
**Sodium hypochlorite solutions for household and industrial use —
Specification**

DRAFT AFRICAN STANDARD FOR COMMENTS ONLY



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Sodium hypochlorite solutions for household and industrial use — Specification

1 Scope

This Draft African Standard specifies the requirements, sampling and test methods of sodium hypochlorite solutions suitable for household and industrial use.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

acceptable

acceptable to the authority administering this standard, or to the parties concluding the purchase contract, as relevant

3.2

batch

material from a single mix or, in the case of a continuous production process, the material from a single day's production

3.3

gassing

gas evolution

3.4

nominal concentration

minimum sodium hypochlorite content of the solution under test, at the time of manufacture

3.5

product unit

a unit of the final product, packed in a plastic bottle or other suitable airtight and opaque container.

3.6

lot

a number of containers consisting of product of the same type and style, which have been manufactured and packed under essentially the same conditions.

4 Requirements

4.1 General

4.1.1 Sodium hypochlorite solutions shall be of one of the following nominal concentrations, as required (see Annex A):

- a) 150 g/L for industrial use;

b) 50 g/L for domestic use; or

c) 35 g/L for domestic use.

4.2 Sodium hypochlorite solution for industrial use

4.2.1 General

The solution shall be a clear liquid free from suspended or particulate matter and shall be miscible in all proportions with distilled water.

4.2.2 Physical and chemical requirements

The solution shall comply with the requirements given in Table 1.

Table 1 — Physical and chemical requirements for a sodium hypochlorite solution for industrial use

S/No	Property	Requirement	Test method
i.	Sediment content, % (mass fraction expressed as a percentage), max.	0.1	Annex A
ii.	Sodium hypochlorite content determined on the date of manufacture, g/L, min.	150	Annex B
iii.	Sodium hypochlorite content determined on the 14th day $\pm$ 2 d after date of manufacture, g/L, min.	130	Annex B
iv.	Sodium hydroxide content, g/L,	5-60	Annex C
v.	Gassing at 37 °C, cm ³ over a 20 h period, max.	35	Annex D
vi.	Iron content, mg/L, max.	2	Annex E
vii.	Cobalt content, mg/L, max.	5	
viii.	Copper content, mg/L, max.	5	
ix.	Nickel content, mg/L, max.	5	
x.	Free sodium carbonate (as Na ₂ CO ₃), g/L, Max	0.5	Annex F
xi.	Sodium chlorate (as NaClO ₃), g/l, Max	3	Annex G

4.2.3 Stability

The material of both the grades shall comply with the minimum available chlorine content for not less than 30 days from the date of packing. After a period of more than 30 days, the minimum available chlorine for both grades shall be as agreed to between the purchaser and the supplier.

NOTE 1. See Annex H for notes to users on the two main ways sodium hypochlorite solution decomposes.

4.3 Sodium hypochlorite solutions for domestic use

4.3.1 General

4.3.1.1 When so required, the solution shall contain laundry blue. The laundry blue might settle on standing but shall disperse completely in the solution when the solution, in the original container, is shaken for 30 s.

4.3.1.2 The solution shall be a clear liquid, and shall be free from sediment and suspended matter other than the required laundry blue. A solution shall be considered to be clear if all salts that have crystallized from the solution dissolve completely when the solution is mixed with twice its volume of distilled water.

4.3.2 Physical and chemical requirements

The solution shall comply with the requirements given in Table 2.

Table 2 — Physical and chemical requirements for a sodium hypochlorite solution for household use

Property	Requirement	Test method
Sediment content, % (mass fraction expressed as a percentage), max.	0.1	Annex A
Sodium hypochlorite content determined on the 14th day $\pm$ 2 d after date of manufacture, g/L, min. 5 % nominal concentration 3.5 % nominal concentration	50 35	Annex B
Sodium hypochlorite content determined on the 60th day $\pm$ 2 d after date of manufacture, g/L, min. 5 % nominal concentration 3.5 % nominal concentration	45 32	Annex B
Sodium hydroxide content, g/L, max.	15	Annex C
Iron content, mg/L, max.	0.4	Annex E
Free sodium carbonate (as Na ₂ CO ₃), g/L, Max	0.5	Annex F
Sodium chlorate (as NaClO ₃), g/l, Max	0.2	Annex G

4.3.3 Stability

When (after receipt) the solution has been stored in the dark at a temperature of 20 °C to 25 °C, the sodium hypochlorite content, determined in accordance with Annex B on the 60th day $\pm$ 2 d after date of manufacture, shall be as follows:

- a) solutions of 5 % nominal concentration: not less than 45 g/L; and
- b) solutions of 3.5 % nominal concentration: not less than 32 g/L.

