 1998 – Labelling of Prepackaged Food.

6. SAMPLING

The salt shall be sampled in accordance with Appendix L.

APPENDIX A

DETERMINATION OF CHLORIDE IN SALT

A.1 REAGENTS

(a) Concentrated nitric acid (HNO_3)

(b) 0. 1M (0. 1N) silver nitrate solution ($AgNO_3$)

(c) Diluted nitric acid (0.5 mL HNO_3 to 200 mL H_2O).

(d) 0.1M (0.1N) Hydrochloric acid (HCI).

A.2 PROCEDURE

Weigh accurately 0.2 g of sample into a 400-ml beaker. Add 150 ml water and stir until the solid is dissolved. Add 0.5 ml concentrated HNO_3. Cool and add 0.1M (0.1 N) $AgNO_3$ slowly with constant stirring. Only a slight excess should be added. This is detected by allowing the precipitate to settle and adding a few drops of $AgNO_3$ until no further precipitation occurs. The determination should be carried out in subdued light.

Heat the suspension nearly to boiling while stirring constantly and maintain it at this temperature until the precipitate coagulates and the supernatant is clear. Ensure that precipitation is complete by adding $AgNO_3$ solution to the supernatant. Allow the solution to stand in the dark for 1 h after precipitation is complete.

Wash the precipitate two or three times by decantation with 10 ml of cold dilute HNO_3 before transferring the precipitate to a previously dried and weighed sintered glass crucible. Transfer the precipitate using a 'policeman' and wash the precipitate with dilute HNO_3 until 5-ml portions of the washing give no turbidity with one or two drops 0.1M (0.1N) HCI. Dry the crucible and contents at 140^{o}C (284^{o}F) in an air oven to constant weight.

A.3 CALCULATION

$$\% \text{ chloride} = \frac{02.2474 \text{ X wt. of residue X } 100}{\text{Wt. of sample used}}$$

APPENDIX B

DETERMINATION OF MOISTURE IN SALT

B.1 PROCEDURE

Place 10 g sample in a previously dried and weighed silica dish. Spread sample evenly over the bottom of the dish. Heat for 1-h periods at 140^{o}C until 2 consecutive weighings agree within 5 mg.

B.2 Calculation

$$\% \text{ Moisture} = \frac{\text{Weight loss} \times 100}{\text{Weight of sample}}$$

APPENDIX C

DETERMINATION OF IODINE IN IODIZED SALT

C.1 REAGENTS

(a) ***Bromine water (Br-H_2O)***. Determine approximate concentration (mg bromine/ml) by adding measured volume from burette to flask containing 50 mL H_20, 5 ml 10% potassium iodide solution and 5 ml H_2SO_4 (1 + 9), and titrating liberated iodine with 0.05M (0.1N) sodium thiosulphate, $Na_2S_2O_3$.

(b) ***Sodium thiosulpbate***, 0.0025M (0.005N), prepare daily by diluting 0.05M (0.1N) solution.

(c) ***Starch solution,*** 1% freshly prepared.

(d) ***Potassium iodide control solution***, 0.3270 g KI/250 ml. Dilute 50 ml to 250 ml, and use 5 ml (=1.0 mg iodine and 1.308 mg KI) for control.

C.2 PREPARATION OF SAMPLE

Dissolve 50 g sample in H_2O and dilute to 250 mL in volumetric flask. Take 25 ml for **C.3.1** or 50 ml for **C.3.2** aliquot for analysis.

C.3 PROCEDURE

C.3.1 (Applicable when $Na_2S_2O_3$ content is ≤0.5%). Place sample aliquot in 600-ml beaker and dilute to 300 ml. Neutralize to methyl orange with phosphoric acid, H_3PO_4, and add 1ml excess. Add excess bromine water and boil solution gently until colourless and then 5 min longer. Add few crystals salicylic acid and cool solution to about 20°C. Add I ml. H_3PO_4 and about 0.5 g KI and titrate iodine with 0.0025M (0.005N) $Na_2S_2O_3$, adding starch solution when liberated iodine colour is nearly gone.

C.3.2 (Not applicable in the presence of $Na_2S_2O_3$). Pipette 50 ml sample solution into 200-ml Erlenmeyer flask. Neutralize to methyl orange with 1M (2N) H_2SO_4. Add Br- H_2O dropwise from burette in amount equivalent to 20 mg Br. After few minutes, destroy greater portion of remaining free bromine by adding 1% sodium sulphite, (Na_2SO_3) solution dropwise while mixing. Wash down neck and sides of flask with H_2O and complete removal of bromine by adding 1 or 2 drops of 50% phenol solution and titrate liberated iodine with $Na_2S_2O_3$ solution, adding 1 ml starch indicator near end of titration. Correct determination for blank on reagents and make one or more control determinations, using 50 ml 20% reagent-grade NaCl solution to which has been added appropriate amounts of dilute control KI solution.

