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Draft Sri Lanka Standard
SPECIFICATION FOR SODIUM HYPOCHLORITE USED FOR TREATMENT OF WATER INTENDED
FOR HUMAN CONSUMPTION
(DSLS :)

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Draft Sri Lanka Standard
SPECIFICATION FOR SODIUM HYPOCHLORITE USED FOR TREATMENT OF
WATER INTENDED FOR HUMAN CONSUMPTION

DSLS.....:

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Draft Sri Lanka Standard
SPECIFICATION FOR SODIUM HYPOCHLORITE USED FOR TREATMENT OF
WATER INTENDED FOR HUMAN CONSUMPTION

FOREWORD

This Sri Lanka Standard was approved by the Sectoral Committee on Chemical and Polymer Technology and was authorized for adoption and publication as a Sri Lanka Standard by the Council of Sri Lanka Standards Institution on

Sodium hypochlorite is a chemical compound and it is commonly known as liquid bleach, soda bleach or Sodium oxychloride. The product is supplied as an aqueous solution.

The stability of hypochlorite solution is greatly affected by heat, light, pH and presence of heavy metals (which may be present in caustic soda). The optimum stability is attained at a pH close to 11 and with the heavy metals content not more than 5 ppm. Storage temperatures should not exceed 32 °C, above which the rate of decomposition becomes too high and the available chlorine content is rapidly depleted. At temperatures above 38 °C, Sodium chlorate formation becomes appreciable resulting in loss of Sodium hypochlorite.

Sodium hypochlorite solution is used in textile and paper bleaching, laundry trade, sterilization of swimming pools, disinfection of drinking water, treatment of cyanide wastes of electroplating industry and treatment of sewage effluent. It is also used as a sanitizer and deodorizer for floors, sinks, and toilets, as a therapeutic adjunct in the treatment of certain skin diseases and as a safe antiseptic. Sodium hypochlorite is also used in domesticity, but this Standard is applicable only for Sodium hypochlorite used in water treatment plants.

All standard values given in this Specification are in SI units.

For the purpose of deciding whether a particular requirement of this Specification is complied with, the final value observed or calculated, expressing the result of a test or an analysis, shall be rounded off in accordance with **SLS 102**. The number of significant places retained in the rounded off value shall be the same as that of the specified value in this Specification.

In the preparation of this Specification, the assistance obtained from the following publications is gratefully acknowledged:

EN	901: 2013	Chemicals used for treatment of water intended for human consumption- Sodium hypochlorite
IS	11673 : Part 1: 2019	Sodium hypochlorite solution- Specification Part 1 Household and industrial use (Second Revision)
IS	11673 : Part 2: 2019	Sodium hypochlorite solution- Specification Part 2 Water treatment use (Second Revision)

1 SCOPE

This Specification prescribes the requirements and methods of sampling and tests for Sodium hypochlorite solution, used in treatment of water intended for human consumption.

2 REFERENCES

- ISO 3696** Water for analytical laboratory use - Specification and test methods
ISO 5667-3 Water quality- Sampling- Part 3: Preservation and handling of water samples
ISO 8288 Water quality- Determination of Cobolt, Nickel, Copper, Zinc, Cadmium and Lead- Flame atomic absorption spectrometric methods
ISO 12846 Water quality – Determination of mercury – Method using atomic absorption spectrometry (AAS) with and without enrichment
SLS 102 Rules for rounding off numerical values
SLS 428 Random sampling methods

3 DEFINITION

3.1 available Chlorine: The measure of the oxidizing power of the chlorine present as hypochlorite. It is expressed in terms of chlorine with a gram equivalent mass of 35.46.

3.2 water intended for human consumption: all water either in its original state or after treatment, intended for drinking, cooking, food preparation, regardless of its origin and whether it is supplied from a distribution network, from a tanker, or in bottles or containers

4 REQUIREMENTS

4.1 General requirements

4.1.1 Sodium hypochlorite solution shall be a pale, yellowish green, clear liquid and shall not produce any sludge or turbidity when mixed with water.

4.1.2 The material shall comply with the minimum available chlorine content for not less than 30 days from the date of packing.

4.1.3 Sodium hypochlorite solution shall also conform to the requirements given in Table 1 when tested in accordance with the relevant methods given in Column (4) of the table.

TABLE 1- Requirements for Sodium hypochlorite solution

Sl. No.	Characteristic	Requirement	Method of test
(1)	(2)	(3)	(4)
i)	Available Chlorine (as Cl ₂), per cent, mass by volume	4.0 - 15.0	Appendix B
ii)	pH at 27 ± 2 °C	9 - 12	Appendix C
iii)	Relative density (at 25 °/ 25 °C)	1.07 - 1.20	Appendix D
iv)	Free alkali (as NaOH), g/ l, max.	1.0	Appendix E
v)	Free Sodium carbonate (as Na ₂ CO ₃), g/ l, max.	0.5	Appendix F
vi)	Iron (as Fe), ppm, max.	0.4	Appendix G
vii)	Sodium chlorate (as NaClO ₃), g/ l, max.	0.2	Appendix H
viii)	Cadmium (as Cd) ppm, max.	2.5	Method A of ISO 8288
ix)	Lead (as Pb) ppm, max.	10	Method A of ISO 8288
x)	Chromium (as Cr). ppm, max.	2.5	Appendix J
xi)	Arsenic (as As) ppm, max.	1	Appendix K
xii)	Antimony (as Sb) ppm, max.	20	Appendix K
xiii)	Selenium (as Se) ppm, max.	10	Appendix K
xiv)	Mercury (as Hg) ppm, max.	1	Appendix M

5 PACKAGING

The material shall be packed in air-tight plastic containers or as agreed between the purchaser and the supplier. The containers used shall be dry and free from grease, dirt or other foreign matter likely to cause decomposition of the material.

6 MARKING

Each package shall be marked legibly and indelibly with the following information:

- a) Name of the product;
- b) The manufacturer's and supplier's or distributor's name and address including country of origin;
- c) Registered trade mark, if any;
- d) Date of manufacture and best before;
- e) Available Chlorine content;
- f) Suitable for water purification for drinking purpose;
- g) Net content, in kilograms (kg) or litres (l);
- h) Batch or code number;
- j) Instructions for use;
- k) Storage instructions with words ("store in a cool place" and "keep away from direct sunlight and heat"); and
- m) Safety instructions.

