UDC 678.743 : 543.848

Ref. No. : ISO/R 1158-1970 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 1158

PLASTICS

DETERMINATION OF CHLORINE IN VINYL CHLORIDE POLYMERS AND COPOLYMERS

1st EDITION

January 1970

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 1158, *Plastics – Determination of chlorine in vinyl chloride polymers and copolymers*, was drawn up by Technical Committee ISO/TC 61, *Plastics*, the Secretariat of which is held by the American National Standards Institute (ANSI).

Work on this question led to the adoption of a Draft ISO Recommendation.

In July 1965, this Draft ISO Recommendation (No. 826) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Argentina	Germany	Romania
Australia	Greece	Spain
Austria	Hungary	Sweden
Belgium	India	Switzerland
Brazil	Ireland	Turkey
Canada	Israel	U.A.R.
Chile	Italy	United Kingdom
Colombia	Japan	U.S.A.
Czechoslovakia	Netherlands	U.S.S.R.
Finland	New Zealand	
France	Poland	

No Member Body opposed the approval of the Draft.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in January 1970, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 1158 January 1970

PLASTICS

DETERMINATION OF CHLORINE IN VINYL CHLORIDE POLYMERS AND COPOLYMERS

1. SCOPE

This ISO Recommendation describes two methods for the determination of the chlorine in polymers 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

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

All the reagents must be of analytical reagent quality. Water should be distilled water or water of at least equivalent purity.

3.1 *Silver nitrate*, 0.1 N standard solution.

3.2 *Nitric acid*, 2 N solution.

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 maintaining a temperature of 50 ± 2 °C or 75 ± 2 °C.

4.2 *Balance* to weigh to an accuracy of 0.0001 g.

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* (e.g. Parr bomb or another bomb which gives the same results), gas or electrically fired. A suitable gas-fired bomb is shown in Figure 1, opposite.

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*, 600 ml.

For method B only :

4.8 *Round-bottomed flask*, 500 ml, with head for oxygen combustion (see Fig. 2, opposite). 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*, 100 ml.

5. TEST SAMPLE

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

The sample should be oven-dried for 2 hours at 75 °C or 16 hours at 50 °C.

