
**Plastics — Vinyl chloride homopolymers
and copolymers — Determination of
chlorine content**

*Plastiques — Homopolymères et copolymères de chlorure de vinyle —
Dosage du chlore*



Foreword

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This third edition cancels and replaces the second edition (ISO 1158:1984), which has been technically revised.

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Plastics — Vinyl chloride homopolymers and copolymers — Determination of chlorine content

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

1 Scope

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

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

2 Principle

A test portion is oxidised with sodium peroxide (method A) or gaseous oxygen (method B) followed by potentiometric 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, $c(\text{AgNO}_3) = 0,1 \text{ mol/l}$ or $0,05 \text{ mol/l}$.

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, sucrose or ethylene glycol, 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 maintained at $50\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ or $75\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

4.2 Balance, to weigh to an accuracy of 0,1 mg (method A) or 0,01 g (method B).

4.3 Equipment for Volhard titration or for potentiometric 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. An example of 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. A smaller crucible could be used if the amount of test sample is reduced.

4.6 Safety oven.

4.7 Beaker, capacity 600 ml.

For method B only:

4.8 Round- or flat-bottomed flask, capacity 500 ml to 1 000 ml, with head designed for oxygen combustion (see figure 2). A platinum wire 1,0 mm in 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 250 ml.

5 Test sample

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

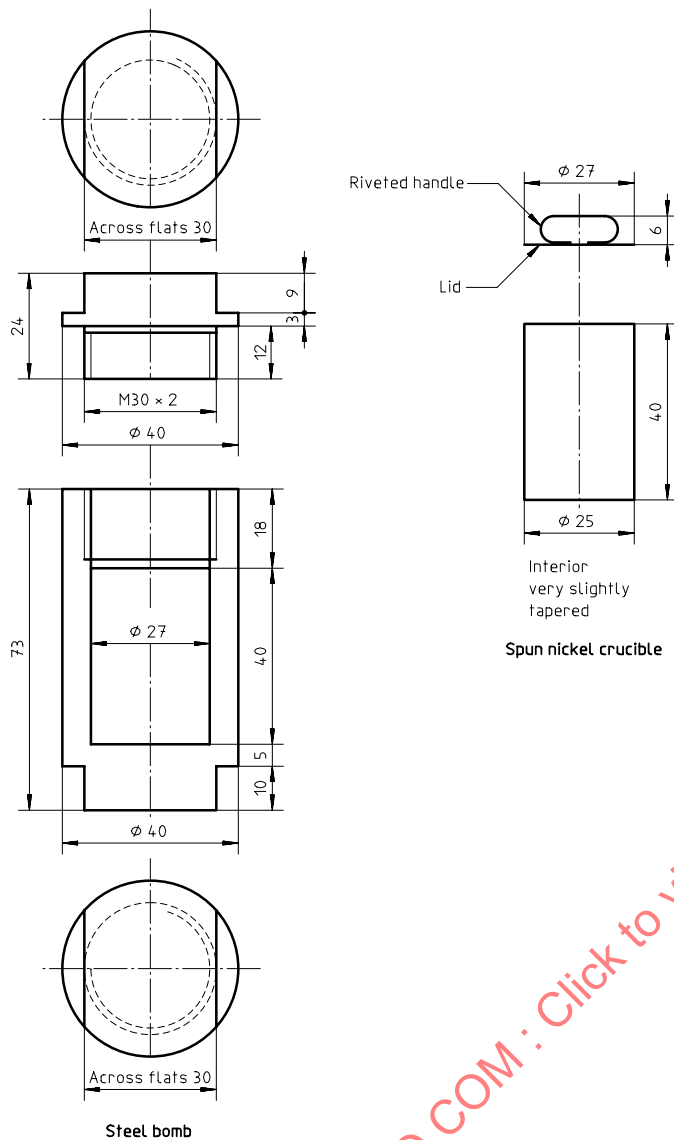


Figure 1 — Example of a combustion bomb, gas-fired type (for method A)

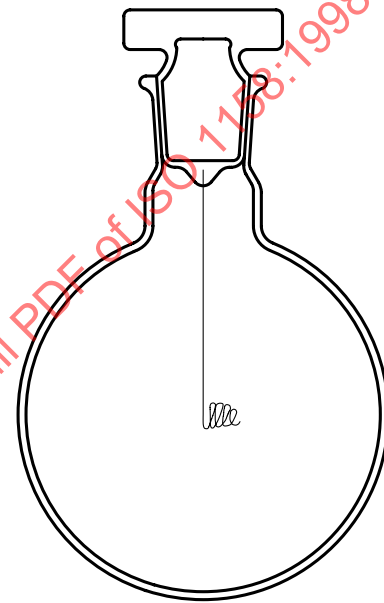


Figure 2 — Example of a flask for oxygen combustion with platinum wire attached to stopper (for method B)

6 Procedure

6.1 Method A (combustion bomb)

6.1.1 First place 7 g to 7,5 g of 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 0,1 mg) mixed with 0,16 g to 0,17 g of combustion aid (3.5), then a further 7 g to 7,5 g of sodium peroxide. The placing of the sodium peroxide in the crucible or fusion cup shall be done behind a shield protecting the operator. Mix 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.

