INTERNATIONAL STANDARD 2120

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Liquid chlorine for industrial use — Determination of the content of chlorine by volume in the vaporized product

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 2120 was drawn up by Technical Committee ISO/TC 47, *Chemistry.*

It was approved in March 1971 by the Member Bodies of the following countries :

Austria	India	South Africa, Rep. of
Belgium	Israel	Spain
Chile	Italy	Switzerland
Czechoslovakia	Netherlands	Thailand
France	New Zealand	Turkey
Germany	Poland	U.S.A.
Hungary	Portugal	U.S.S.R.

The Member Body of the following country expressed disapproval of the document on technical grounds :

United Kingdom

Liquid chlorine for industrial use — Determination of the content of chlorine by volume in the vaporized product

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the chlorine content by volume in liquid chlorine for industrial use, after vaporization of the product.

2 PRINCIPLE

Sampling of a known volume of chlorine (about 100 ml) obtained by gasification of liquid chlorine.

Absorption of the chlorine by a 2 % zinc amalgam in the presence of 1 ml of saturated sodium chloride solution.

Measurement of the residual, non-condensable gases such as H_2, O_2, N_2, CO, CO_2, etc.

Calculation of the volume of chlorine (difference from total volume), referred to 100 ml of vaporized sample.

3 REAGENTS

3.1 Zinc amalgam

WARNING: MERCURY AND ZINC AMALGAM SHOULD BE HANDLED TAKING ALL THE PRECAUTIONS MADE NECESSARY BY THE TOXICITY OF THESE PRODUCTS. IN PARTICULAR OPERATE IN A WELL-VENTILATED HOOD AND TAKE CARE TO AVOID ALL CONTACT WITH THE SKIN.

Place about 4 kg of pure mercury in a porcelain dish of 1 l capacity. Add about 80 g of granulated zinc and cover all with an approximately 0.01 N sulphuric acid solution.

Heat to 80 °C; keep the mixture at this temperature, stirring from time to time with a glass stirrer, until the zinc is completely amalgamated, and allow to cool.

Keep the amalgam under 0.01 N sulphuric acid, in order to avoid its oxidation by the air.

3.2 Sodium chloride solution, saturated at ambient temperature.

3.3 Sodium hydroxide, ρ 1.22 g/ml approximately, 20 % (m/m) solution, approximately.

3.4 Sulphuric acid, approximately 2 N solution.

3.5 Methanol, technical.

3.6 Chlorine-resistant grease

Greases based on fluorinated or chlorofluorinated products are suitable.

4 APPARATUS

The apparatus used is shown diagrammatically in the Figure.

It comprises the following components:

— **Gas burette** (A), of known volume (approximately 100 ml), the upper part under the stopcock consisting of 7 mm diameter tube of approximately 2 ml capacity graduated downwards in 0.05 ml.

— **Steel needle valve** (C)

— **Flask with side nozzle** (D), capacity 250 ml and containing 150 ml of zinc amalgam (3.1).

— **Upper reservoir** (E) of the gas burette.

— **Levelling bottle** (F), capacity 250 ml and containing 150 ml of zinc amalgam (3.1).

— **Bubbler** (G), containing water allowing the gas flow to be controlled and having dipping tubes with an internal diameter of 5 mm.

— **Safety bottle** (H).

— **Receiver** (K), capacity 2 l and containing 1 l of sodium hydroxide solution (3.3) to absorb the excess chlorine.

— **Filter** (L), consisting of a threaded steel tube having an inside diameter of approximately 6 mm, 150 mm long and filled with glass wool.

— **Glass tube** (M), having an inside diameter of approximately 6 mm.

— **T-bore stopcock** (R).

— **3 V-bore stopcocks** (R1, R2 and R4).

— **Straight bore stopcock** (R3).

— **Tilting stand** (S) for the chlorine bottle.

— **Draining vessel** (V).

— **Connections** (a, b, c, d).

— **Rubber tubes** (f).

— **Elastomer tubes** (g, g1, g2) resistant to chlorine.

— **Elastomer connectors** (h) resistant to chlorine.

5 PROCEDURE

SAFETY MEASURES: PERSONNEL WORKING WITH CHLORINE SHALL BE INSTRUCTED IN THE HAZARDS OF THE PRODUCT AND THE SAFETY MEASURES TO BE OBSERVED.

CHLORINE IS A VESICATORY, IRRITANT AND SUFFOCATING GAS. THE CHLORINE CONCENTRATION IN THE ATMOSPHERE SHALL NOT EXCEED 1 ppm (V/V), OR 3 mg/m^3.

