

International Standard



1158

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Plastics — Vinyl chloride homopolymers and copolymers — Determination of chlorine

Plastiques — Homopolymères et copolymères de chlorure de vinyle — Détermination de la teneur en chlore

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 1158 was prepared by Technical Committee ISO/TC 61, *Plastics*.

ISO 1158 was first published in 1978. This second edition cancels and replaces the first edition, of which it constitutes a minor revision.

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Plastics — Vinyl chloride homopolymers and copolymers — Determination of chlorine

1 Scope and field of application

This International Standard specifies two methods for the determination of the chlorine in homopolymers and copolymers of vinyl chloride, free from plasticizers or additives, namely:

- method A (combustion in a bomb);
- method B (combustion in a flask).

WARNING — All precautions must be taken, particularly those mentioned in the text of the present International Standard, to protect operators from risks presented by both methods.

2 Principle

Oxidation of the test portion with sodium peroxide (method A) or gaseous oxygen (method B) followed by electrometric or volumetric titration of the resulting chlorides.

3 Reagents

During the analysis, use only reagents of recognized grade and only distilled water or water of equivalent purity.

3.1 Silver nitrate, standard volumetric solution, $c(\text{AgNO}_3) = 0,1 \text{ mol/l}$.

3.2 Nitric acid, solution, $c(\text{HNO}_3) = 2 \text{ mol/l}$.

For method A only:

3.3 Nitric acid, concentrated.

3.4 Sodium peroxide, granulated.

3.5 Starch or **sucrose** as combustion aids.

For method B only:

3.6 Oxygen, gaseous.

3.7 Sodium nitrate.

3.8 Potassium hydroxide solution, 100 g/l.

3.9 Hydrogen peroxide solution, 300 g/l.

4 Apparatus

4.1 Drying oven, capable of being controlled at $50 \pm 2 \text{ }^\circ\text{C}$ or $75 \pm 2 \text{ }^\circ\text{C}$.

4.2 Balance, to weigh to an accuracy of 0,1 mg.

4.3 Equipment for Volhard titration or for electrometric titration, with a burette having a capacity and accuracy appropriate to the chosen method (A or B).

For method A only:

4.4 Combustion bomb (for example Parr bomb or another bomb which gives the same results), gas or electrically fired. A suitable gas-fired bomb is shown in figure 1.

4.5 Nickel crucible with lid, to fit into the bomb (gas-fired). Suitable dimensions are: diameter 25 mm, height 40 mm.

4.6 Safety oven.

4.7 Beaker, capacity 600 ml.

For method B only:

4.8 Round-bottomed flask, capacity 500 ml, with head for oxygen combustion (see figure 2). A platinum wire 1,0 mm diameter and 120 mm long in the shape of a tapered spiral is attached to the stopper, a suitable spiral being 15 mm in diameter and 15 mm long. **It is recommended that metal gauze be wrapped around the flask for safety.**

4.9 Filter paper, about 3 cm × 3,5 cm, free from halogens and ash.

4.10 Beaker, capacity 100 ml.

5 Test sample

The sample shall in powdered or granular form, and if necessary shall be cut into pieces 1 to 3 mm in size.

The sample shall be oven-dried for 2 h at $75 \text{ }^\circ\text{C}$ or 16 h at $50 \text{ }^\circ\text{C}$.

Approximate dimensions in millimetres

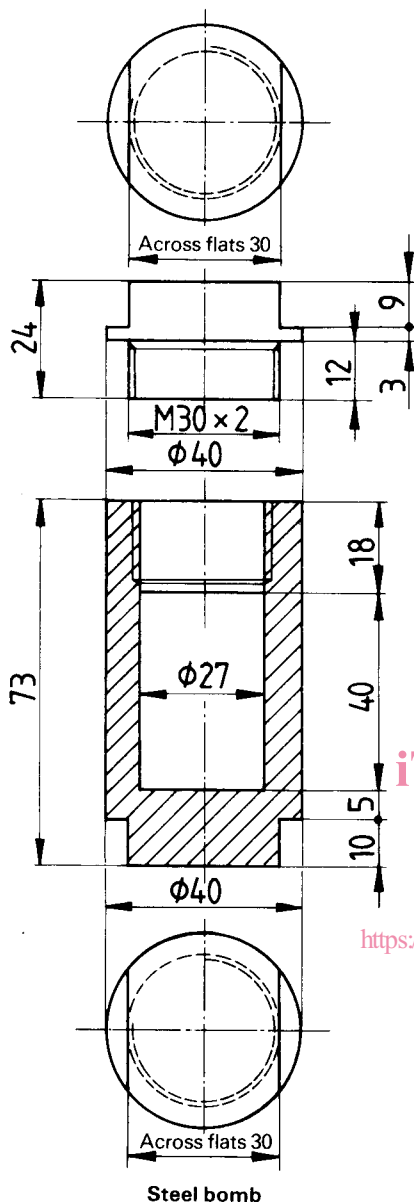


Figure 1 — Combustion bomb, gas-fired type (for method A)