A smaller crucible and test portion could be used.

6.1.2 Fire the bomb.

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 °C to 400 °C for about 10 min. Ignition usually starts at 50 °C 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 a 600 ml 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 the contents 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. Remove the crucible and lid, or the fusion cup and head, rinsing them with water and collecting the washings in the beaker.

6.1.5 Slowly add 20 ml of concentrated nitric acid (3.3), stirring constantly, followed by nitric acid solution (3.2) until the mixture is neutral. Then add a further 2 ml of 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 potentiometrically or by the Volhard method with silver nitrate solution (3.1).

6.1.7 Carry out a blank test by firing the same amount of sodium peroxide (3.4) and combustion aid (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 in water by the normal procedure because this might cause a violent explosion. The contents of the bomb should be spread out on 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 mg to 35 mg, weighed to 0,01 mg, on a filter paper (4.9) cut as shown in figure 3a) and having previously marked folds. Then fold the paper as shown in figures 3b), c) and d) and clamp it in the platinum spiral (see figure 2), with the paper tail protruding.

Dimensions in millimetres

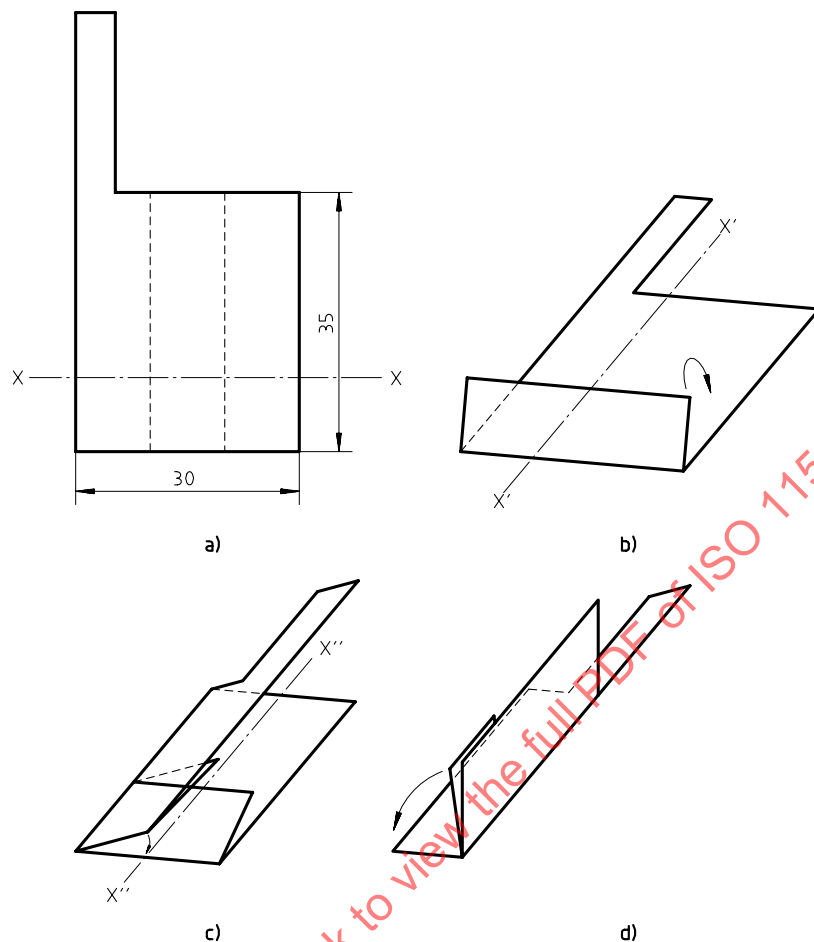


Figure 3 — Folding of filter paper containing test portion

6.2.2 Introduce about 20 ml of water, 1 ml of potassium hydroxide solution (3.8) and 0,15 ml of hydrogen peroxide solution (3.9) into the flask (4.8). Pass oxygen (3.6) through a glass tube at 250 ml/min 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 complete absorption of the hydrochloric acid produced.

6.2.5 After 30 min, open the flask and transfer the contents quantitatively to a 250 ml beaker (4.10), rinsing so that the final volume is about 60 ml. Add about 1 g of sodium nitrate (3.7) and 2,5 ml of nitric acid solution (3.2), and boil the solution for 5 min. After cooling, determine the chlorine content by potentiometric titration or by the Volhard method with 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

$$3,545\ 3 \times \frac{0,1 \times (V_1 - V_2)}{m}$$

or

$$3,545\ 3 \times \frac{0,05 \times (V_1 - V_2)}{m}$$

depending on the concentration of the silver nitrate solution used

where

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

V_2 is the volume, in millilitres, of 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 0,2 % (absolute).

8 Precision

The precision of this test method is not known because interlaboratory data are not available. Interlaboratory data are being obtained and a precision statement will be added at a subsequent revision.

9 Test report

The test report shall include the following information:

- a reference to this International Standard;
- all details necessary for complete identification of the sample;
- the test procedure used (A or B);
- the dimensions of the crucible and the mass of the test portion, if different from those given in 4.5 and 6.1.1;
- the percentage chlorine content of the sample;
- the date of the test.