5 Inspection and test methods

5.1 General

Unless inconsistent with the text, all reagents used shall be of analytical reagent grade or the purest grade available, and all water shall be distilled or deionized water.

5.2 Inspection

Sample and inspect the containers taken in accordance with Annex G for compliance with all the relevant requirements of this standard for which tests to assess compliance are not given in Annex A to Annex J (inclusive).

6 Packing and marking

6.1 Packing

6.1.1 Sodium hypochlorite solutions shall be packed in containers of good fabrication and shall be so designed, constructed and closed as to prevent deformation and leakage of the contents due to vibration, stacking, impact or changes in environmental conditions, such as temperature, pressure or humidity that can be encountered during handling.

6.1.2 The containers and closing devices shall not be susceptible to adverse attack by the sodium hypochlorite solution, or be liable to form dangerous compounds with the contents.

6.1.3 The closing devices shall be strong and solid to ensure that they will not be worked loose during handling and that they will withstand the normal stress and strain of handling.

6.1.4 Sodium hypochlorite solutions for industrial use (except when delivered in intermediate bulk containers (IBCs) and tankers) shall be packed in containers that comply with the relevant East African Standards.

6.1.5 Only solutions from the same batch shall be packed in the same container and, when relevant, in the same pack.

6.2 Marking

Each container (other than an IBC and a tanker) shall bear the following information in prominent, legible and indelible marking:

- a) the manufacturer's name or trademark or both;
- b) the words "SODIUM HYPOCHLORITE";
- c) the nominal available sodium hypochlorite content;
- d) the batch identification (which may be given in code);
- e) the net volume of the contents;
- f) the words "STORE IN A COOL PLACE AND NOT IN DIRECT SUNLIGHT; Avoid contact with aluminium, zinc, tin and their alloys and DO NOT mix with acids";
- g) in the case of a sodium hypochlorite solution for industrial use, the date of manufacture, and the necessary caution labelling, "CORROSIVE";
- h) in the case of individual containers of sodium hypochlorite solutions for domestic use, the following additional information:
 - i. the instructions for use; and
 - ii. the words:
 - "DO NOT USE BLEACH ON WOOL, SILK, RAYON AND LEATHER";

NOTE Sodium hypochlorite can have a deleterious effect on certain resin-treated materials, such as crease-resistant, drip-dry, embossed and glazed fabrics. Coloured fabrics will lose their colour if their dyes are not colourfast to hypochlorites.

- "DO NOT USE WITH ANY OTHER TOILET OR HOUSEHOLD CLEANER"; and
- "DO NOT MIX WITH ACID"; and

- i) any additional information required in terms of the regulations promulgated under the current relevant national legislation (see foreword) and by the local transportation authority.

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Annex A

(normative)

Determination of Sediment content

A.1 Procedure

A.1.1 Mix the test specimen of sodium hypochlorite solution thoroughly and then accurately weigh out approximately 300 g into a 500 mL beaker.

A.1.2 Filter the solution through a Whatman GF/A glass-fibre filter paper that has been dried at $100\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ and tared, or equivalent.

A.1.3 Wash the beaker and the sediment five times with 20 mL portions of cold water and then dry the glass fibre filter paper with the sediment at $100\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ until a constant mass is attained.

A.2 Calculation

A.2.1 Calculate the sediment content, S , as a percentage, as follows:

$$S = \frac{m_2}{m_1} \times 100$$

where

m_2 is the mass of the sediment after it has been dried, in grams;

m_1 is the mass of test specimen taken, in grams.

A.2.2 Check for compliance with 4.2.2.

Annex B

(normative)

Determination of Sodium hypochlorite content

B.1 Reagents

B.1.1 Potassium iodide (KI).

B.1.2 Glacial acetic acid (CH₃COOH).

B.1.3 Sodium carbonate (Na₂CO₃).

B.1.4 Mercuric iodide (HgI₂).

B.1.5 Starch indicator solution

Prepare the starch solution immediately before use, as follows:

Make a paste of 1 g of soluble starch and a small amount of water. Pour the paste into 100 mL of boiling water, stirring it constantly. Boil for approximately 1 min and cool.