7 METHODS OF TEST

7.1 Tests shall be carried out as prescribed in Appendices **B** to **M** of this Specification and **ISO 8288**.

7.2 During the analysis, unless otherwise stated, use only reagents of analytical grade and distilled water. For metal analysis, deionized water shall be used.

9 SAMPLING

Representative samples of the product for ascertaining conformity to the requirements of this Specification shall be drawn as prescribed in Appendix **A**.

APPENDIX A COMPLIANCE OF A LOT

The sampling scheme given in Appendix A shall be applied where compliance of a lot to the requirements of this Specification is to be assessed based on statistical sampling and inspection.

Where compliance with this Specification is to be assured based on manufacturer's control systems coupled with type testing and check tests or any other procedure, appropriate schemes of sampling and inspection should be adopted.

A.1 LOT

In any consignment, all the packages and containers of the same size and belonging to one batch of manufacture or supply shall constitute a lot.

A.2 GENERAL REQUIREMENTS OF SAMPLING

In drawing, handling, and preparing samples, the following precautions shall be observed.

A.2.1 Sampling shall be carried out by a trained and experienced person as it is essential that the sample should be representative of the lot to be examined.

A.2.2 The sampling equipment shall be clean and dry when used.

A.2.3 The samples shall not be taken from broken or opened packages.

A.2.4 The samples shall be placed in clean, dry and air-tight glass or suitable containers while drawing and handling the samples.

A.2.5 Samples shall be drawn from a protected place not exposed to air, light or heat.

A.2.6 The sample containers shall be air-tight after filling and marked with necessary details of sampling.

A.2.7 The sample containers shall be filled only up to 80% of the full volume leaving provision for gas exchange.

A.3 SAMPLING EQUIPMENTS

A.3.1 Sampling tube, liquid sampler or other effective liquid sampling device shall be used for sampling liquids in drums and cans. Sampling equipment shall be made of either stainless steel or other suitable material.

A.3.2 The appropriate equipment shall be designed so that it will reach to about 3 mm of the bottom of the container and have a capacity of approximately 0.5 litre or 1.0 litre.

A.4 SCALE OF SAMPLING

A.4.1 Samples shall be tested from each lot for ascertaining its conformity of the material to the requirements of this Specification.

A.4.2 The number of packages to be selected from a lot shall be in accordance with Table 2.

TABLE 2 – Scale of sampling

Number of packages in the lot (1)	Number of packages to be selected (2)
Up to 150	3
151 to 1 200	5
1 201 to 35 000	8
35 001 and above	13

NOTE

Minimum total sample volume shall be 1 000 ml.

A.4.3 The packages shall be selected at random. In order to ensure randomness of selection, random number tables as given in **SLS 428** shall be used.

A.4.4 The sample obtained from each package shall be thoroughly mixed to make the composite sample and reduced to obtain a test sample.

A.5 NUMBER OF TESTS

A.5.1 Each package selected as in **A.4.2** shall be inspected for packaging and marking requirements given in Clause 5 and 6. This shall be done at the place of sampling.

A.5.2 Sample selected as in **A.4.4** shall be examined for each relevant requirement given in Clause 4.

A.6 CRITERIA FOR CONFORMITY

A lot shall be declared as conforming to the requirements of this Specification, if the following conditions are satisfied.

A.6.1 Each package inspected for packaging and marking as in **A.5.1** satisfies the relevant requirements.

A.6.2 The test results on sample tested as in **A.5.2** satisfy the relevant requirements.

APPENDIX B DETERMINATION OF AVAILABLE CHLORINE CONTENT

B.1 PRINCIPLE

The sample is added to an acidified solution of Potassium iodide and the liberated iodine is titrated with standard Sodium thiosulphate solution to the usual starch end point.

B.2 REAGENTS

B.2.1 Glacial Acetic acid

B.2.2 Standard Potassium iodate solution, 0.1 M

B.2.3 Starch indicator solution, 0.5 per cent

Mix 0.5 g of soluble starch with 5 ml of cold water and add 95 ml of boiling water. Mix, cool and store in a glass bottle. Replace frequently or add 0.1 per cent salicylic acid to the starch solution to minimize deterioration.

B.2.4 Potassium iodide, iodate-free

B.2.5 Standard Sodium thiosulphate solution (Hypo), 0.1 M

Dissolve approximately 25 g of Sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) crystals in freshly boiled and cooled water, and dilute to 1 000 ml.

NOTE

The solution is more stable if the glassware is cleaned with Sulphuric or Chromic acids and thoroughly rinsed with water.

B.2.5.1 Standardization of Sodium thiosulphate solution

Weigh accurately 3.567 g of dry Potassium iodate (KIO_3) and transfer to a 1 000 ml volumetric flask. Dissolve in water, mix thoroughly and make up to the mark. This solution will be exactly 0.1 M. To standardize the Sodium thiosulphate solution, carefully pipette out a 50 ml aliquot of the Potassium iodate solution into a 250-ml stoppered conical flask and dilute to 100 ml with water. Add 1 g of Potassium iodide crystals. When it is dissolved, add 15 ml of 0.1 M Hydrochloric acid and titrate immediately with the Sodium thiosulphate solution. When the solution becomes light yellow, add 1 ml of starch indicator solution and complete the titration to the disappearance of the blue colour. Standardize at least monthly. Calculate the molality of the Sodium thiosulphate solution as follows:

$$\text{Molality} = \frac{50 \times 0.1}{A}$$

where,

A is the volume, in milliliters, of standard Sodium thiosulphate solution required for titration.

B.3 PROCEDURE

B.3.1 Preparation of sample solution

B.3.1.1 Strong solutions of Sodium hypochlorite sample shall be accurately diluted and aliquots taken for determination of available Chlorine, free Sodium carbonate and Sodium chlorate. The size of aliquots shall be such that approximately 40 ml of the 0.1 M reagent is required.