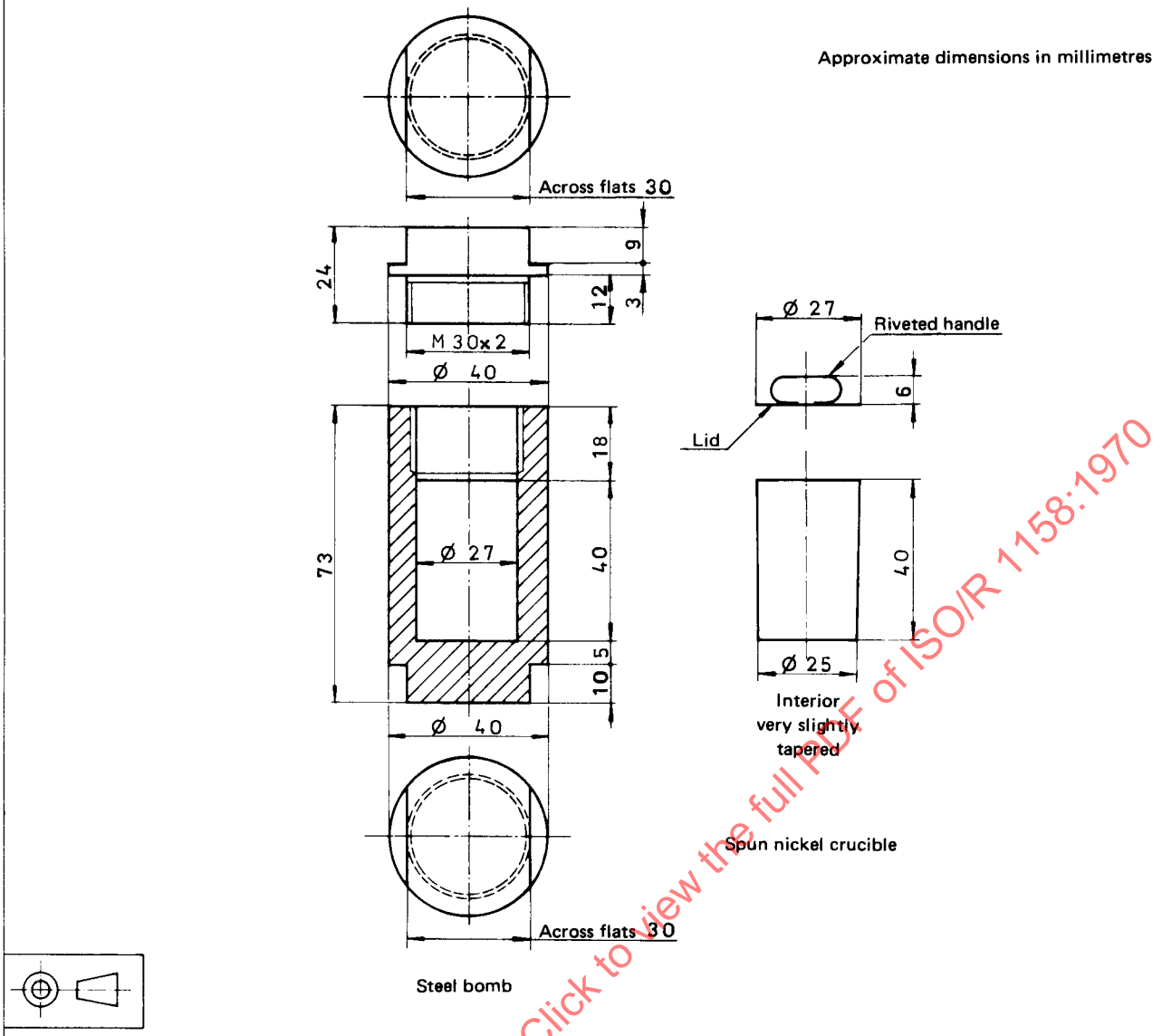

FIG. 1 – **Combustion bomb. gas-fired type**
(for method A)

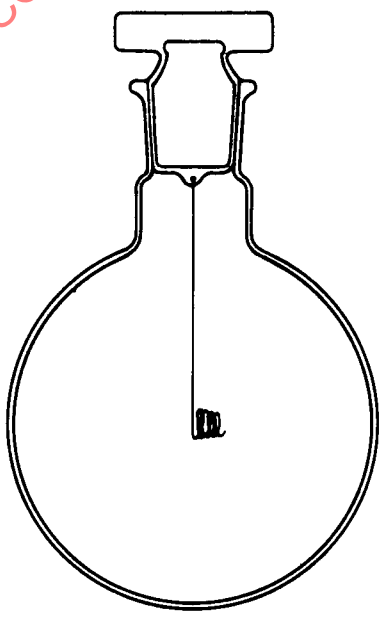

FIG. 2 – **Flask for oxygen combustion with platinum wire attached to stopper**
(for method B)

6. PROCEDURE

6.1 Method A (combustion bomb)

6.1.1 First place 7 to 7.5 g of 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 about 0.25 g of test sample (weighed to 0.0005 g) mixed with 0.16 to 0.17 g of starch or sucrose, then a further 7 to 7.5 g of sodium peroxide. The filling with sodium peroxide should 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, the bomb is placed in the safety oven. The flame is adjusted 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. The empty bomb is then removed. The test bomb is heated to 300 to 400 °C for about 10 minutes. 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 a 600 ml beaker and immediately cover the beaker with a watchglass. 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 a 600 ml beaker. Lay the fusion cup in the same beaker and immediately cover with a watchglass.

NOTE. – 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 distilled water, then remove them.

6.1.5 Slowly add 20 ml of concentrated nitric acid (3.3), stirring constantly, followed by 2 N nitric acid solution (3.2) until the mixture is neutral. Then add a further 2 ml of 2 N nitric acid (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 distilled water, and titrate electrometrically 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 sucrose or starch (3.5) as was used with the test sample, and repeating the procedure (but without the test sample) described in clauses 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 lead to heavy 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.