

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 1270

PLASTICS

PVC RESINS

DETERMINATION OF ASH AND SULPHATED ASH

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 1270, *Plastics – PVC resins – Determination of ash and sulphated ash*, 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 Draft ISO Recommendation No. 1270, which was circulated to all the ISO Member Bodies for enquiry in May 1967. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

The following Member Body opposed the approval of the Draft :

Switzerland

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

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PLASTICS

PVC RESINS

DETERMINATION OF ASH AND SULPHATED ASH

1. SCOPE

This ISO Recommendation describes 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, $d = 1.84$.

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 0.0001 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 minutes at 850 ± 50 °C and cool in the desiccator (4.6). Weigh the crucible to the nearest 0.0005 g, and add about 5 g of PVC resin. Reweigh the crucible and its contents to the nearest 0.0005 g and calculate the mass m 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 minutes at 850 ± 50 °C.

After cooling in the desiccator, weigh the crucible to the nearest 0.0005 g.

Heat again for 10 minutes, 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 0.0005 g.

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

- 5.1.2 *Method B (Determination of sulphated ash).* Heat the crucible (4.1) for 10 minutes at 850 ± 50 °C and cool in the desiccator (4.6). Weigh the crucible to the nearest 0.0005 g, and add about 5 g of PVC resin. Reweigh the crucible and its contents to the nearest 0.0005 g and calculate the mass m 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.

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 minutes at 850 ± 50 °C.

After cooling in the desiccator, weigh the crucible to the nearest 0.0005 g.

Heat again for about 10 minutes, 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 0.0005 g.

From the difference in the weighings calculate the mass m'_1 , in grammes, of sulphated ash.