B.3.1.2 Dissolve 2 to 3 g of Potassium iodide crystals in 50 ml of water in a 250 ml conical flask. Add 10 ml of glacial Acetic acid, then pipette out the aliquot of sample into the solution, keeping the tip of the pipette beneath the surface of the solution until drained. Titrate at once with 0.1 M standard Sodium thiosulphate solution until the iodine colour is nearly gone (pale yellow), then add 1 ml of starch indicator solution and complete the titration to the disappearance of the blue colour.

B.4 CALCULATION

B.4.1 Available Chlorine (as Cl_2), per cent mass by volume = $\frac{(V_1 \times M \times 0.03546) \times 100}{V_2}$

where,

V_1 is the volume, in milliliters, of standard Sodium thiosulphate solution required for titration of the sample;

M is the molality, of the standard Sodium thiosulphate solution; and

V_2 is the volume, in milliliters, of original sample in aliquot used.

APPENDIX C DETERMINATION OF pH

C.1 PROCEDURE

Weigh 2.0 ± 0.1 g of the liquid material of Sodium hypochlorite and dissolve in 100 ml of freshly boiled and cooled distilled water. Measure the pH of the solution at 27 ± 2 °C by a suitable pH meter, using a glass electrode.

APPENDIX D DETERMINATION OF RELATIVE DENSITY

D.1 PRINCIPLE

Relative density shall be determined by a capillary-stoppered relative density bottle, but subject to agreement between the purchaser and the supplier, a Twaddell or Baume hydrometer may also be used.

D.2 APPARATUS

D.2.1 Density Bottle, 25 ml capacity

D.2.2 Weighing Balance, least count 0.1 mg

D.2.3 Water Bath

D.3 PROCEDURE

D.3.1 Clean the capillary-stoppered relative density bottle with a saturated solution of Chromic acid in concentrated Sulphuric acid followed by washing with distilled water and finally by alcohol. Dry the bottle in hot air oven, cool it to room temperature and weigh. Fill the weighed bottle with distilled water and place it in the constant temperature bath maintained at 25.0 ± 0.5 °C for an hour. Then insert the capillary stopper which has also been brought to 25.0 ± 0.5 °C. Wipe excess liquid from the top of the stopper, remove the bottle from the bath, wipe the outside to dryness, bring to room temperature and weigh. The difference between the mass of the filled and empty bottle gives the water equivalent, that is, the mass in air of the water contained in the bottle at 25.0 ± 0.5 °C.

D.3.2 Empty the bottle, rinse several times with alcohol and finally with ether. Remove the ether fumes with the aid of an air blast and permit the bottle to dry thoroughly in hot air oven and cool to room temperature. Fill the bottle with the material, warming both the material and bottle to assist filling and removal of air bubbles, and then bring the bottle and contents to 25.0 ± 0.5 °C. When the temperature is constant, insert the capillary stopper which has also been brought to 25.0 ± 0.5 °C and remove excess liquid from the top of the stopper. Remove the bottle from the bath, wipe the outside dry, bring to room temperature and weigh.

D.4 CALCULATION

$$\text{Relative density (at } 25^\circ / 25^\circ \text{C)} = \frac{M_3 - M_1}{M_2 - M_1}$$

where,

M_1 is the mass in g, of the empty relative density bottle;

M_2 is the mass in g, of water at 25 °C and the relative density bottle; and

M_3 is the mass in g, of sodium hypochlorite solution at 25 °C and the relative density bottle.

APPENDIX E DETERMINATION OF FREE ALKALI

E.1 PRINCIPLE

The sample is added to a neutralized, mixed solution of Barium chloride and Hydrogen peroxide, which precipitates any Carbonate and reduces the Hypochlorite to Chloride. The free alkali is then titrated with standard Hydrochloric acid using Phenolphthalein indicator.

E.2 REAGENTS

E.2.1 Barium chloride solution, 10 per cent (m/ v)

Dissolve 100 g of Barium chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in water and dilute to 1 000 ml. Filter, if the solution is turbid.

E.2.2 Standard Hydrochloric acid, 0.1 M

E.2.3 Hydrogen peroxide solution, 3 per cent (v/ v)

E.2.4 Phenolphthalein indicator solution, 0.5 per cent (m/ v)

Dissolve 0.5 g of phenolphthalein in 60 ml of 95 per cent Ethyl alcohol and dilute to 100 ml with water.

E.2.5 Sodium hydroxide solution, 0.4 per cent (m/ v)

E.3 PROCEDURE

Place 50 ml of Barium chloride solution and 30 ml of Hydrogen peroxide solution in a 250 ml conical flask, add 10 drops of Phenolphthalein indicator solution and neutralize with Sodium hydroxide solution. Introduce into this neutral mixture 10 ml of the liquid sample solution, shake or stir vigorously for 1 min, and titrate Sodium hydroxide solution with 0.1 M Hydrochloric acid until the pink colour disappears.

E.4 CALCULATION

$$\text{Free alkali (NaOH), g/l} = \frac{V_1 \times M \times 40}{V_2}$$

where,

V_1 is the volume, in milliliters, of standard Hydrochloric acid solution required for titration of sample;

M is the molality, of standard Hydrochloric acid; and

V_2 is the volume, in milliliters, of original sample solution in aliquot used.

APPENDIX F DETERMINATION OF FREE SODIUM CARBONATE

F.1 PRINCIPLE

For determining free Sodium carbonate, a number of determinations like total alkalinity, free Sodium hydroxide and Sodium bicarbonate are required. Finally, free Sodium carbonate is estimated from the data obtained.

F.2 TOTAL ALKALINITY (AS SODIUM MONOXIDE)

F.2.1 Reagents

F.2.1.1 *Standard Hydrochloric acid, 0.1 M*

F.2.1.2 *Dilute Hydrogen peroxide solution, 10 percent (v/ v)*

F.2.1.3 *Standard Sodium hydroxide solution, 0.1 M*

F.2.1.4 *Mixed indicator, Mixture of 3 parts of 2 per cent of Methyl red in alcohol and 1 part of 0.1 per cent of Bromocresol green in alcohol.*

F.2.2 Procedure

Use a volume of sample solution (see **B.3.1.1**) to require at least 10 ml of 0.1 M standard Hydrochloric acid for titration. Add the sample to three times its volume of Hydrogen peroxide solution, previously neutralized with 0.1 M Sodium hydroxide solution, using methyl red-bromocresol mixed indicator solution. Add a few drops more of the indicator solution and titrate to the end point with 0.1 M Hydrochloric acid.