B.1.6 Standard potassium dichromate solution, $c(\text{K}_2\text{Cr}_2\text{O}_7) = 0,01667 \text{ mol/L}$

Dissolve exactly 4.904 g of potassium dichromate, previously dried at $105^\circ\text{C} \pm 2^\circ\text{C}$ for 2 h and cooled in a desiccator, in water and dilute to 1 000 mL in a volumetric flask.

B.1.7 Sodium thiosulfate solution ($\pm 0.1 \text{ mol/L}$).

B.1.7.1 Prepare a stock volumetric solution as follows:

B.1.7.1.1 Dissolve 25 g ± 1 g of sodium thiosulfate pentahydrate (NaS₂O₃·5H₂O) in freshly boiled and cooled water.

B.1.7.1.2 Add 0.1 g of sodium carbonate (see B.1.3) and 0.01 g of mercuric iodide (see B.1.4) and dissolve.

B.1.7.1.3 Dilute to 1 000 mL with freshly boiled and cooled water in a volumetric flask.

B.1.7.1.4 Transfer to an amber-coloured glass-stoppered bottle and store in the dark.

B.1.7.1.5 Standardize the solution before use (see B.1.7.2).

B.1.7.1.6 Discard the solution when it becomes turbid.

B.1.7.2 Standardize the sodium thiosulfate solution as follows:

B.1.7.2.1 Pipette 20.0 mL of the potassium dichromate solution (see B.1.6) into a glass-stoppered iodine flask.

B.1.7.2.2 Remove the stopper and quickly add 3 g of potassium iodide, 2 g of sodium bicarbonate and 5 mL of concentrated hydrochloric acid.

B.1.7.2.3 Insert the stopper gently in the flask, swirl to mix, and allow to stand in the dark for 10 min.

B.1.7.2.4 Rinse the stopper and the inner walls of the flask with water and titrate with the sodium thiosulfate volumetric solution (see B.1.7) until a pale straw colour is obtained.

B.1.7.2.5 Add 2 mL of the starch solution (see B.1.5) and continue the titration until a blue colour is discharged.

B.1.7.2.6 Carry out a blank determination by repeating the procedure but omitting the potassium dichromate.

B.1.7.2.7 Calculate the concentration, M , of the sodium thiosulfate solution, in moles per litre, as follows:

$$M = \frac{20.0 \times 0.01667 \times 6}{V}$$
$$= \frac{2.0}{V}$$

where

V is the volume of sodium thiosulfate solution used in the titration after the correction for the blank, in millilitres.

NOTE Alternative methods of standardization may be used provided that they give equivalent results.

B.2 Procedure

B.2.1 Pipette 25.0 mL of the sample into a 1 000 mL volumetric flask and dilute with water up to the mark.

B.2.2 Pipette 25.0 mL of the diluted sample into a 250 mL glass-stoppered iodine flask and add 50 mL of water.

B.2.3 Add 2 g of potassium iodide and 10 mL of glacial acetic acid. Insert the stopper and allow to stand in the dark for 10 mins.

B.2.4 Rinse the stopper and the inner walls of the flask with water.

B.2.5 Titrate the liberated iodine with the standardized sodium thiosulfate solution (see B.1.7) to a pale straw yellow colour.

B.2.6 Add 1 mL of starch indicator solution (see B.1.5) and titrate drop by drop until the faint blue colour changes to colourless, for at least 30 s.

B.2.7 Carry out a blank determination by repeating the procedure but omitting the sample.

B.3 Calculation

B.3.1 Calculate the sodium hypochlorite (NaOCl) content, C , in grams per litre, as follows:

$$C = V \times 59.56 \times M$$

where

V is the volume of sodium thiosulfate solution used in the titration after correction for the blank, in millilitres;

M is the molarity of sodium thiosulfate solution, in moles per litre.

B.3.2 Check for compliance with 4.2.2.

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Annex C (normative)

Determination of Sodium hydroxide content

C.1 Reagents

C.1.1 Barium chloride solution, 10 g/100 mL

Dissolve 100 g of barium chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in water and dilute to 1 L with water. Filter the solution if turbid.

C.1.2 Hydrogen peroxide solution

Approximately 3 % (10 volumes).

C.1.3 Sodium hydroxide solution, 0.4 g/100 mL

Dissolve 0.4 g of sodium hydroxide in water, cool and dilute to 100.0 mL in a volumetric flask. Store in a plastics container.

C.1.4 Phenolphthalein indicator solution

Dissolve 1 g of phenolphthalein in 100 mL of ethanol (95 %).

C.1.5 Screened methyl orange indicator solution

Dissolve 0.2 g of methyl orange and 0.28 g of xylene cyanol FF in 100 mL of ethanol (50 %).