FOR THESE REASONS IT IS RECOMMENDED THAT PROTECTIVE CLOTHING AND GOGGLES BE WORN AND THAT THE WORK PLACE BE SUITABLY VENTILATED, HAVING IN MIND THAT THE GAS, WHICH IS HEAVIER THAN AIR, COLLECTS IN LOW AREAS.

IN THE CASE OF A SIGNIFICANT LEAK, ONLY PERSONNEL WEARING APPROPRIATE MASKS SHALL REMAIN IN THE CONTAMINATED AREA. THE LEAK CAN BE LOCATED BY MEANS OF A RAG SOAKED IN AMMONIA.

PERSONS POISONED BY INHALING THE GAS SHALL BE TAKEN FROM THE CONTAMINATED ZONE AS QUICKLY AS POSSIBLE, AVOIDING ALL MUSCULAR EFFORT. THEY SHALL BE KEPT QUIET, AWAY FROM COLD, AND FIRST AID SHALL BE APPLIED AS NECESSARY, WHILE AWAITING THE ARRIVAL OF THE DOCTOR. IF BREATHING CEASES, IMMEDIATELY APPLY ARTIFICIAL RESPIRATION.

5.1 Determination

Place the bottle containing the sample of liquid chlorine to be analysed on a tilting stand as shown in the Figure, with the opening downward, so that the chlorine is taken in a liquid state and vaporizes immediately thereafter.

Connect the various parts of the apparatus after having cleaned and dried them, and coat the taps R, R1, R2, R3 and R4 with a thin layer of the grease (3.6).

Close R1, R2 and R4. Place 150 ml of the amalgam (3.1) in each of the levelling bottles D and F. Allow the amalgam to flow from F towards b in the burette A. For this purpose, turn R1 in order to link these two vessels, remove the ground stopper from the container E and turn R2 in order to link this container with the burette.

By a repeated up and down movement of the levelling bottle F, completely remove any air that may be trapped in the tube between F and the burette A, as well as in the connection b. Then lower F so that a little amalgam still remains in the burette.

Then turn R1 so as to link the burette A with the connection a, turn R to link the connection a to the small draining vessel V, and turn R2 so as to make the amalgam contained in D flow through the tube f until the connection c is filled. Then turn R2 so as to link the burette A with its upper container E. The amalgam still remaining in the burette then flows into the small draining vessel V through the connection a and the tap R.

Turn the latter immediately in order to link the stopcock R4 with the connection a of the burette, insert the ground stopper in the container E and adjust R3 so as to link the safety bottle H with the receiver K containing the sodium hydroxide solution (3.3). Then turn R4 to link the needle valve C with the bubbler G through the tube g2.

Open the main valve on the bottle containing the sample then, with caution, the valve C and pass the chlorine to be analysed into the apparatus at a speed of 5 bubbles per second, counted upon passing into the bubbler bottle G. Then turn R4 in order to allow chlorine to pass into the burette A, at the same speed, for 15 min, and turn R4 back to its preceding position.

Then close the two stopcocks R1 and R2 of the burette A and the valve of the bottle containing the chlorine. Wait until the bubbling ceases in G, then close the valve C and the stopcock R3. Remove the ground stopper from the container E then turn the stopcocks R and R4 so as to link the tube g2 with the draining vessel V (to the open air).

Wait 10 minutes to allow the chlorine contained in the burette A, cooled by the expansion, to reach room temperature.

Place a few millilitres of the sodium chloride solution (3.2) in the container E, then open R2 and close it immediately, so that there is communication for a brief moment between A and E through the connection d, which enables atmospheric pressure to be re-established in the burette A, by allowing the small quantity of chlorine in excess to escape.

Open R2 again and this time allow about 1 ml of the sodium chloride solution (3.2) to enter the burette, whilst creating a slight reduction of pressure through the connection b, with the aid of the levelling bottle F, and opening R1. The sodium chloride solution ensures the dissolution of the zinc chloride resulting from the action of the chlorine on the amalgam.

Then turn R2 so as to link the burette A with the bottle D containing the zinc amalgam (3.1). While the latter flows slowly downward into the burette, raise the levelling bottle F progressively so that the vacuum produced in the burette does not become too great.