F.2.3 Calculation

$$\text{Total alkalinity (as Sodium monoxide), g/l} = \frac{V_1 \times M \times 31}{V_2}$$

where,

V_1 is the volume, in milliliters, of standard Hydrochloric acid;

M is the molality of Hydrochloric acid; and

V_2 is the volume, in milliliters, of original sample solution aliquot used.

F.3 FREE SODIUM HYDROXIDE

F.3.1 Reagents

F.3.1.1 *Dilute Barium chloride solution, 10 per cent (m/ v)*

F.3.1.2 *Hydrogen peroxide solution, 10 per cent (v/ v)*

F.3.2 Apparatus

F.3.2.1 pH Meter

F.3.3 Procedure

Place 50 ml of Barium chloride solution and 30 ml of Hydrogen peroxide solution in a 250 ml beaker and, using a pH meter, titrate the solution with 0.1 M Sodium hydroxide to bring the pH to 7.5. Introduce into this solution 10 ml of the sample solution (see **B.3.1.1**), stir vigorously for 1 min, and titrate with 0.1 M Hydrochloric acid to a pH of 7.5 with continuous stirring.

F.3.4 Calculation

$$\text{Sodium hydroxide (as NaOH), g/l} = \frac{V_1 \times M \times 40}{V_2}$$

where,

V_1 is the volume, in milliliters, of standard Hydrochloric acid consumed in titration;

M is the molality of Hydrochloric acid; and

V_2 is the volume, in milliliters, of original sample solution aliquot used.

F.4 SODIUM BICARBONATE

If free Sodium hydroxide is not found, Sodium bicarbonate may be present and can be determined as follows.

F.4.1 Pipette out volume of 0.1 M Sodium hydroxide solution equal to the volume of 0.1 M Hydrochloric acid required for the determination of total alkalinity (see **F.2.2** and **F.3.3**), in a 250 ml conical flask and add the same volume of sample solution as used for determination of total alkalinity (see **F.2.2**). In a 250-ml beaker, place 50 ml of Barium chloride solution, 30 ml of Hydrogen peroxide, and 10 drops of Phenolphthalein indicator solution, neutralize with 0.1 M Sodium hydroxide solution. Add this neutralized solution to the prepared sample solution and shake vigorously for 1 min. Titrate the excess Sodium hydroxide with 0.1 M Hydrochloric acid to the disappearance of the pink colour.

F.4.2 CALCULATION

$$\text{Sodium bicarbonate (as NaHCO}_3\text{) g/l} = \frac{V_1 M_1 \times V_2 M_2 \times 84}{V_3}$$

where,

V_1 is the volume, in milliliters, of Sodium hydroxide solution;

M_1 is the normality of Sodium hydroxide solution;

V_2 is the volume, in milliliters, of Hydrochloric acid consumed in titration;

M_2 is the normality of Hydrochloric acid; and

V_3 is the volume, in milliliters, of original sample solution aliquot used.

F.5 FREE SODIUM CARBONATE

F.5.1 If Sodium hydroxide is present, calculate free Sodium carbonate as follows:

$$\text{Free Sodium carbonate} = [\text{Total alkalinity} - 0.775 (\text{Sodium hydroxide})] \times 1.709$$

(as Na_2CO_3), g/l

F.5.2 If Sodium bicarbonate is present, calculate free Sodium carbonate as follows:

$$\text{Free Sodium carbonate} = [\text{Total alkalinity} - 0.369 (\text{Sodium bicarbonate})] \times 1.709$$

(as Na_2CO_3), g/l

APPENDIX G DETERMINATION OF IRON

G.1 APPARATUS

G.1.1 Nessler Cylinders, 50 ml capacity

G.2 REAGENTS

G.2.1 Ammonium persulphate

G.2.2 Butanolic potassium thiocyanate solution

Dissolve 10 g of Potassium thiocyanate in 10 ml of water. Add sufficient n- butanol to make up to 100 ml and shake vigorously till the solution is clear.

G.2.3 Standard Iron solution A

Dissolve 0.7022 g of Ferrous ammonium sulphate $[(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}]$ in 100 ml of water, add 5 ml of 1:5 (v/ v) Sulphuric acid and add dilute solution of Potassium permanganate (0.2 per cent, m/ v) drop wise until a slight pink coloration remains after stirring. Dilute with water to 1 000 ml and mix thoroughly. One millilitre of this solution contains 0.1 mg of iron as Fe.

G.2.4 Standard Iron solution B

Take 100 ml of the standard Iron solution A (see **G.2.3**) and dilute to 1 000 ml with water in a 1 000 ml volumetric flask. This dilute solution should be prepared fresh. One millilitre of this solution contains 0.01 mg of Iron (as Fe).

G.3 PROCEDURE

G.3.1 Weigh 50.0 g of the material in a silica dish (capacity, 200 ml) and evaporate it almost to dryness over water bath. Dissolve the dry mass in 30 ml water, transfer quantitatively in a nessler cylinder of 50 ml capacity, add about 30 mg of Ammonium persulphate and 15 ml of Butanolic potassium thiocyanate solution. Make up to 50 ml, shake vigorously for about 30 s and allow the layers to separate. Carry out a control test in another Nessler cylinder using 2 ml of standard Iron solution B (see **G.2.4**). Compare the intensity of the colour produced in the Butanol layers in the two cylinders.

G.3.2 The limit prescribed 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 produced in the control test.

APPENDIX H DETERMINATION OF SODIUM CHLORATE

H.1 PRINCIPLE

Sodium chlorate is reduced with Sodium bromide in 8 M Hydrochloric acid. After dilution and addition of Potassium iodide, the released Iodine (equivalent to the hypochlorite plus chlorate) is titrated with standard Sodium thiosulphate solution and starch indicator.

