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INTERNATIONAL STANDARD ISO 2121

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Liquid chlorine for industrial use — Determination of water content — Gravimetric method

First edition — 1972-07-01

UDC 661.41 : 546.212

Ref. No. ISO 2121-1972 (E)

Descriptors : chemical analysis, chlorine, liquefied gases, moisture content, gravimetric analysis.

Price based on 6 pages

FOREWORD

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International Standard ISO 2121 was drawn up by Technical Committee ISO/TC 47, *Chemistry.*

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

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Printed in Switzerland

Liquid chlorine for industrial use — Determination of water content — Gravimetric method

1 SCOPE

This International Standard specifies a gravimetric method for the determination of the "volatile water", i.e. that which volatilizes at the same time as the chlorine, originating from the gasification of liquid chlorine for industrial use.

2 FIELD OF APPLICATION

This method is applicable to products the water content of which is greater than 5 ppm (0.000 5 % m/m).

NOTE — This method should not be applied to the analysis of samples stored in bottles subject to corrosion. The determination of water would then be falsified by the presence of ferric chloride, which is hygroscopic.

3 PRINCIPLE

Absorption of the water by passing the gasified sample into tared absorbers containing phosphorus pentoxide or magnesium perchlorate.

Elimination of the gaseous chlorine and volatile impurities, other than water, also present, by flushing the absorbers with air or dry nitrogen heated to 80 °C.

Absorption of the chlorine leaving the absorbers in a tared bottle containing sodium hydroxide solution..

Weighing of the absorbers and the flask containing the sodium hydroxide solution to determine respectively, by difference from the original masses, the mass of water found and that of the sample taken initially.

4 REAGENTS

4.1 Absorbent

Choose one of the following two reagents :

4.1.1 *Phosphorus pentoxide,* or

4.1.2 *Magnesium perchlorate,* in the form of tablets or granules of 3 to 5 mm, in order to avoid loss of mass of material during the absorption of water. This reagent should not contain more than 2 molecules of water of hydration (i.e. 13.9 %), this being confirmed by the method given in the Annex.

Do not use this reagent if the chlorine to be analysed contains organic matter.

4.2 Air or nitrogen, dry.

It is convenient to dry these gases by passing them through a tower containing the absorbent (4.1.1) or (4.1.2).

4.3 Sodium hydroxide, 200 g/l solution or approximately 5 N.

4.4 Iodized starch, solution containing 5 g/l of starch and 150 g/l of potassium iodide.

4.5 Acetone.

4.6 Chlorine-resistant grease. Greases based on fluorinated or chlorofluorinated products are suitable.

5 APPARATUS

The apparatus used is shown diagrammatically in Figure 2. It comprises the following components :

- **Bottle** (A) containing 2 l of the sodium hydroxide solution (4.3) for absorbing the sample of chlorine used.
- **Bubbler** (C) containing iodized starch solution (4.4), for regulating the rate of chlorine absorption.
- **Capillary flowmeter** (D) and differential manometer (M) containing carbon tetrachloride to permit a delivery of 25 l/h of gaseous chlorine. A suitable floating indicator flowmeter can be used instead.
- **U-tube** (E) fitted with ground glass joints, filled with mineral wool previously dried in an oven at 110 °C for 1 h.
- **Filter** (F), consisting of a threaded steel tube having an inside diameter of approximately 6 mm, 150 mm long and filled with glass wool.
- **Safety bottle** (G).
- **Electric heater** (H).
- **Glass valve** (P) filled with carbon tetrachloride.

- **T-bore stopcock** (R1).
- **Straight bore stopcock** (R2).
- **Steel coil** (S) immersed in a tank of water heated to 80 °C by means of an electric heater.
- **3 U-tubes** (T1, T2 and T3), with ground glass stoppers, containing phosphorus pentoxide (4.1.1) or magnesium perchlorate (4.1.2), covered by a wad of glass wool in each of the two branches. The packing of the absorption tubes with phosphorus pentoxide should be performed in a current of dry nitrogen shielded from atmospheric moisture. Carefully adjust the corresponding male and female components in order to ensure that the assembly is tightly sealed. Each loaded tube, weighed separately, should not have a mass of more than 100 g. After each weighing replace the tubes in the same order, and in such a way as to pass the gas in the same direction.
- **Valve** (V1).
- **Steel needle valve** (V2).
- **Flexible connections,** of chlorine-resistant rubber.