C.4. CALCULATION

1 mL 0.0025M (0.OO5N) $Na_3S_2O_2$ = 0.1058 mg 12 and 0.1384 mg KI.

APPENDIX D

DETERMINATION OF POTASSIUM HEXACYANOFERRATE (II) (FERROCYANIDE) IN SALT

D.1 REAGENTS

(a) ***Sulphuric acid***, 0.25M (0.5N)

(b) ***Iron (II)/Iron (III) (ferrous/ferric) solution.*** Dissolve 20 g ammonium iron (II) sulphate, $(NH_4)_2\ SO_4.\ FeSO_4.\ 6H_2O$] and 2.5 g ammonium iron (III) sulphate, [$(NH_4)_2\ SO_4.\ Fe_2\ (SO_4)_3.\ 4H_2O$] in water to which 10 ml of 0.25M (0.5N) $H_2\ SO_4$ has been added. Dilute to 100 ml, filter and store in a dark bottle.

(c) ***Phosphate solution***. Dissolve 70 g of potassium dihydrogen phosphate ($KH_2\ PO_4$) in water, add 50 ml O.25M (0.5N) $H_2\ SO_4$ and dilute to 1 L.

(d) ***Sodium chloride***. Before use, ignite at 500^0G for 2 h and allow to cool.

(e) ***Potassium hydroxide***. 0.1M (0.1N)

(f) ***Standard potassium hexacyano ferrate (II) solutions*** [$K_4Fe\ (CN)_6$]

(1) ***Stock solution***. Dissolve 2.294 g potassium hexacyanoferrate (**II**) trihydrate [$K_4Fe\ (CN)_6.\ 3H_2O$] in water, add 5 ml potassium hydroxide,0.IM (0. IN) arid dilute to 1L with freshly boiled and cooled water. Store in the dark.

(2) *Working solution*. Take 25 ml of hexacyanoferrate (**II**) stock solution, add 5 ml potassium hydroxide solution, 0.1M (0.1N.) and dilute to 1 L with freshly boiled and cooled water.

$$1ml = 0.05\ mg\ K_4Fe\ (CN)_6$$

D.2 PROCEDURE

Dissolve 10 $\pm$ 0.1 g of sample in about 40 ml of water in a Nessler tube graduated at 100 ml. Add 10 ml H_2SO_4, 0.25M (0.5N) and 5 ml of iron (**II**)/iron (**III**) 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 15 ppm $K_4Fe\ (CN)_6$ standard which is prepared similarly using 10 g of the sodium chloride reagent to which has been added 3 ml of the hexacyanoferrate (**II**) working solution in place of the 10 g of sample.

NOTE. This test is designed for 15 ppm of potassium hexacyanoferrate (II) although this amount of potassium hexacyanoferrate (II) is unlikely to be used as an anticaking additive.

APPENDIX E

DETERMINATION OF IRON IN SALT

E.1 REAGENTS

(a) *Standard iron (III) (ferric) solution*. Dissolve 0.864 g of AR ammonium iron **(III)** sulphate in 100 ml water, add 10 ml of concentrated HCI and dilute to 1 L

1 ml $\equiv$ 0.1 mg of Fe

(b) *0-phenanthroline*, 0.25% solution of the monohydrate in water

(c) *Sodium acetate*, **0.2M (0.2N)**

(d) *Hydroxylamine hydrochloride*, 10%

(e) *Bromophenol* blue indicator.

E.2 PROCEDURE

Take an aliquot of the unknown slightly acid solution containing 0.1 mg to 0.5 mg of iron and transfer it to a 50 ml volumetric flask. Determine by the use of a similar aliquot containing a few drops of bromophenol blue, the volume of sodium acetate solution required to bring the pH to 3.5 $\pm$ 1.0. Add the same volume of acetate solution to the original aliquot and then 5 ml of the 10% hydroxylamine hydrochloride and 5 ml of the 0-phenanthroline reagent. Dilute to 50 ml, mix and measure the intensity after 5 min to 10 min at 510 mμ.

Compare the intensity of the colour produced with standards similarly prepared.