H.2 APPARATUS

The apparatus (see Figure 1) consists of 1 000 ml wide-mouthed reaction bottle (A), fitted with a double hole rubber stopper carrying a separating funnel B, conveniently graduated or marked at the 10, 20 and 100 ml levels, and a delivery tube leading to a 50 ml test tube gas trap C, which is fitted with rubber tubing and a glass mouth piece, D.

H.3 REAGENTS

H.3.1 Concentrated Hydrochloric acid

H.3.2 Sodium bromide solution, 10 per cent (m/ v)

H.3.3 Potassium Iodide solution, 10 per cent (m/ v)

Prepare a 10 per cent solution of Potassium iodide (KI). Decolourize with $\text{Na}_2\text{S}_2\text{O}_3$, when necessary.

H.3.4 Standard Sodium thiosulphate solution, 0.1 M (see B.2.5)

H.3.5 Starch indicator solution, 0.5 per cent (m/ v)

H.4 PROCEDURE

H.4.1 Pipette an aliquot of the sample [same amount as used for available chlorine determination (see B.3.1.1)] into the reaction vessel. Assemble the apparatus and put 25 ml of Potassium iodide solution in the gas trap. Close the funnel stopcock. Pour 20 ml of Sodium bromide solution into the funnel. Open the stopcock, and with gentle suction on the mouthpiece, draw the Sodium bromide solution into the sample. Close the stopcock and pour 100 ml of Hydrochloric acid into the funnel. Open the stopcock and allow the acid to drain into the sample. Draw in the last drops with the suction. Close the stopcock. Swirl the vessel to mix the acid, and let stand exactly

for 5 min (use time clock). There will be a tendency for a vacuum to form and draw Potassium iodide solution from the trap back into the sample. This must be avoided by filling the funnel with water and relieving the vacuum by opening the stopcock and adding a small amount of water.

H.4.2 After 5 min open the stopcock and allow the water to drain into the sample swirling to dilute the acid. Add water through the funnel sufficient to dilute the sample to about 700 ml. Close the stopcock, and add 10 ml of Potassium iodide solution to the funnel. Apply pressure at the mouthpiece to blow the contents of the trap back into the vessel, opening the stopcock to allow the necessary amount of gas to escape through the funnel. Rinse the trap twice with water each time blowing the contents into the vessel as above. Finally allow the contents of the funnel to drain into the vessel. Rinse down the funnel and stopper and thoroughly mix the contents of the vessel. Titrate at once with 0.1 M Sodium thiosulphate solution till the colour of the solution becomes pale yellow, add 5 ml of starch indicator solution and complete the titration to the disappearance of the blue colour.

H.5 CALCULATION

$$\text{Sodium chlorate (NaClO}_3\text{), g/l} = \frac{(V_2 - V_1) \times M \times 17.74}{V_3}$$

where,

V_1 is the volume, in milliliters, of Sodium thiosulphate solution required for titration for available chlorine (see **B.3.1.2**);

V_2 is the volume, in milliliters, of Sodium thiosulphate solution required for titration for Sodium chlorate;

M is the molality of the Sodium thiosulphate solution; and

V_3 is the volume, in milliliters of original sample in aliquot used (see **B.3.1.1**).

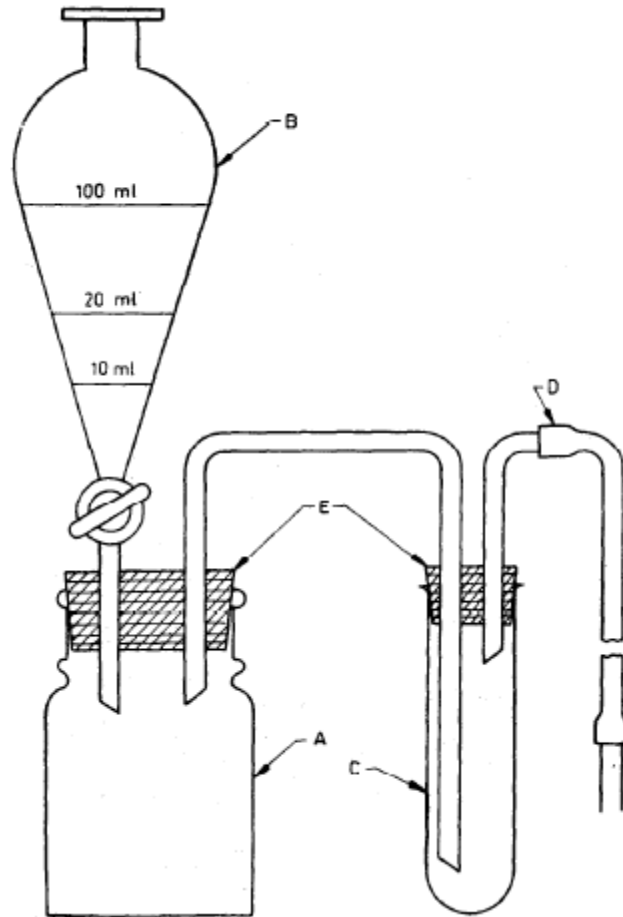


Figure 1- Apparatus for determination of Sodium chlorate

APPENDIX J DETERMINATION OF CHROMIUM

Two methods have been prescribed for the determination of Chromium. The method prescribed in **J.1** shall be the reference method and shall be carried out in case of any dispute.

J.1 METHOD 1

Inductively coupled plasma mass spectrometry (ICP-MS) methodology shall be used for determination of Chromium.

J.2 METHOD 2

Atomic Absorption Spectrometry methodology shall be used for determination of Chromium.

J.2.1 REAGENTS

J.2.1.1 *Hydrochloric acid, $\rho = 1,18$ g/ ml*

J.2.1.2 *Nitric acid, $\rho = 1,42$ g/ ml*

J.2.1.3 *Nitric acid, $c(\text{HNO}_3) = 1.5$ mol/ l*

J.2.1.4 *Hydrogen peroxide, 30 percent (m/ m) solution*

J.2.1.5 *Lanthanum chloride, solution, $\rho(\text{La}) = 20$ g/ l*

Weigh about 23.5 g of Lanthanum oxide in 1 000 ml volumetric flask and dissolve it in 200 ml of Hydrochloric acid (see J.2.1.1) and dilute to the 1 000 ml mark with water and mix.