6 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.

6.1 Test portion

The best procedure consists of connecting the apparatus, containing absorbers, weighed ready for the determination, directly to a countercurrent sampling point in the liquid chlorine pipeline (see Figure 1). The test portion, taken in this way during filling or emptying of the tank wagon, is perfectly representative of the supply.

If necessary, if this operation is not possible and if small, clean and dry stainless steel bottles are available, test samples may be taken with the aid of these, on condition that the determinations are carried out without delay, and the bottles are then emptied and the insides cleaned with care, by flushing with dry, hot nitrogen, ready for further sampling.

6.2 Preliminary operations

Before filling the U-tubes (T1, T2 and T3) with absorbent (4.1), clean them with water and then with acetone (4.5), and dry them. After filling, coat the taps with a little of the grease (4.6) and place these absorber tubes in the apparatus.

When the absorbent is used for the first time, prepare the tubes by passing through gaseous chlorine at a rate of 25 l/h for 2 h, in order to allow the reaction between chlorine and certain impurities in the reagent. Without this preliminary operation, the first result will be in excess of the correct value.

Then operate the stopcocks R 1 and R 2 so as to put the apparatus into communication with the dry air or the dry nitrogen (4.2), and close the chlorine feed valves V1 and V2. Flush the absorber tubes for 10 min at the rate of 25 l/h with air or nitrogen at 80 °C, to eliminate any possible volatile impurities other than water remaining in the absorber tubes. Remove the hot water bath and continue the flushing for a further 10 min with the same gas at room temperature, then close R1 and R2, disconnect the absorber tubes, T1 and T2, dab them with a cloth lightly soaked in acetone to disperse any electrostatic charge. After 2 to 3 min weigh them separately to the nearest 0.1 mg.

Place 2 l of the sodium hydroxide solution (4.3) in the bottle A, weigh complete to the nearest 0.5 g and assemble the different parts of the apparatus.

6.3 Determination

Open in turn valves V1 and V2, and turn the tap R1 so as to allow the filtered and gasified chlorine to flow towards the valve P. After 10 min open the stopcock R2 to allow the chlorine to enter the absorbers, at a flow rate of 25 l/h, determined by the difference of the level n of the carbon tetrachloride in the two branches of the differential manometer M of the flowmeter D or by the height of the ball in the floating indicator flowmeter. This flow is obtained by regulating the opening of the valves and the height h of the carbon tetrachloride in the valve P.

In this way, allow about 100 l of chlorine to pass into the absorbers. The exact mass is determined by weighing the absorption flask A before and after the determination.

Throughout the time taken by the determination (about 4 h) ensure that the chlorine is totally absorbed. If this condition is satisfied, the iodized starch solution (4.4) in the bubbler (C) should not become coloured intense blue.

When the quantity of chlorine used is judged to be sufficient, close the valve V1, wait until the flow of chlorine measured in M has ceased, close the valve V2 and then adjust the stopcock R1 so as to put the apparatus in communication with the dry air or dry nitrogen (4.2).

Flush the absorber tubes for 10 min at the rate of 25 l/h with air or nitrogen at 80 °C in order to eliminate the chlorine and any possible volatile impurities other than water from the chlorine remaining in the absorber tubes. Remove the hot water bath and continue the flushing for a further 10 min with the same gas at room temperature, then close stopcocks R1 and R2, disconnect the absorber tubes, T1 and T2, and dab them with a cloth lightly soaked in acetone to disperse any electrostatic charge. After 2 to 3 min weigh them separately to the nearest 0.1 mg.

Normally, the water is completely absorbed in the tube T1. If the tube T2 shows an increase in mass of more than 20 % of the quantity absorbed in T1, the determination should be recommenced after recharging T1 and reconditioning the tube as laid down in the second paragraph of 6.2.

The presence of the tube T3 makes it possible to ascertain, in the same way, the moment when the contents of T2 must also be renewed, and it serves as an extra safety bottle.