APPENDIX F

DETERMINATION OF SULPHATE, CALCIUM AND MAGNESIUM IN SALT

F.1 REAGENTS

(a) 10% barium chloride ($BaCl_2$) solution

(b) 10% oxalic acid ($H2C_6O_4$) solution

(c) Ammonia (NH_4)

(d) 1% ammonium oxalate [$(NH_4C_2O_4$] solution

(e) Hydrochloric acid (HCl) (1 + 1)

(f) Diammonium hydroxide orthoposphate $(NH_4)_2 HPO_4$

(g) Ammonium hydroxide (NH_4OH) solution (1 +10)

(h) Methyl orange.

F.2 PROCEDURE

F.2.1 *Preparation of sample*. Weigh about 20 g sample accurately and transfer to a 400 ml beaker and dissolve in 200 ml HCl (1 + 3). Cover beaker, heat to boiling, and continue boiling gently for 10 min. Filter the solution and wash residue with small amounts of hot water until filtrate is chloride-free. Unite filtrate and washings, cool and dilute to 500 ml (solution A).

F.2.2 **Sulphate determination**

F.2.2.1 *Procedure*. Place 250 ml of solution A in a 400 ml beaker. Heat to boiling and add a slight excess of hot 10% $BaCl_2$ solution dropwise while stirring. Concentrate by heating gently and finally evaporate to dryness on steam-bath. Facilitate removal of free acid by stirring partly dried residue. Wash precipitate by decantation with small amounts of hot water. Test filtrate for presence of Ba. Wash precipitate on paper until filtrate is chloride-free, Dry and ignite the filter paper containing the precipitate carefully over Bunsen flame without inflaming. Heat for one-hour periods at 600^{o}C in a furnace to constant weight. Calculate the result on a moisture-free basis.

F.2 .2.2 *Calculation*

$$\% \text{ sulphate} = \frac{\text{Wt.BaSO}_4 \text{ X } 4\ 113}{\text{½ original wt. of sample x (100 - \% moisture)}}$$

F.2.3 **Calcium determination**

F.2.3.1 *Procedure*. Place the remainder of solution A in a 400-ml beaker. Add excess of 10% oxalic acid solution (10 ml usually is enough). Add few drops of methyl orange; neutralize while hot by adding NH_4OH (1:1) dropwise, stirring constantly. Add about 1 ml excess NH_4OH stir, and let it stand in a warm place for 3 h. Decant through filter paper, reserving the filtrate for magnesium determination. Test filtrate for Ca with ammonium oxalate solution. Wash the precipitate in the beaker once with 10 ml 1% ammonium oxalate solution, decanting through the filter paper. Reserve the filtrate and washings (solution B). Dissolve precipitate on the paper with hot HCl (1 + 1), using the same beaker; dilute to 100 ml, add a little more oxalic acid solution and precipitate as before. Let the solution stand for 3 h, filter and wash with 1% ammonium oxalate solution, as before, reserving filtrate and washings. Transfer the precipitate to a previously dried and weighed platinum crucible, dry, ignite and heat over a Meker burner to constant weight. Report as percent Ca on water-free basis.

F.2.3.2 *Calculation*

$$\% \text{ Ca on water-free basis} = \frac{\text{Wt. } CaC_2O_4 \text{ X } 3\ 128.2}{\text{½ original wt. of sample X (100 - \% moisture}}$$

F.2.4 Magnesium determination

F.2 .4.1 *Procedure*. Use the reserved combined filtrate and washing from the Ca determination (solution B). Concentrate the solution, if necessary, by boiling gently to about 200 ml (solution C); acidify with HC1 (1 + 1) and add 2 g to 3 g $(NH_4)_2\ HPO_4$ and enough HCI (1 +1) to produce a clear solution when all $(NH_4)_2\ HPO_4$ is dissolved. When cold, make slightly alkaline with NH_4 OH, stirring constantly. Add 2 ml excess NH_4 OH and let it stand For 12 h. Filter and wash 4 times by decant-tation with NH_4 OH (1 + 10). Dissolve the precipitate in HC1 (1 + 1), dilute to about 150 ml, add some $(NH_4)_2\ HPO_4$ and precipitate with NH_4 OH as before. Let stand 12 h, filter, wash chloride-free with NH_4 OH (1 + 10). Place in a weighed platinum crucible, char without inflaming and heat to constant weight in a furnace at 1000°C. Weigh as $Mg_2P_2O_7$