C.1.6 Standard hydrochloric acid solution ($c(\text{HCl}) = 0.10 \text{ mol/L}$)

Dilute 8.9 mL of concentrated hydrochloric acid (d at $25^\circ\text{C} \geq 1.160$) with water. Standardize against sodium carbonate, using the screened methyl orange as indicator.

C.2 Procedure

C.2.1 Place 50 mL of the barium chloride solution and 40 mL of the hydrogen peroxide solution in a 250 mL conical flask.

C.2.2 Add 10 drops of the phenolphthalein indicator and then add the sodium hydroxide solution drop by drop until a permanent faint pink colour is obtained.

C.2.3 Immediately pipette 10 mL of the test specimen of sodium hypochlorite solution drop by drop into the flask, taking care that the effervescence does not become excessive.

C.2.4 When the effervescence subsides, swirl the flask vigorously for 1 min.

C.2.5 Add another drop of phenolphthalein indicator and rapidly titrate the solution with the standard hydrochloric acid solution (see C.1.6) until the pink colour first disappears.

C.2.6 Do not continue the titration if the pink colour re-appears on standing.

C.3 Calculation

C.3.1 Calculate the sodium hydroxide content, X , in grams per litre, as follows:

$$X = V \times M \times 4.0$$

where

V is the volume of hydrochloric acid solution used in the titration, in millilitres;

M is the concentration of the standard hydrochloric acid solution, in moles per litre.

C.3.2 Check for compliance with 4.2.2.

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Annex D (normative)

Determination of Gassing rate of sodium hypochlorite

D.1 Outline of method

The gas evolved from a weighed sample maintained at a controlled temperature is collected and measured volumetrically.

D.2 Apparatus (see Figure 1).

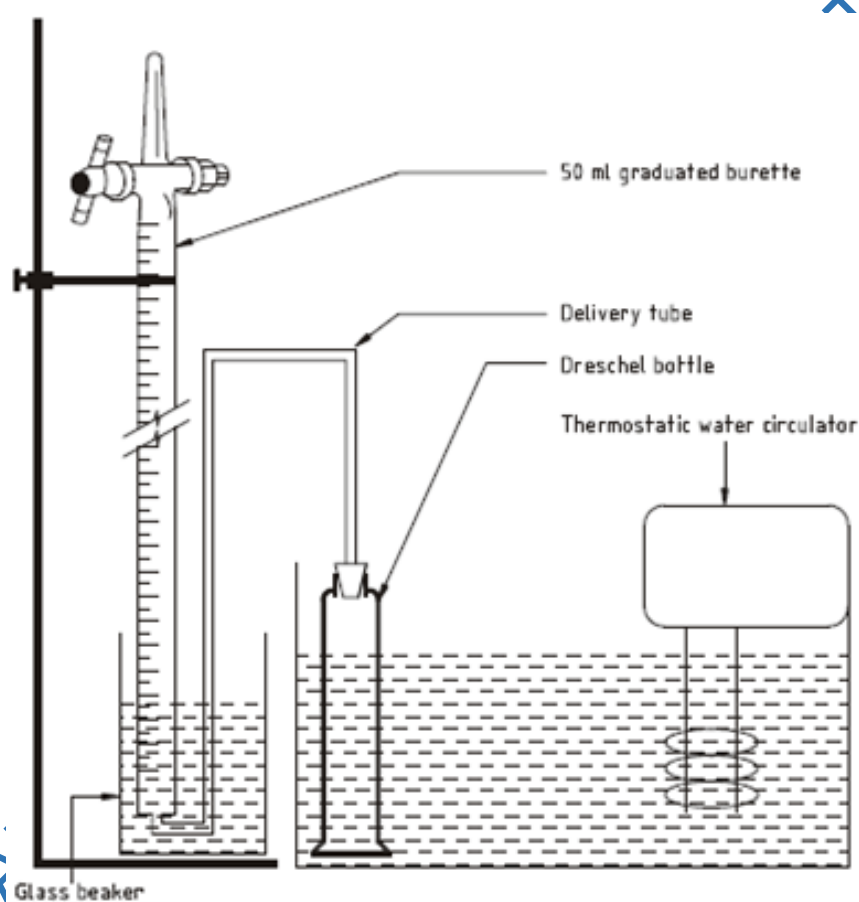


Figure 1 — Apparatus for measuring the gassing rate of sodium hypochlorite

- D.2.1 Water-bath and thermostatic water circulator, capable of maintaining a temperature of 37 °C.
- D.2.2 Dreschel bottle, of 250 mL and that is externally covered to exclude light.
- D.2.3 Graduated burette, of 50 mL capacity.
- D.2.4 Top-pan balance, capable of weighing to an accuracy of 0.1 g.
- D.2.5 Tallform beaker, of 500 mL capacity.
- D.2.6 Delivery tube, of glass connected to the Dreschel bottle.