NOTE

Appropriate precautions shall be taken when preparing this solution because the reaction of Lanthanum oxide with Hydrochloric acid is strongly exothermic.

J.2.1.6 Chromium, stock solution, $\rho(\text{Cr}) = 1\ 000$ g/ l

J.2.2 APPARATUS

J.2.2.1 Atomic absorption spectrometer, equipped with a chromium hollow cathode lamp and a Nitrous oxide/ Acetylene burner, and operated in accordance with the manufacturer's instructions, it is essential that the manufacturer's safety recommendations are strictly observed when using the Nitrous oxide/ Acetylene flame.

J.2.2.2 **Membrane filters**, of nominal pore diameter 0.45 μm , washed thoroughly with Nitric acid (see K.2.1.3) and rinsed with water.

J.2.3 PROCEDURE

J.2.3.1 *Sampling and preparation of test portions*

Collect and preserve samples according to **ISO 5667-3**.

J.2.3.2 *Acid soluble Chromium*

Treat the samples immediately after collection, by addition of sufficient Nitric acid (see J.2.1.3) to adjust the pH to between 1 and 2. Take 90 ml of the acidified sample, or another suitable aliquot in accordance with the expected concentration and add 1 ml of Hydrogen peroxide

(see **J.2.1.4**) and 2 ml of Nitric acid (see **J.2.1.2**). Boil and evaporate to a volume of approximately 50 ml. The sample shall not be reduced to dryness. In the case of samples with low chromium concentrations this digestion method should be carried out in an autoclave or a microwave oven. Add 10 ml of Nitric acid (see **J.2.1.2**) to the evaporated solution. Transfer the solution to a 100 ml volumetric flask. Add 10 ml of Lanthanum chloride solution (see **J.2.1.5**) into the flask, dilute to the mark with water and mix.

J.2.3.3 *Water soluble chromium*

After collection filter the sample through a membrane filter (see **J.2.2.2**) and acidify the filtrate immediately with Nitric acid (see **J.2.1.3**) to give a pH between 1 and 2. Add 10 ml of Lanthanum chloride solution (see **J.2.1.5**) to a 100 ml volumetric flask and make up to the mark with the acidified filtrate, or use another suitable aliquot in accordance with the expected concentration, and mix.

J.2.3.4 *Calibration*

Set up the instrument in accordance to the manufacturer's instructions (at wavelength $A = 357.9$ nm) using a Nitrous oxide/ Acetylene flame. Prepare 5 calibration solutions and aspirate a calibration solution and optimize the aspiration, the burner height and the flame conditions. Adjust the response of the instrument to zero absorbance with water. In turn, aspirate the set of 5 calibration solutions and the blank solution. Plot a graph having the Chromium concentrations of the calibration solutions in milligrams per litre as abscissa and the corresponding absorbance values as ordinate. Alternatively, data processing facilities can be used to equate the concentration against the absorbance.

J.2.3.5 *Test portion measurement*

Aspirate the prepared test portion (see **J.2.3.2** or **J.2.3.3**) into the flame and measure the absorbance for chromium. After each measurement aspirate water and readjust the zero, if necessary. Determine the concentration of Chromium corresponding to the absorbances of the test portion and of the blank solution.

APPENDIX K

DETERMINATION OF ARSENIC (As), ANTIMONY (Sb) AND SELINIUM (Se)

Two methods have been prescribed for the determination of Arsenic, Antimony and Selenium. The method prescribed in **K.1** shall be the reference method and shall be carried out in case of any dispute.

K.1 **METHOD 1**

Inductively coupled plasma mass spectrometry (ICP-MS) methodology shall be used for determination of Arsenic, Antimony and Selenium.

K.2 METHOD 2

Atomic Absorption Spectrometry methodology shall be used for determination of Arsenic (As), Antimony (Sb) and Selenium (Se).

K.2.1 REAGENTS

All reagents shall be of an analytical grade and the water used shall conform to grade 3 in accordance with ISO 3696.

K.2.1.1 *Freshly prepared Sodium tetrahydroborate (Sodium borohydride)*

Dissolve 8 g of NaBH₄ in 200 ml of NaOH, c(NaOH) = 0.1 mol/l

K.2.1.2 *Freshly prepared Sodium iodide, prereductant solution*

Dissolve 50 g of NaI in 500 ml of water.

K.2.1.3 *Sulphuric acid solution, c(H₂SO₄) = 9 mol/l*

K.2.1.4 *Nitric acid, density = 1.42 g/ml*

K.2.1.5 *Perchloric acid, density = 1.66 g/ml*

K.2.1.6 *Hydrochloric acid, density = 1.16 g/ml*

K.2.1.7 *Argon (or Nitrogen), commercial grade*

K.2.1.8 *Hydrogen, commercial grade*

K.2.1.9 *Arsenic(III) solutions*

K.2.1.9.1 Stock Arsenic(III) solution

Dissolve 1.320 g of Arsenic trioxide (As₂O₃) in water containing 4 g of NaOH. Transfer quantitatively to 1 000 ml one-mark volumetric flask and make up to the mark with water and mix.

K.2.1.9.2 Intermediate As(III) solution

Pipette out 10 ml of stock As(III) solution in a 1 000-ml volumetric flask and add 5 ml of Hydrochloric acid (see K.2.1.6) and dilute to 1 000 ml mark with water and mix.

K.2.1.9.3 Standard As(III) solution

Pipette out 10 mL of intermediate As(III) solution in a 1 000-ml volumetric flask and add 2 ml to 5 ml of Nitric acid (see **K.2.1.4**) and dilute to 1 000 ml mark with water and mix. Prepare diluted solutions daily.