7 EXPRESSION OF RESULTS

Water content, expressed in grams per kilogram, is given by the formula :

$$\frac{m_1 \times 1\,000}{m_0}$$

where

m_1 is the mass, in grams, of the weighed water (difference between the masses of absorbers T1 + T2, before and after the passage of the chlorine);

m_0 is the mass, in grams, of the test portion (difference in the mass of the bottle A before and after the determination).

Express the results to two decimal places.

8 TEST REPORT

The test report shall include the following particulars :

a) the method used;

b) the results and the method of espression used;

c) any unusual features noted during the determination;

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

ANNEX

METHOD FOR CHECKING THE DEGREE OF HYDRATION OF THE MAGNESIUM PERCHLORATE USED AS DRYING AGENT IN THE DETERMINATION OF WATER CONTENT OF CHLORINE

A.1 PRINCIPLE

Measurement of the increase in temperature resulting from the dissolution of the magnesium perchlorate in water. Determination of the corresponding water content, using the attached diagram (Figure 3).

A.2 APPARATUS

A.2.1 Silvered Dewar flask, of about 60 mm internal diameter, 100 mm high and of 300 ml capacity.

A.2.2 Thermometer, covering the range from 0 to 100 °C, graduated in 1 °C intervals.

A.2.3 Paraffined cork stopper, pierced with a hole to take the thermometer and intended to form a stopper for the Dewar flask (A.2.1).

A.2.4 Filter paper cylinder, 40 mm in diameter and 75 mm high, open at the top, made from a rectangular sheet of filter paper measuring 100 mm X 150 mm, rolled on a suitable former and turned in towards the bottom to form the base, the whole being held in shape with adhesive paper.

A.2.5 Graduated cylinder, 100 ml.

A.3 PROCEDURE

A.3.1 Test portion

At the moment of use, weigh, to the nearest 0.1 g, 20 g of magnesium perchlorate in the filter paper cylinder (A.2.4) and close the cylinder by folding the paper.

A.3.2 Determination

Pour 75 ml of distilled water into the Dewar flask (A.2.1). Close the flask with the paraffined cork (A.2.3) fitted with the thermometer (A.2.2) and read the temperature of the water when equilibrium is reached.

Place the filter paper cylinder (A.2.4) containing the test portion (A.3.1) in the Dewar flask (A.2.1) and replace the cork. Shake the flask with a gentle circular movement to aid dissolution of the product and read the temperature when it reaches equilibrium (i.e. after 20 to 25 s).

A.4 EXPRESSION OF RESULTS

Ascertain from the diagram in Figure 3 the water content of the product, as a percentage by mass, corresponding to the increase in temperature observed at the time of dissolution.