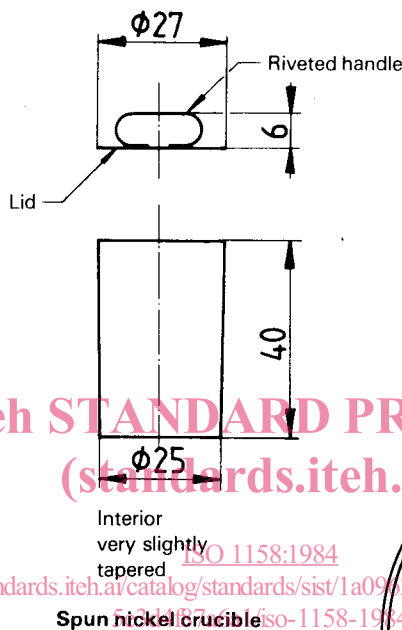


Figure 2 — Flask for oxygen combustion with platinum wire attached to stopper (for method B)

6 Procedure

6.1 Method A (combustion bomb)

6.1.1 First place 7 to 7,5 g of the sodium peroxide (3.4) in the nickel crucible (4.5) (for the gas-fired bomb) or in the fusion cup of the bomb (for the electrically fired bomb), then add a test portion of about 0,25 g (weighed to 0,000 5 g) mixed with 0,16 to 0,17 g of starch or sucrose (3.5), then a further 7 to 7,5 g of the sodium peroxide. **The filling with sodium peroxide shall be done behind a shield protecting the operator.** Mix all by stirring, then place the crucible, with the lid in position, inside the bomb and close the bomb tightly. If an electrically fired bomb is used, assemble the bomb and tap it to settle the charge.

6.1.2 Fire the bomb.

NOTE — If a gas-fired bomb is used, place it in the safety oven (4.6). Adjust the flame beforehand, using an empty bomb in the safety oven, so that the top of the flame is a few millimetres from the base of the bomb. Then remove the empty bomb. Heat the test bomb to 300 to 400 °C for about 10 min. Ignition usually starts at 50 to 60 °C, and is detected by a cracking sound, and the fact that the bottom of the bomb starts to glow.

6.1.3 Cool the bomb. Open it and, if a gas-fired bomb is used, remove the crucible and carefully place it in 100 ml of distilled water in the beaker (4.7) and immediately cover the beaker with a watch-glass. When the reaction has subsided, wash down the inside of the bomb and the plug, collecting the washings in the beaker.

If an electrically fired bomb is used, dismantle it after cooling, remove the head and tip it into 100 ml of distilled water in the beaker (4.7). Lay the fusion cup in the same beaker and immediately cover with a watch-glass.

WARNING — If the bomb is cooled in water, take care that the water does not reach the joint between the plug and the bomb.

6.1.4 Heat the beaker and its contents to boiling, then cool. Rinse the crucible and lid, or the fusion cup and head, into the beaker with water, then remove them.

6.1.5 Slowly add 20 ml of the concentrated nitric acid (3.3), stirring constantly, followed by the nitric acid solution (3.2) until the mixture is neutral. Then add a further 2 ml of the nitric acid solution (3.2).

NOTE — Methyl orange is a suitable indicator for the neutralization.

6.1.6 Dilute the contents of the beaker to about 200 ml with water, and titrate electrometrically or by the Volhard method with the silver nitrate solution (3.1).

6.1.7 Carry out a blank test by firing the same amount of sodium peroxide (3.4) and sucrose or starch (3.5) as was used with the test portion, and repeating the procedure (but without the test portion) described in 6.1.4 to 6.1.6.

6.1.8 When doubt exists as to whether the reaction has taken place, do not dissolve the contents of the bomb into water according to the normal procedure because this might cause a violent explosion. The contents of the bomb should be spread out onto dry sand after which they should be sprayed with water from a safe distance and then washed with more water.

6.2 Method B (oxygen flask)

6.2.1 Place a test portion of about 25 to 35 mg, weighed to 0,1 mg, on a filter paper (4.9) cut as shown in figure 3 a) below and having previously marked folds. Then fold the paper as shown in figures 3 b), c) and d) and clamp it in the platinum spiral (see figure 2), with the paper tail protruding.