D.3 Procedure

D.3.1 Weigh out $300 \text{ g} \pm 0.5 \text{ g}$ of the sample into the Dreschel bottle.

D.3.2 Place the Dreschel bottle in the water-bath at $37^\circ\text{C} \pm 1^\circ\text{C}$.

D.3.3 Fill the beaker with approximately 400 mL of water and immerse the end of the delivery tube in the beaker. Check that the water does not suck back beyond the second bend in the delivery tube

D.3.4 Completely fill the burette with water and invert in the beaker, ensuring that no air is trapped in the burette and that the end of the burette is well submerged in the water.

D.3.5 Secure the open end of the burette over the end of the delivery tube so that the gas evolved is trapped in the burette.

D.3.6 Use the burette tap to adjust the volume of water in the burette to the 50 mL mark.

D.3.7 Leave the sample in the water-bath for 1 h to equilibrate to the correct temperature.

D.3.8 Record the volume (V_1) of gas (mL) collected in the inverted burette.

D.3.9 Leave the sample in the water-bath for a further 20 h and then record the total volume (V_2) of gas (mL) collected in the inverted burette.

NOTE Take the reading in millilitres as equivalent to the volume of gas in cubic centimetres.

D.3.10 Calculate the gassing rate per 20 h (see D.4).

D.3.11 Repeat D.3.1 to D.3.10 (inclusive) at least once more.

D.4 Calculation

D.4.1 Calculate the gassing rate, GR , of the gas collected, in cubic centimetres per 20 h, as follows:

$$GR = V_2 - V_1$$

where

V_2 is the total volume of gas collected after 20 h, in cubic centimeters;

V_1 is the volume of gas collected after 1 h, in cubic centimeters.

D.4.2 Express the final results as an average of the calculated results for each determination.

D.4.3 Check for compliance with 4.2.2.

Annex E

(normative)

Determination of Iron content, cobalt content, copper content and nickel content

E.1 Apparatus

Atomic absorption spectrometer, capable of operating under the conditions given in Table E.1.

E.2 Reagents

E.2.1 Nitric acid solution (1:1)

Carefully add 100 mL of concentrated nitric acid (d at 25 °C ≥ 1.339) to 100 mL of water while mixing.

E.2.2 Hydrochloric acid

Concentrated hydrochloric acid (a mass fraction of 32 %: d at 25 °C ≥ 1.160).

E.2.3 Hydrochloric acid solution (1:1)

Carefully add 100 mL of concentrated hydrochloric acid (see E.2.2) to 100 mL of water while mixing.

E.2.4 Sodium chloride solution (30 g/L)

Dissolve 30.00 g of sodium chloride in 100 mL water in a 1 L volumetric flask, and make up to the mark with water.

E.3 Standard solutions

E.3.1 General

Prepare four standard solutions, as detailed in E.3.2 to E.3.5 (inclusive), that contain iron, cobalt, copper and nickel respectively, in a concentration of 1 000 mg/L.

E.3.2 Iron solution

Dissolve exactly 1.000 g of a mass fraction of 99.99 % metallic iron in 25 mL of 1:1 hydrochloric acid in a 150 mL tall form beaker. Leave the beaker covered with a watch glass in a water-bath until the metallic iron is completely dissolved. Transfer quantitatively to a 1 L volumetric flask, and make up to the mark with water.

E.3.3 Cobalt solution

Dissolve exactly 1.000 g of a mass fraction of 99.99 % metallic cobalt in a minimum volume of 1:1 nitric acid, transfer quantitatively to a 1 L volumetric flask, and make up to the mark with water.

E.3.4 Copper solution

Dissolve exactly 1.000 g of a mass fraction of 99.99 % metallic copper in a minimum volume of 1:1 nitric acid, transfer quantitatively to a 1 L volumetric flask, and make up to the mark with water.

E.3.5 Nickel solution

Dissolve exactly 1,000 g of a mass fraction of 99.99 % metallic nickel in 20 mL of 1:1 nitric acid, transfer quantitatively to a 1 L volumetric flask, and make up to the mark with water.

