
INTERNATIONAL STANDARD



1270

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Plastics — PVC resins — Determination of ash and sulphated ash

Matières plastiques — Résines de polychlorure de vinyle — Détermination des cendres et des cendres sulfatées

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 61 has reviewed ISO Recommendation R 1270 and found it technically suitable for transformation. International Standard ISO 1270 therefore replaces ISO Recommendation R 1270-1970 to which it is technically identical.

ISO Recommendation R 1270 was approved by the Member Bodies of the following countries :

Australia	Hungary	Poland
Austria	India	Romania
Belgium	Iran	South Africa, Rep. of
Bulgaria	Israel	Spain
Canada	Italy	Sweden
Czechoslovakia	Japan	Turkey
Egypt, Arab Rep. of	Korea, Dem. P. Rep. of	United Kingdom
France	Korea, Rep. of	U.S.A.
Germany	Netherlands	Yugoslavia
Greece	New Zealand	

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

Switzerland

The Member Bodies of the following countries disapproved the transformation of ISO/R 1270 into an International Standard :

Canada
United Kingdom

Plastics — PVC resins — Determination of ash and sulphated ash

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies two conventional methods of determining ash and sulphated ash content of a PVC resin.

2 PRINCIPLE

Method A — For the determination of the *percentage of ash*: ashing of the test sample at 850 ± 50 °C to constant mass.

Method B — For the determination of the *percentage of ash as sulphates*: preliminary combustion of the test sample, treatment of the residue with sulphuric acid, heating, neutralization of excess acid by the addition of ammonium carbonate and heating. Then ashing at 850 ± 50 °C to constant mass.

3 REAGENTS (used only for sulphated ash)

3.1 Sulphuric acid, pure, ρ 1,84 g/ml.

3.2 Ammonium carbonate, anhydrous, powdered, analytically pure.

4 APPARATUS

4.1 Crucibles, of silica, porcelain or platinum, 45 to 75 mm diameter and at least the same depth. Deep crucibles are particularly recommended, especially for the determination of sulphated ash.

4.2 Bunsen burner or equivalent apparatus.

4.3 Muffle furnace, gas or electric, controlled at 850 ± 50 °C.

4.4 Balance, accurate to 0,000 1 g.

4.5 Pipette.

4.6 Desiccator.

5 PROCEDURE

5.1 Procedure for one determination

5.1.1 Method A (Determination of ash)

Heat the crucible (4.1) for 10 min at 850 ± 50 °C and cool in the desiccator (4.6). Weigh the crucible to the nearest 0,000 5 g, and add about 5 g of PVC resin. Reweigh the crucible and its contents to the nearest 0,000 5 g and calculate the mass m_0 of resin used.

Place the crucible in the opening of the muffle furnace (4.3) controlled at 850 ± 50 °C (the temperature in this area will be of the order of 300 to 400 °C), and then move it by stages, gently, into the interior of the furnace. The ashing must be carried out slowly so that the volatiles do not carry off particles of ash.

Heat for 10 min at 850 ± 50 °C.

After cooling in the desiccator, weigh the crucible to the nearest 0,000 5 g.

Heat again for 10 min, recool in the desiccator and reweigh.

Repeat these operations until constant mass is obtained; i.e. until two successive weighings do not differ by more than 0,000 5 g.

From the difference in the weighings calculate the mass m_1 , in grams, of ash.

5.1.2 Method B (Determination of sulphated ash)

Heat the crucible (4.1) for 10 min at 850 ± 50 °C and cool in the desiccator (4.6). Weigh the crucible to the nearest 0,000 5 g, and add about 5 g of PVC resin. Reweigh the crucible and its contents to the nearest 0,000 5 g and calculate the mass m_0 of resin used.

Gently heat the crucible with the Bunsen burner (4.2) or the equivalent apparatus, until the resin and any products of combustion of the sample are completely volatilized. (This is shown by the disappearance of the black colour from the inside walls of the crucible.) The ashing must be carried out slowly so that the volatiles do not carry off particles of ash.

Allow the crucible and contents to cool.

By means of the pipette (4.5) add to the crucible successive small quantities of sulphuric acid (3.1), warming carefully (at about 400 °C) until the reaction is completed.

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Then add ammonium carbonate (3.2) to neutralize excess acid, warming until the emission of white fumes ceases.

Place the crucible in the opening of the muffle furnace (4.3) controlled at 850 ± 50 °C (the temperature in this area will be of the order of 300 to 400 °C), and then move it by stages, gently, into the interior of the furnace. The ashing must be carried out slowly so that the volatiles do not carry off particles of ash.

Heat for about 10 min at 850 ± 50 °C.

After cooling in the desiccator, weigh the crucible to the nearest 0,000 5 g.

Heat again for about 10 min, recool in the desiccator and reweigh.

Repeat these operations until constant mass is obtained; i.e. until two successive weighings do not differ by more than 0,000 5 g.

From the difference in the weighings calculate the mass m_1' , in grams, of sulphated ash.

5.2 Number of determinations

Carry out two determinations.

Calculate the values of the percentage of ash or sulphated ash from the formulae in clause 6.

If these two percentages differ by less than 0,10 % in *absolute* value, use them for the calculation.

If not, carry out further determinations until two values satisfying this requirement are obtained.

However, if the two absolute values obtained are each less than 0,20 % – no matter what the difference between them – new determinations are not necessary.

6 EXPRESSION OF RESULTS

For each determination, calculate the percentage of ash or sulphated ash to two decimal places from the formulae

$$\frac{m_1}{m_0} \times 100 \quad \text{or} \quad \frac{m_1'}{m_0} \times 100$$

where

m_0 is the mass, in grams, of resin used;

m_1 is the mass, in grams, of ash;

m_1' is the mass, in grams, of sulphated ash.

Calculate the mean, to two decimal places (to the nearest 0,05), of the two values finally retained.

In the test report, quote this mean as the value of the percentage of ash or sulphated ash.

NOTE – Co-operative tests between different laboratories have shown reproducibility on the percentage values obtained, of $\pm 0,05$ %.

7 TEST REPORT

The test report shall include the following particulars :

- a) reference to this International Standard or to an equivalent national standard;
- b) complete identification of the product tested;
- c) the result expressed according to clause 6;
- d) any circumstances which may have affected the result;
- e) date of test.