F.2.4.2 *Calculation*

$$\% \text{ magnesium} = \text{Wt. } Mg_2P_2O_7 \text{ x } 0.2184 \text{ x } 100$$

APPENDIX G

DETERMINATION OF INSOLUBLE MATTER IN SALT

G.1 PROCEDURE

Place 10 g sample in a 250 ml beaker, add 200 ml H_2O at room temperature and let it stand for 30 min, stirring frequently. Filter through a weighed Gooch crucible with an asbestos mat dried at 110°C (230°F). Transfer residue to Gooch crucible with the aid of a policeman using 10 ml portions of H_2O to wash the residue until a 10 ml portion of filtrate shows only a faint opalescence upon the addition of a few drops silver nitrate, ($AgNO_2$) solution.

Dry the crucible and contents to constant weight at 110°C (230°F).

Report the increase in weight of the Gooch crucible as insoluble matter and calculate the result on a moisture-free basis.

G.2 CALCULATION

$$\% \text{ insoluble matter} = S \times \frac{1 \times 100}{(10 - \% \text{ moisture})}$$

where:

I = increase in weight of crucible
S = weight of sample.

APPENDIX H

DETERMINATION OF ARSENIC - SILVER DIETHYL DITHIO-CARBAMATE RED COMPLEX ABSORPTIOMETRIC METHOD

H.1 PRINCIPLE

Arsenic reacts with a solution of silver diethyl dithio-carbamate, [AgS.CS.N $(C_2H_5)_2$] in pyridine to form a soluble red complex which has an adsorption maximum at 540 nm. 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 absorptiometer, 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 range 0 μg to 10μg (as 0 nm to 10 nm As), stand are 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 is directly proportional to the concentration anti only a few points are required to establish the graph for the determination of arsenic under the experimental condition.

H.2 REAGENTS

(a) *Potassium iodide*, 150 g/l solution. Store in a dark place

(b) *Stannous chloride solution*

(c) *Zinc shots*, arsenic-free

(d) *Silver diethyl ditbio-carbamate*, 5 g/l solutuin in pyridine. Dissolve 1 g of silver diethyl dithic-carbamate (SDDC) in pyridine (relative density = 0.980 approx.) and dilute to 200 ml with this pyridine. Store in a well-stoppered glass bottle protected from light. This solution is stable for 2 months.

H.3 APPARATUS

The evolution and absorption apparatus 'is shown in figure 1 shall consist of the following:

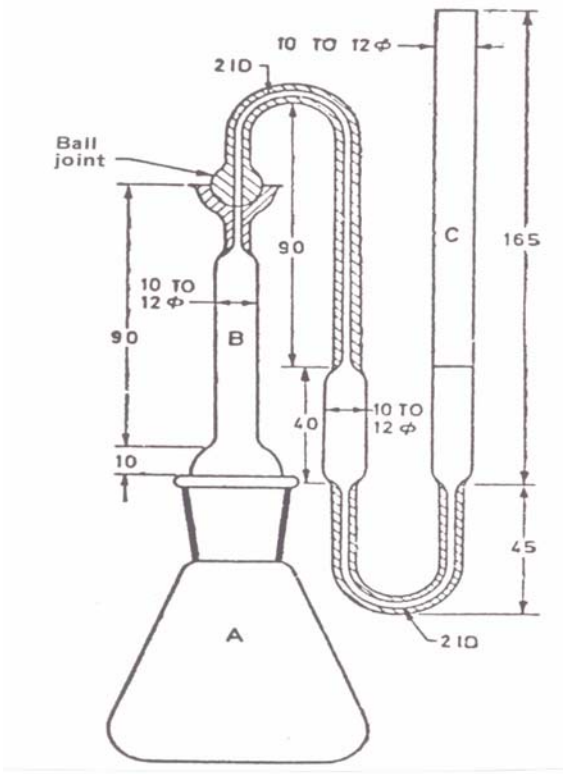
(a) *Conical flask A*, 100 ml. for the evolution of arsine
(b) *Connection tube B*, to trap hydrogen sulphide
(c) *absorption tube C*
(d) *Spring clip*, to secure the joint connecting B to C. (It may be either ground cone-socket joint or ball joint with hooks,)
(e) Spectrophotometer or photoelectric absorptiometer

NOTE. If suitable reagent is not available it may he prepared from sodium dietbyl dithio-carbamate by the method given below:

(1) Purification of sodium diethyl dithio-carbamate. Dissolve l0g sodium diethyl dithin-carbarmate [$(C_2H_2)_2$ .N.$CS_2Na.3H_2O$] in 35 ml of ethanol (95% 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.