NOTE Commercially available standard solutions may also be used.

E.4 Procedure

E.4.1 Instrument operating conditions

Adjust the instrument controls to the settings given in table E.1, appropriate to the element being determined, using acetylene as the fuel for the burner, and air as the support.

Table E.1 — Instrument settings

Instrument setting	Setting ^a for the determination of			
	Iron	Cobalt	Copper	Nickel
Wavelength, nm	248.3	240.7	324.7	232.0
Slit width, nm	0.2	0.2	0.2	0.2
Lamp current, mA	5.0	7.0	3.5	3.5
Scale expansion	Up to × 10	Up to × 10	Up to × 10	Up to × 10
^a Small variations in the above settings might be necessary for different instruments.				

E.4.2 Calibration of instrument:

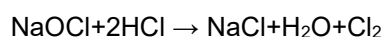
From the standard solutions (see E.3), prepare calibration solutions as follows:

- pipette 10 mL of 1 000 mg/L of iron, cobalt, copper and nickel into separate 100 mL volumetric flasks, make up to the mark with water and mark it 100 mg/L;
- prepare 1 mg/L, 5 mg/L and 10 mg/L standards by pipetting 1 mL, 5 mL and 10 mL of each of the 100 mg/L standards into separate 100 mL flasks;
- make up to the mark, using the 30 g/L sodium chloride solution to ensure a similar matrix to the test specimen of sodium hypochlorite solution;
- as a blank, use the same 30 g/L sodium chloride solution;
- aspirate each solution through the instrument and read its absorbance value at the specified settings (see Table E.1); and
- for each element, prepare a calibration curve by plotting the concentrations of the three standard solutions, in milligrams per litre, against their absorbance values minus the absorbance value of the blank.

E.4.3 Determination of iron content, cobalt content, copper content and nickel content

E.4.3.1 Pipette 10 mL of the test specimen of sodium hypochlorite solution into a 150 mL tallform beaker and slowly add 1 mL of concentrated hydrochloric acid while stirring. (The test specimen is treated with hydrochloric acid to remove the formed chlorine and to form sodium chloride in solution.)

E.4.3.2 Cover the beaker with a watch glass and allow the ensuing heat to complete the following exothermic reaction:



E.4.3.3 If the solution is not clear, add another 1 mL of concentrated hydrochloric acid and allow the reaction to continue; repeat the procedure until the solution is clear.

E.4.3.4 Allow the solution to cool and as soon it has lost all of its yellow colour and a crystalline precipitate has formed, transfer the contents quantitatively into a 100 mL volumetric flask and make up to the mark with water.

E.4.3.5 Aspirate the solution through the instrument and read its absorbance value for each element, at the specified settings (see Table E.1).

NOTE It is recommended that procedures be conducted in the following sequence:

- optimize instrument;
- read blank;
- read standards; and
- read test specimen.

E.5 Calculation

E.5.1 Calculate the concentration, Y , in milligrams per litre, of each of the four elements in the test specimen, using the following formula:

$$Y = \frac{100 \times C}{V}$$

where

C is the concentration of the element in the dilute test specimen of sodium hypochlorite solution (as read off from the appropriate calibration curve), in milligrams per litre;

V is the volume of test specimen used (see E.4.3.1).

E.5.2 Check for compliance with 4.2.2.

Annex F (normative)

Determination of free sodium carbonate

F.1 General

For determining free sodium carbonate, a number of determinations like total alkalinity, free sodium hydroxide and sodium bicarbonate are required. Finally, free 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 N.

F.2.1.2 Dilute Hydrogen Peroxide Solution — 10 percent (v/v).

F.2.1.3 Standard Sodium Hydroxide Solution — 0.1 N.

F.2.1.4 Mixed Indicator

Mixture of 3 parts of 2 percent of methyl red in alcohol and 1 part of 0.1 percent of bromocresol green in alcohol.

F.2.2 Procedure

Strong solutions of sodium hypochlorite sample shall be accurately diluted and aliquots taken for determination of free sodium carbonate (Annex F) and sodium chlorate (Annex G). The size of aliquots shall be such that approximately 40 ml of the 0.1 N reagent is required.

Use a volume of sample solution at least 10 ml of 0.1 N standard hydrochloric acid for titration. Add the sample to three times its volume of hydrogen peroxide solution, previously neutralized with 0.1 N 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 N hydrochloric acid.