When all the chlorine has been absorbed, close R2, keep open R1, place the bottle F near the burette A and lower it in order to create a difference of about 20 cm between the levels of liquids in the two containers for degassing the sodium chloride solution. After 3 min raise the bottle F slowly so that the amalgam is at the same level in the two containers and take a reading on the burette of the volume of the non-absorbed gas, using the meniscus of the sodium chloride solution.

5.2 Preparation of apparatus for the next determination

Turn R2 so as to link the burette A with the bottle D and transfer to it the amalgam, up to the starting level, by raising the bottle F. Then turn this tap to link the burette to the container E through the connection d, remove the ground glass stopper from this container and make the rest of the amalgam pass from the burette into the bottle F by lowering the latter. Avoid allowing air to enter the connection b, and turn R1 towards the connection a and R towards the small draining vessel V.

Then clean the burette, through the container E, in turn with the sulphuric acid solution (3.4), distilled water and methanol (3.5). Dry it in a current of air.

6 EXPRESSION OF RESULTS

Chlorine content of the vaporized sample, expressed as a percentage by volume, is given by the formula :

$$\frac{V_1 - V_2}{V_1} \times 100$$

where

V_1 is the volume, in millilitres, of the burette A;

V_2 is the volume, in millilitres, of residual gas read during the test.

7 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional.

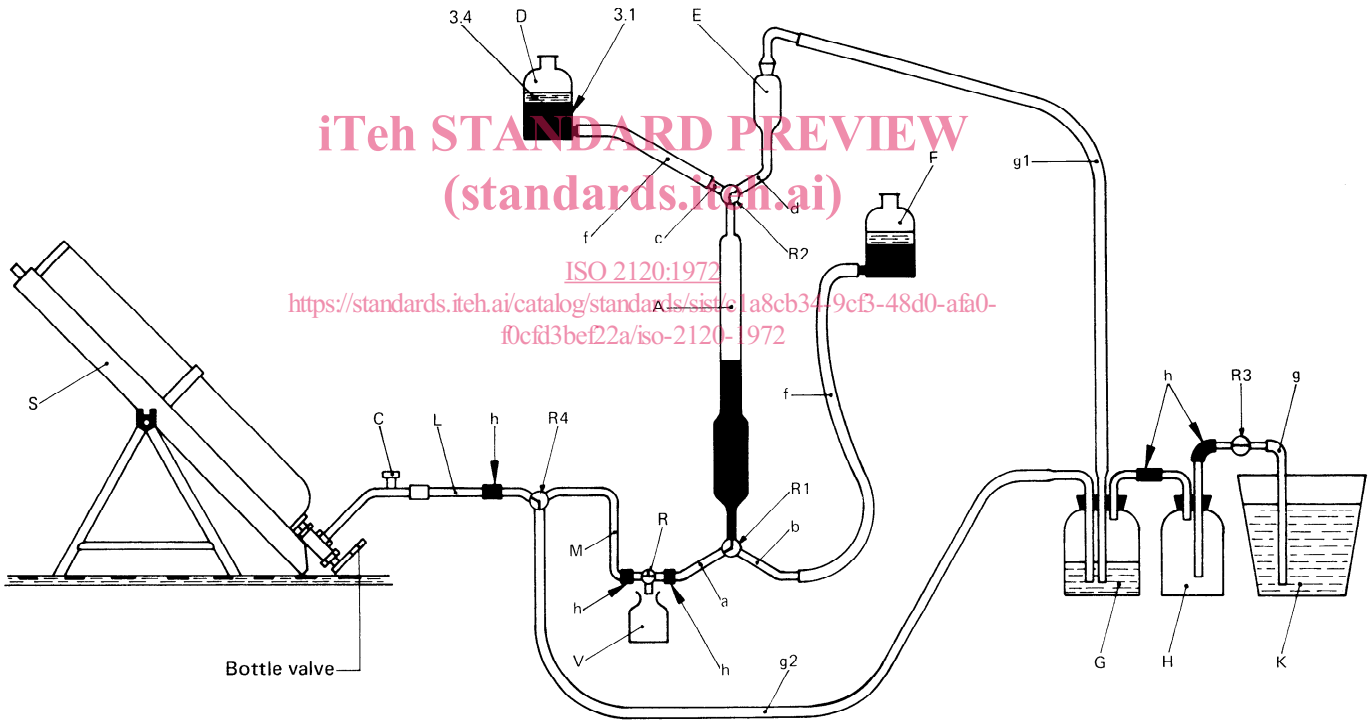

FIGURE — **Diagram of the apparatus for the determination of the chlorine content by volume**

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