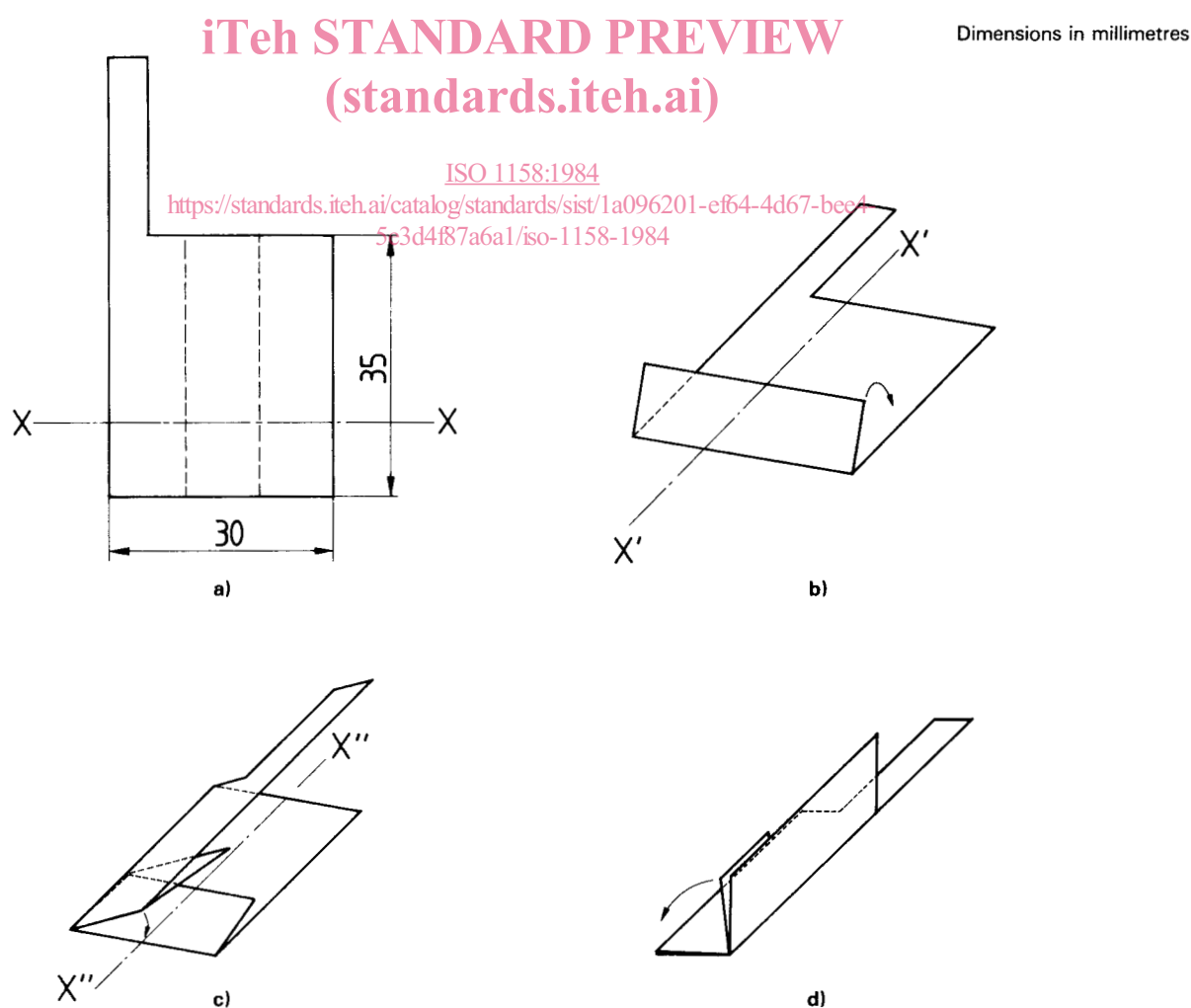


Figure 3 — Folding of filter paper containing test portion

6.2.2 Introduce about 10 ml of water, 1 ml of the potassium hydroxide solution (3.8) and 0,15 ml of the hydrogen peroxide solution (3.9) into the flask (4.8). Pass oxygen (3.6) through a glass tube at 250 to 350 ml/min for 5 min to displace the air.

6.2.3 Ignite the filter paper tail with a gas flame and quickly insert the stopper carrying the platinum wire and burning filter paper into the flask.

6.2.4 During combustion keep the flask inverted so that the liquid covers the bottom of the stopper and leakage through the stopper and escape of gas are avoided. When combustion is finished, turn the flask upright and gently shake under a stream of cold water, to cause rapid and complete absorption of the hydrochloric acid produced.

6.2.5 After 30 min, open the flask and transfer the contents quantitatively to the beaker (4.10), rinsing so that the final volume is about 30 ml. Add about 1 g of the sodium nitrate (3.7) and 2,5 ml of the nitric acid solution (3.2), and boil the solution for 5 min. After cooling, determine the chlorine content by electrometric titration or by the Volhard method with the silver nitrate solution (3.1).

6.2.6 Carry out a blank test following the procedure described in 6.2.1 to 6.2.4 and using the same quantities of all the reagents used in the determination, but without the test portion.

7 Expression of results

7.1 The chlorine content of the dry material, expressed as a percentage by mass, is given by the formula

$$3,55 \times \frac{0,1 (V_1 - V_2)}{m}$$

where

V_1 is the volume, in millilitres, of the silver nitrate solution (3.1) used for the determination;

V_2 is the volume, in millilitres, of the silver nitrate solution (3.1) used for the blank test;

m is the mass, in grams, of the test portion.

7.2 Express the result as the arithmetic mean of two determinations that do not differ by more than $\pm 0,2 \%$.

8 Test report

The test report shall include the following information:

- a) reference to this International Standard;
- b) full details necessary for complete identification of the sample;
- c) the test procedure used (A or B);
- d) the percentage of chlorine in the sample.

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