K.2.1.10 *Arsenic(V) solutions*

K.2.1.10.1 Stock Arsenic(V) solution

Dissolve 1.534 g of Arsenic pentoxide, As_2O_5 , in water containing 4 g of NaOH. Transfer quantitatively to a 1 000 ml one-mark volumetric flask and make up to the mark with water and mix.

K.2.1.10.2 Intermediate As(V) solution

Pipette out 10 ml of stock As(V) solution in a 1 000-ml volumetric flask and add 5 ml of Hydrochloric acid (see **K.2.1.6**) and dilute to 1 000 ml mark with water and mix.

K.2.1.10.3 Standard As (V) solution

Pipette out 10 ml of intermediate As(III) solution in a 1 000-ml volumetric flask and add 2 ml to 5 ml of Nitric acid (see **K.2.1.4**) and dilute to 1 000 ml mark with water and mix. Prepare diluted solutions daily.

K.2.1.11 *Selenium(IV) solutions*

K.2.1.11.1 Stock Se(IV) solution

Dissolve 2.190 g of Sodium selenite, Na_2SeO_3 in water containing 10 ml of Hydrochloric acid (see **K.2.1.6**) and transfer quantitatively to a 1 000 ml one-mark volumetric flask and make up to the mark with water and mix.

K.2.1.11.2 Intermediate Se(IV) solution

Pipette out 10 ml of stock Se(IV) solution in a 1 000-ml volumetric flask and add 10 ml of Hydrochloric acid (see **K.2.1.6**) and dilute to 1 000 ml mark with water and mix.

K.2.1.11.3 Standard Se (IV) solution

Pipette out 10 mL of intermediate Se (IV) solution in a 1000-ml volumetric flask and add 2 ml to 5 ml of Nitric acid (see **K.2.1.4**) and dilute to 1000 ml mark with water and mix. Prepare the solution daily.

K.2.1.12 *Selenium(VI) solutions*

K.2.1.12.1 Stock Se(VI) solution

Dissolve 2.393 g of Sodium selenate Na_2SeO_4 in water containing 10 ml of Nitric acid (see **K.2.1.4**). Transfer quantitatively to a 1 000 ml one-mark volumetric flask and make up to the mark with water and mix. 1.00 ml solution contains 1.00 mg Se(VI)

K.2.1.12.2 Intermediate Se(VI) solution

Pipette out 10 ml of stock Se(VI) solution in a 1 000-ml volumetric flask and add 10 ml of Hydrochloric acid (see **K.2.1.6**) and dilute to the 1 000 ml mark with water and mix.

K.2.1.12.3 Standard Se(VI) solution

Pipette out 10 ml of intermediate Se(VI) solution in a 1 000-ml volumetric flask and add 2 ml to 5 ml of Nitric acid (see **K.2.1.4**) and dilute it into the mark and mix. Prepare the solution daily.

K.2.1.13 *Antimony solutions*

K.2.1.13.1 Stock Antimony solution

Dry 2 g of Potassium antimonyl tartrate hemihydrate (Antimony potassium tartrate) ($\text{C}_4\text{H}_4\text{O}_7\text{SbK}\cdot 0.5\text{H}_2\text{O}$) at 100 °C for 1 h. Dissolve 1.669 g in water, transfer quantitatively to a 1 000 ml one-mark volumetric flask and make up to the mark with water and mix.

K.2.1.13.2 Intermediate Antimony solution

Pipette out 10 ml of stock Antimony solution in a 1 000-ml volumetric flask and add 10 ml of Hydrochloric acid (see **K.2.1.6**) and dilute to the 1 000 ml mark with water and mix.

K.2.1.13.3 Standard Antimony solution

Pipette out 10 ml of intermediate Antimony solution in a 1 000-ml volumetric flask and add 2 ml to 5 ml of Nitric acid (see **K.2.1.4**) and dilute to the 1 000 ml mark with water and mix. Prepare the solution daily.

K.2.2 APPARATUS

K.2.2.1 *Atomic absorption spectrometer*, equipped with gas flow meters for Argon (or Nitrogen) and Hydrogen, Arsenic, Antimony and Selenium electrodeless discharge lamps with background correction at measurement wavelengths and an appropriate strip-chart recorder.

NOTE

Certain atomic absorption atomisers and hydride reaction cells are available commercially for use with the Sodium borohydride reagent.

K.2.2.2 *Atomiser*

K.2.2.3 *Reaction cell for producing Arsenic, Antimony or Selenium hydrides*

K.2.2.4 *Dropper and syringe*, capable of delivering 0.5 ml to 3.0 ml Sodium borohydride reagent.

K.2.3 PROCEDURE

K.2.3.1 Preparation of the apparatus

Connect the inlet of the reaction cell with the auxiliary purging gas controlled by a flow meter. If a drying cell between the reaction cell and atomiser is necessary, use only anhydrous CaCl_2 and not CaSO_4 as it can retain SeH_2 . Before using the hydride generation/ analysis system, optimise operating parameters. Aspirate the aqueous solutions of Arsenic, Antimony and Selenium directly into the flame to facilitate atomiser alignment. Align quartz atomisers for maximum absorbance. Establish a purging gas flow, concentration and rate of addition of Sodium borohydride reagent, solution volume, and stirring rate for optimum instrument response for the chemical species to be analyzed. If a quartz atomiser is used, optimise the cell temperature. If Sodium borohydride reagent is added too quickly, rapid evolution of Hydrogen will unbalance the system. If the volume of solution being purged is too large, the absorption signal will be decreased. Recommended wavelengths are 193.7 nm, 196.0 nm and 217.6 nm for Arsenic, Selenium and Antimony.