(2) Preparation of the reagent. Dissolve 2.25 g sodium diethyl dithio-carbamate 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 (50°F). Filter with the aid of a suction pump and dry the product in vacuum at room temperature. Preserve in a cool place protected from light.

This solution is unsatisfactory if the optical density is loss than 0.03 μg of arsenic using 5 ml of this solution.



All dimensions in millimeters.

Figure 1. Apparatus for arsenic determination

H.4 PROCEDURE

H.4.1 Transfer 10 ml of the standard arsenic solution containing 10 μg of arsenic and 10 ml of the concentrated hydrochloric acid into a 100 ml conical flask A and dilute it approximately to 40 ml. with water. Add 2 mL of the potassium iodide solution and 0.5 ml of the stannous chloride solution. Mix, 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 dithio-carbamate solution to the absorption tube C. After the 15-min standing period, introduce 5 g of the zinc shots into the conical flask A and rapidly replace the cone into the neck of the tusk. Allow the reaction to continue for 45 min. Disconnect the absorption tube C and tilt the absorber tube so that the reagent solution flows back and forth between the absorber and bulb to dissolve any red complex and to thoroughly mix the solution. Transfer the solution to the photometric cell. Absorption measurement is done at 540 nm with 5 ml coloured red complex solution in a cell of 1 cm thickness against blank reagent for total transmittance. Volume and optical path of the comparison cell shall be 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.

H.4.2 Transfer 10 g of the dried sample into the conical flask and carry out 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 per cent transmittance stall be done immediately.

H.4.3 If it is desired to know the exact amount of arsenic, determine the per cent 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 per cent transmittance (log T) or optical density determined fort he standard solution against arsenic content. Straight line is obtained passing through 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 taken and blank solution.

H.5 CALCULATION

Arsenic content in the sample in μg/g (ppm) $0.1 (M_2 – M_2)$

where:

M_1 = mass in μg in the sample, and

M_2 = mass in μg in the blank.

APPENDIX J

DETERMINATION OF COPPER

J.1 REAGENTS

(a) *Citric acid*, solid
(b) *Ammonium hydroxide solution*, sp gr 0.90
(c) *Sodium diethyl dithio-carbamate solution*, 0.1% (w/v), aqueous
(d) *Carbon tetrachloride*, redistilled
(e) *Sodium sulphate*, anhydrous
(f) *Dilute nitric acid*, concentrated nitric acid of sp gr 1.42 diluted with an equal volume of water
(g) *Standard copper solution*, Weigh accurately 0.100 g of pure copper turnings, carefully dissolve in the minimum amount of nitric acid, cool and dilute to I L in a graduated flask. Pipette 10 ml of this solution into a 100 ml graduated flask and dilute to the mark. This solution contains 10 μg of copper per ml.

J.2 APPARATUS

Spectrophotometer

J.3 PROCEDURE

J.3.1 Transfer a 10 ml aliquot of the test solution **K.2.1** to a separating funnel. Add 1 g of nitric acid and dissolve it by shaking. Make the solution alkaline to litmus by adding ammonium hydroxide solution in small quantities. Add 5 ml of the sodium diethyl dithio-carbamate solution, shake thoroughly and extract with 5 ml portions of carbon tetrachloride until the final extract is colourless. Dry the combined extracts by shaking thoroughly with anhydrous sodium sulphate. Filter the dry extract and wash the filter paper with carbon tetrachloride. Make up the volume of the filtrate to 25 ml with carbon tetrachloride and measure the absorption at 437 nm by means of the spectrophotometer. Simultaneously, carry out blank determinations on the water and the reagents.

J.3.2 Prepare a series of standards by treating aliquots of the standard copper solution **J.1 (g)** in the same manner as the test solution. From the absorption of the standard solutions, prepare a standard curve plotting absorption values against concentrations. From the curve, obtain the weight of copper present in the test solution.

APPENDIX K

DETERMINATION OF LEAD

K.1 REAGENTS

(a) *Citric acid,* solid
(b) *Ammonium hydroxide solution*, sp. Gr. 0.88 or diluted as required
(c) *Potassium cyanide solution*, 10% (w/v)
(d) Dithizone (dipheyl thiocarbazone solution, 0.1% chloroform, freshly prepared
(e) *Dilute hydrochloric acid*, approximately 0.1M (0.1N)
(f) Standard lead solution Two reference solutions of lead nitrate are required in this test as given below:

(i) Standard strong lead solution, obtained by dissolving 0.160 g of lead nitrate [Pb $(NO_3)_2$] in 50 ml of dilute nitric acid and making up to volume in a 100 ml graduated flask.