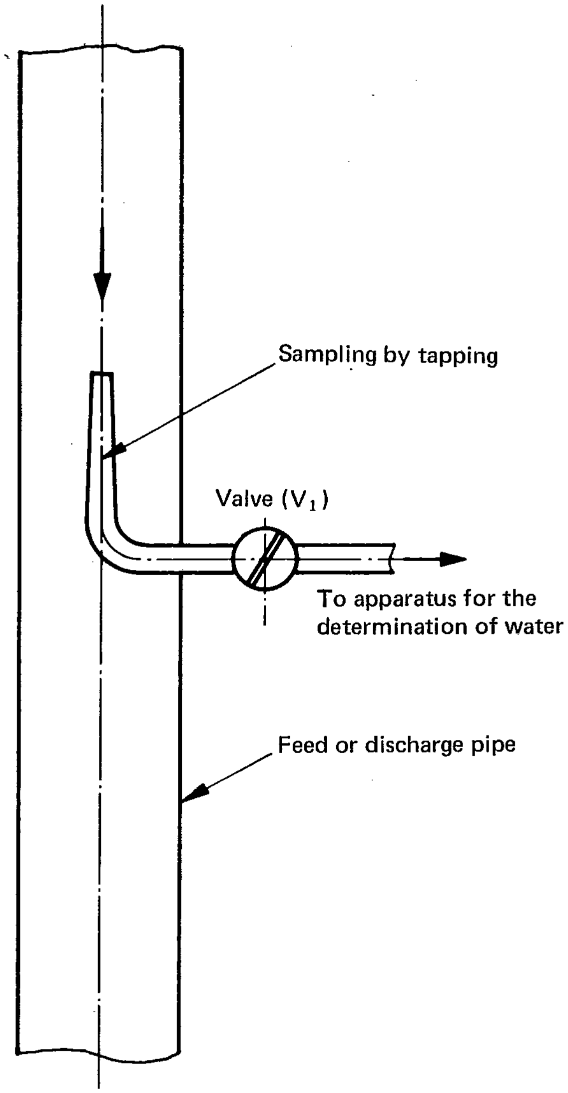
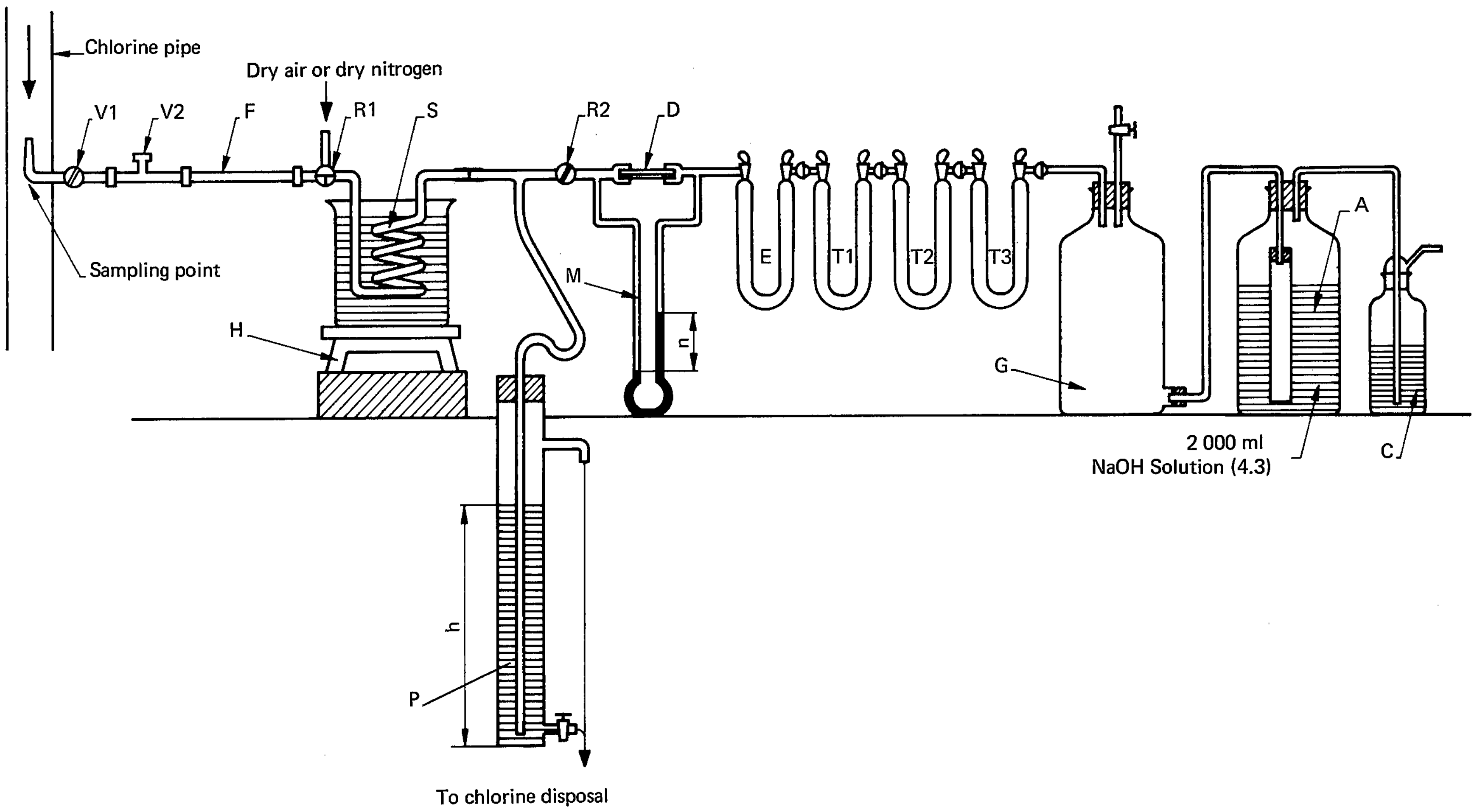


FIGURE 1 — **Diagram of sampling by tapping the feed or discharge pipes of a tank wagon**



NOTE — Ground joints are coated with the grease (4.6).