F.2.3 Calculation

Total alkalinity (as sodium monoxide), g/l

$$= \frac{V_1 \times N \times 31}{V}$$

Where,

V_1 = volume in ml, of standard hydrochloric acid;

N = normality of hydrochloric acid; and

V = volume in ml, of original sample solution in aliquot used.

F.3 Free sodium hydroxide

F.3.1 Reagents

F.3.1.1 Dilute Barium Chloride Solution — 10 percent (m/v)

F.3.1.2 Hydrogen Peroxide Solution — 10 percent (v/v)

F.3.2 Apparatus

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 N sodium hydroxide to bring the pH to 7.5. Introduce into this solution 10 ml of the sample solution (see B-3.1), stir vigorously for 1 min, and titrate with 0.1 N hydrochloric acid to a pH of 7.5 with continuous stirring.

F.3.4 Calculation

Sodium hydroxide (as NaOH), g/l

$$= \frac{V_1 \times N \times 40}{V}$$

where,

V_1 = volume in ml, of standard hydrochloric acid consumed in titration;

N = normality of hydrochloric acid; and

V = volume in ml, of original sample solution in aliquot used.

F.4 Sodium bicarbonate

F.4.1 general

If no free sodium hydroxide is found, sodium bicarbonate may be present and can be determined as follows.

F.4.1 procedure

Pipette out volume of 0.1 N sodium hydroxide solution equal to the volume of 0.1 N hydrochloric acid required for the determination of total alkalinity (see F.2.2 and F.2.3), 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 N 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 N hydrochloric acid to the disappearance of the pink colour.

F.4.2 Calculation

Sodium bicarbonate (as NaHCO₃), g/l

$$= \frac{V_1 N_1 \times V_2 N_2 \times 84}{V}$$

where,

V_1 = volume in ml, of standard sodium hydroxide solution;

N_1 = normality of sodium hydroxide solution;

V_2 = volume in ml, of hydrochloric acid consumed in titration;

N_2 = normality of hydrochloric acid; and

V = volume in ml, of original sample solution in aliquot used.

F.5 Free sodium carbonate

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

Free sodium carbonate (as Na_2CO_3), g/l = [Total alkalinity — 0.775 (sodium hydroxide)] $\times$ 1.709

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

Free sodium carbonate (as Na_2CO_3), g/l = [Total alkalinity — 0.369 (sodium bicarbonate)] $\times$ 1.709.

Annex G (normative)

Determination of sodium chlorate content

G.1 General

Sodium chlorate is reduced with sodium bromide in 8 N 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.

G.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.

G.3 Reagents

G.3.1 Concentrated Hydrochloric Acid.

G.3.2 Sodium Bromide Solution — 10 percent (m/v).

G.3.3 Potassium Iodide Solution — 10 percent (m/v).

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

G.3.4 Standard Sodium Thiosulphate Solution — 0.1 N (see B.1.7)

G.3.5 Starch Indicator Solution — 0.5 percent (m/v).

G.4 Procedure

Strong solutions of sodium hypochlorite sample shall be accurately diluted and aliquots taken for determination of free sodium carbonate (Annex F) and sodium chlorate (Annex G). The size of aliquots shall be such that approximately 40 ml of the 0.1 N reagent is required.

Pipette an aliquot of the sample 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.

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 N 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.

G.5 Calculation

where,

A = volume in ml, of sodium thiosulphate solution required for titration for sodium hypochlorite (see B.2);

B = volume in ml, of sodium thiosulphate solution required for titration for sodium chlorate;

N = normality of the sodium thiosulphate solution; and

V = original sample in aliquot used (see G.4).

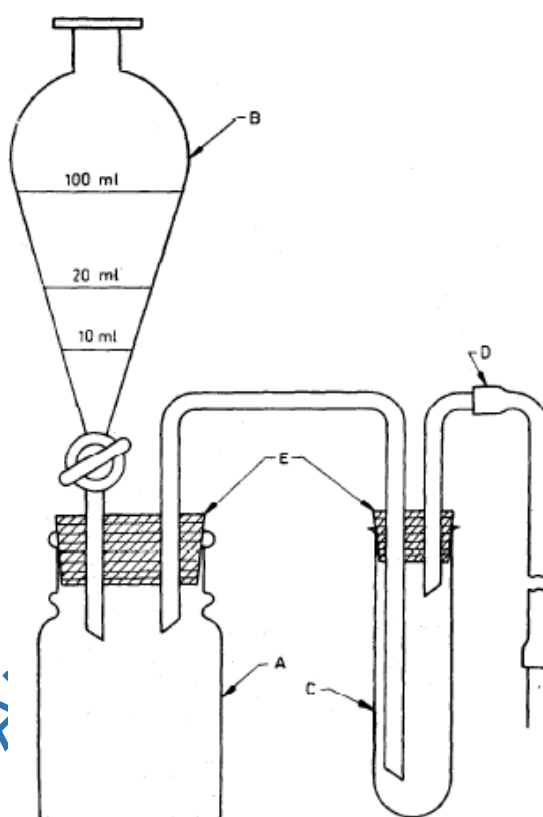


Figure 1: apparatus for determination of sodium chlorate

Annex H (informative)