K.2.3.2 Preparation of test solutions and standard solutions

Add 50 ml of the sample or Arsenic(III), Selenium(VI) or Antimony standard solution to a 250 ml beaker. Alternatively, prepare standard solutions by adding aliquots of solutions containing 5 μg of Arsenic, Selenium or Antimony directly to the beaker and dilute to 50 ml in this beaker, achieving a concentration of 100 $\mu\text{g}/\text{l}$ of the respective solutions. Add 7 ml of Sulphuric acid $c(\text{H}_2\text{SO}_4) = 9 \text{ mol}/\text{l}$ (see **K.2.1.3**) and 5 ml of Nitric acid (see **K.2.1.4**). If necessary add a small boiling chip or glass beads. Evaporate to SO_3 fumes. Maintain oxidising conditions at all times by adding small amount of Nitric acid, to prevent solution from darkening. Maintain an excess of Nitric acid until all organic matter is destroyed. Complete digestion is usually indicated by a light-coloured solution. Cool slightly, add 25 ml of water and 1 ml of Perchloric acid (see **K.2.1.5**) and again evaporate to SO_3 fumes to expel oxides of Nitrogen. Monitor effectiveness of digestion

procedure used by adding 5 ml of a standard Arsenic solution, 5 ml of a standard Selenium solution or 5 ml of a standard Antimony solution to 50 ml of the sample and measuring recovery. Alternatively, use 100 ml micro-Kjeldahl flasks for the digestion of total recoverable Arsenic, Selenium or Antimony, thereby improving digestion effectiveness. After final evaporation of SO_3 fumes, dilute to 50 ml for Arsenic measurements or 30 ml for Selenium and Antimony measurements.

K.2.3.3 Determination of Arsenic with Sodium borohydride

Add 5 ml of Hydrochloric acid (see **K.2.1.6**) to 50 ml of the digested standard solution or test solution in a 250 ml beaker and mix. Add 5 ml Sodium iodide prereductant solution (see **K.2.1.2**), mix and wait 30 min.

Attach one beaker at a first time to the rubber stopper containing the gas dispersion tube for the purging gas, the Sodium borohydride reagent inlet, and the outlet to the atomiser. Turn on a strip-chart recorder and wait until the base line is established by the purging gas and all air is expelled from the reaction cell. Add 0.5 ml of Sodium borohydride reagent (see **K.2.1.1**). After the instrument absorbance has reached a maximum and returned to the base line, remove the beaker, rinse the dispersion tube with water, and proceed to the next test solution or standard solution. Periodically compare the standard As(III) and As(V) curves for response consistency. Determine the concentration of Arsenic corresponding to the absorbances of the test portion and of the blank solution.

K.2.3.4 Determination of selenium with Sodium borohydride

Add 15 ml of Hydrochloric acid (see **K.2.1.6**) to 30 ml of the digested standard solution or test solution, or to 30 ml of the undigested standard, or the sample in a 250 ml beaker, and mix. Heat for a predetermined period at a temperature between 90 °C to 100 °C. Alternatively, autoclave at 121 °C in capped containers for 60 min, or heat for a predetermined time in open test tubes using a 90 °C to 100 °C hot water bath or an aluminium block digester. Check the effectiveness of the selected heating by demonstrating equal instrument responses for calibration curves prepared either from standard Se(IV) or from Se(VI) solutions. Effective heat exposure for converting Se(VI) to Se(IV), with no loss of Se(IV), ranges between 5 min to 60 min when open beakers or test tubes are used. Do not digest standard Se(IV) and Se(VI) solutions used for this check of equivalency. After prereduction of Se(VI) and Se(IV), attach beakers, one at a time, to the purge apparatus. For each, turn on the strip-chart recorder and wait until the base line is established. Add 0.50 ml Sodium borohydride reagent (see **K.2.1.1**). After the instrument absorbance has reached a maximum and returned to the base line, remove beaker, rinse dispersion tube with water and proceed to the next test solution or standard solution. Determine the concentration of Selenium corresponding to the absorbances of the test portion and of the blank solution.

K.2.3.5 Determination of Antimony with Sodium borohydride

To 30 ml of the digested standard solution or the test solution, or to 30 ml of the undigested standard solution, or the test solution in a 250 ml beaker, add 15 ml of Hydrochloric acid (see **K.2.1.6**) and mix. Heat for a predetermined period (between 5 min and 60 min) at a

temperature between 90 °C to 100 °C. After prereduction of Sb, attach beakers, one at a time, to the purge apparatus. For each, turn on the strip-chart recorder and wait until the base line is established. Add 0.50 ml Sodium borohydride reagent (see **K.2.1.1**). After the instrument absorbance has reached a maximum and returned to the base line, remove beaker, rinse dispersion tube with water and proceed to the next test solution or standard solution. Determine the concentration of Antimony corresponding to the absorbances of the test portion and of the blank solution.

APPENDIX M DETERMINATION OF MERCURY

Two methods have been prescribed for the determination of Arsenic, Antimony and Selenium. The method prescribed in **M.1** shall be the reference method and shall be carried out in case of any dispute.

M.1 METHOD 1

Inductively coupled plasma mass spectrometry (ICP-MS) methodology shall be used for determination of Mercury.

M.2 METHOD 2

Atomic Absorption Spectrometry methodology shall be used for determination of Mercury.

M.2.1 Reagents

All reagents shall be of analytical grade and the water used shall conform to the grade 3 as specified in **ISO 3696**.

M.2.1.1 *Nitric acid*, mass fraction 65 per cent

M.2.1.2 *Hydroxylammonium chloride solution* (NH₂OH.HCl), mass fraction 10 per cent

M.2.1.3 *Tin(II) chloride solution*, mass fraction 10 per cent

M.2.2 PROCEDURE

M.2.1 Test solution

Weigh about 10 g (m_s) of the sample solution and add approximately 70 ml of water and care should be taken to avoid sputtering. Quantitatively transfer the test solution to a gas washing flask, capacity 250-ml, with the gas inlet equipped with a porous glass frit. Dilute the contents of the gas washing flask with water to obtain a total volume of 100 ml. Transfer it to a volumetric flask (solution A). Pipette accurately 5 ml of the Sodium hypochlorite (solution A) and dilute in a 250 ml graduated gas washing flask. During gentle shaking, add 30 ml of Hydroxylammonium chloride solution (see **M.2.1.2**), 5 ml of Nitric acid (see **M.2.1.1**) and 2 ml of Tin (II) chloride solution

(see **M.2.1.3**) successively. Close immediately the gas washing flask with the gas inlet equipped with a porous glass frit.

M.2.2 Determination of Mercury content

Determine the Mercury content as per the method described in ISO **12846**.

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