FIGURE 2 — Diagram of apparatus for the determination of water in chlorine

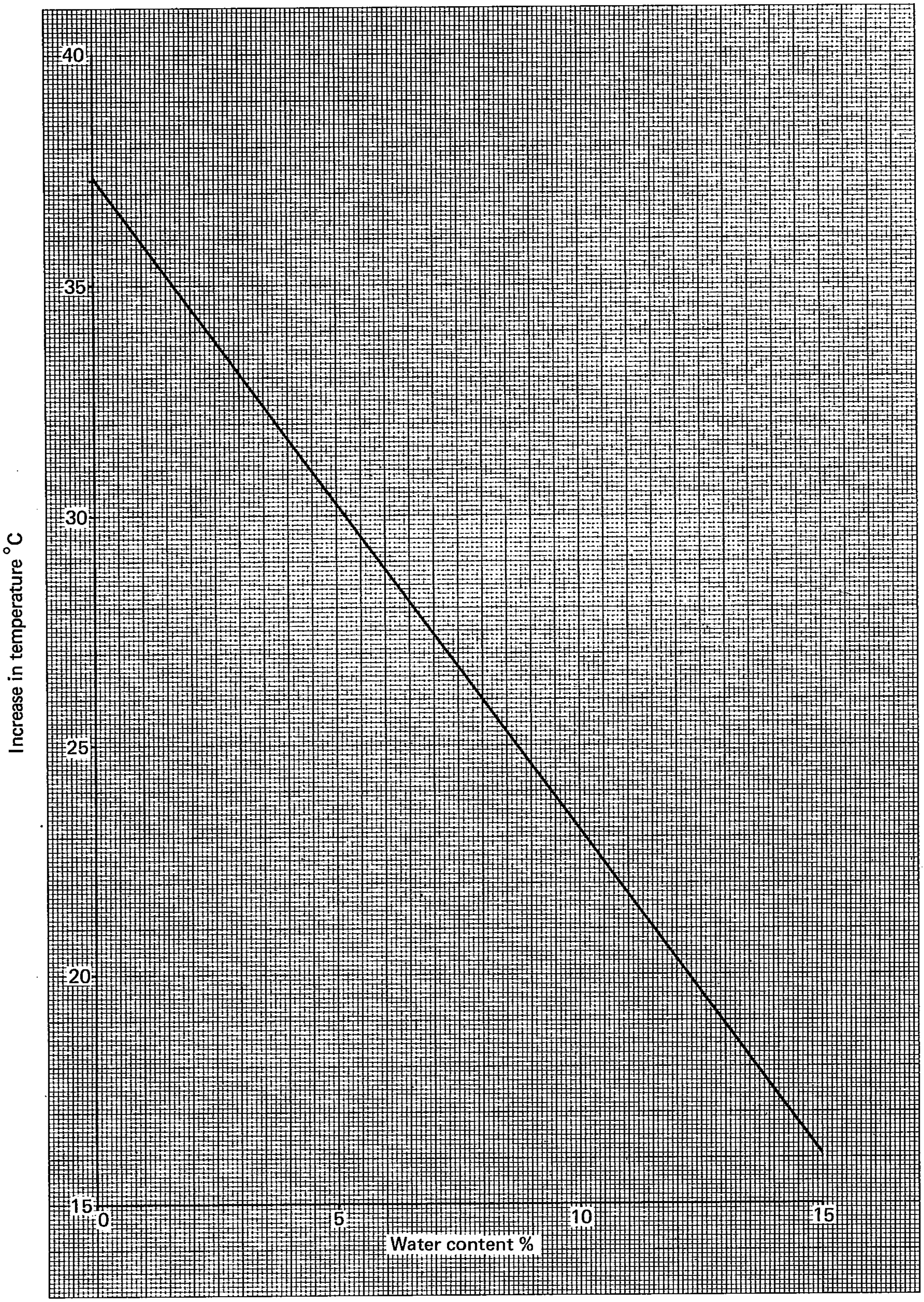


FIGURE 3 — Calibration curve giving the water content of the magnesium perchlorate as a function of the increase in temperature caused by its dissolution