(ii) Standard dilute teat/solution, freshly prepared before the test by diluting 1 ml of the standard strong lead solution to 100 ml with water in a graduated flask.

(g) *Ammonium acetate*, solid
(h) *Sodium sulphide solution*, 10% (w/v)

K.2 PROCEDURE

K.2.1 Weigh accurately 50 g to 100 g of the sample, transfer to a 500 ml Kjeldahl digestion flask and wet with 25 ml to 50 ml. of concentrated nitric acid. Add 10 ml to 20 ml of concentrated H_2SO_2 and heat cautiously. Add concentrated HNO_3 dropwise from time to time from a pipette to speed up the oxidation of the material. Note the total amount of concentrated H_2SO_2 and HNO_3 added. When the oxidation is complete and the solution is colourless, add 20 ml of water and again boil to fuming. Cool and dilute with water to 50 ml in a graduated flask.

K.2.2 Take a suitable aliquot of the solution **K.2.1** add 2 g of citric acid and just neutralize with ammonia. Add 1 ml of the potassium cyanide solution and transfer the whole to a separating funnel. Extract the liquid with the dithizone solution. Carry out 3 extractions, using 10 ml for the first extraction and 5 ml each for the subsequent extractions. If the last extraction gives any indication of a reddish tinge, extract again to ensure complete removal of lead.

K.2.3 Take 10 ml of water in another separating funnel and wash each extract with this water. If suspended matter is present in the chloroform extract, this shall be filtered before passing to the separating funnel containing the 10 ml of wash-water. Transfer the combined chloroform extracts to a separating funnel and extract lead by shaking successively with 50, 20 and 10 ml of dilute hydrochloric acid. Combine the acid extracts in a separating funnel, wash once or twice with 10 ml of chloroform and filter through a previously wetted filter

paper into a 100 ml graduated flask. Make up the volume of the filtrate to 100 ml with dilute hydrochloric acid and use this as the test solution.

K.2.4 Estimate colorimetrically the lead present by comparison with the standard dilute lead solution containing 0.000 0l g of lead per ml (using not more than 10 ml of the standard solution for matching) in the following manner:

(a) Transfer a suitable volume of the test solution to a Nessler cylinder.

(b) Add 2 g of ammonium acetate, followed by ammonia until just alkaline and then 1 ml of potassium cyanide solution.

(c) Dilute to 50 ml, add 2 drops of sodium sulphide solution and match the colour against a set of standards prepared in the same way.

K.2.5 A blank determination shall be run under the same conditions, on the same reagents and by the same person but without using the material.

APPENDIX L
SAMPLING PLAN FOR SALT

L.1 SCALE OF SAMPLING

L.1.1 The number of packages to be selected from the lot or batch shall depend upon the size of the lot and shall be in accordance with table 1. This plan was taken from ISO 2859 using Inspection Level S_2

Table 1

Sampling size

Lot or batch size	**Number of packages to be selected**
1 to 25	2
26 to 150	3
151 to 1 200	5
1 201 to 35 00	8
over 35 000	13

L.1.2 These packages shall be selected at random from the lot; and to ensure randomness of selection, a random number table may be used.

L.2 PREPARATION OF TEST SAMPLES

From each package selected in accordance with **L.1.1**, a portion of the material (dependent on the number of tests to be done) shall be drawn with a suitable instrument. The portions shall be mixed thoroughly to form a composite sample. This composite sample shall then be divided into the required number of test samples.

L.3 CRITERIA FOR CONFORMITY

The lot shall be considered as conforming to this standard if test results satisfy the detailed requirements set out in paragraph 3.

APPENDIX M

VOLUMETRIC CONVERSION TABLE (AS THEY OCCUR IN SEQUENCE)

0.1N	Silver nitrate ($AgNO_3$)	=	0.1M $AgNO_3$
0.1N	Hydrochloric acid (HCI)	=	0.1M HC1
0.5N	Sulphuric acid (H_2SO_4)	=	0.25M H_2SO_4
0.1N	Potassium hydroxide (KOH)	=	0.1M KOH
0.2N	Sodium acetate (CH_2 COONa)	=	0.2M CH_2COONa
0.1N	Sodium thiosulphate ($Na_2S_4O_3$)	=	0.05M $Na_2S_2O_3$