User information

H.1 The decomposition of sodium hypochlorite takes place in two main ways:

- a) $3\text{NaOCl} \rightarrow 2\text{NaCl} + \text{NaClO}_3$; and
- b) $2\text{NaOCl} \rightarrow 2\text{NaCl} + \text{O}_2$.

Normally, in the dark, over 90 % of the decomposition follows the chlorate-forming mechanism (see (a)) and the remainder follows the oxygen-forming reaction (see (b)).

H.2 The following are some of the factors that increase the rate at which decomposition of the solution occurs:

- a) **concentration**: the rate of decomposition falls off rapidly as the solution loses strength;
- b) **temperature**: elevated temperatures greatly increase the rate of decomposition;
- c) **metallic impurities**: certain trace metallic impurities such as copper, nickel, cobalt and iron catalyse the oxygen-forming reaction;
- d) **exposure to light**: the rate of both the chlorate-forming reaction and the oxygen-forming reaction is increased by exposure to blue or ultraviolet light; and
- e) **pH value of solution**: sodium hypochlorite is stabilized by sodium hydroxide, and the pH value of the solution should exceed 11.

H.3 As a guide, the following Table H.1 provides a general guidance on the stability of the material:

Table H.1 — Sample sizes

Available chlorine Trade percentage	Chlorine concentration g/l	Half-life Days (at 25°C)
3	30	1,700
6	60	700
9	90	250
12	120	180
15	150	100
18	180	60

Annex I

(normative)

Sampling and compliance with this standard

I.1 Sampling

I.1.1 General

The following sampling procedure shall be applied in determining whether a lot submitted for inspection and testing, complies with the relevant requirements of this standard. The sample so drawn shall be deemed to represent the lot.

I.1.2 Definitions

I.1.2.1 defective

solution or a container that fails in one or more respects to comply with the relevant requirements of this standard

I.1.2.2 lot

not less than 25 containers and not more than 5 000 containers (or one tanker) of sodium hypochlorite solution bearing the same batch number, from one manufacturer, and submitted at any one time for inspection and testing

I.2 Sample for inspection

After inspecting the lot for compliance with clause 5, take, at random, the number of containers, as relevant, shown in column 2 of table I.1, relative to the appropriate lot size shown in column 1. Reserve half the containers for the determination of the characteristics other than stability, and the other half for the determination of stability (see Annex J).

Table I.1 — Sample sizes

Lot size number of containers	Sample for inspection number of containers
25 – 50	4
51 – 100	6
101 – 500	8
501 – 1 500	10
1 501 – 5 000	12

I.3 Sample for testing

I.3.1 After inspection of the sample taken in accordance with I.2 for compliance with clause 5.2,

- take, from four levels in the tanker or at four stages during the filling of the tanker, equal increments that make up a composite sample of about 4 L;
- thoroughly mix and divide this sample into two equal portions;

- c) place each portion in a 2 L amber bottle that is fitted with a ground-glass stopper, or any other suitable inert stopper.

I.3.2 Reserve half the containers for the determination of the characteristics other than stability, and the other half for the determination of stability (see Annex J).

I.4 Compliance with this standard

The lot shall be deemed to comply with the requirements of this standard if, after inspection and testing of the sample taken in accordance with I.1, I.2 and I.3, no defective is found.

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Annex J

(normative)

Determination of stability and other characteristics other than stability

J.1.1 Determination of characteristics other than stability

Carry out these determinations on the contents of each of the appropriate containers reserved in accordance with 1.2 for these determinations. Keep the containers closed until the test for sodium hypochlorite content is to be carried out.

J.1.2 Determination of the stability

Keeping the containers closed, store the appropriate containers (reserved in accordance with 1.2 for these determinations) in a dark place at a temperature of 20 °C to 25 °C until the tests for stability (see 4.2.3 or 4.3.3, as relevant) are to be carried out.

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