INTERNATIONAL STANDARD

ISO 1269

Third edition
2006-11-15

Plastics — Homopolymer and copolymer resins of vinyl chloride — Determination of volatile matter (including water)

Plastiques — Résines d'homopolymères et de copolymères de chlorure de vinyle — Détermination des matières volatiles (y compris l'eau)

Reference number
ISO 1269:2006(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 1269 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This third edition cancels and replaces the second edition (ISO 1269:1980), which has been technically revised.

The revision includes an additional method, method B, that uses an automatic thermobalance to determine the volatile-matter content.

Plastics — Homopolymer and copolymer resins of vinyl chloride — Determination of volatile matter (including water)

1 Scope

This International Standard specifies two methods for determining the volatile matter (including water) in homopolymer and copolymer resins of vinyl chloride.

2 Principle

A test portion of resin, spread out in a weighing dish of specified dimensions, is heated at an appropriate temperature to constant mass.

3 Apparatus

3.1 Method A (using an oven and balance)

3.1.1 Oven, capable of being controlled at 110 °C ± 2 °C, with slight natural draught or equipped with a low-speed circulation fan.

3.1.2 Weighing dish, shallow, about 80 mm in diameter and more than 5 mm in height, made of glass, aluminium or, preferably, stainless steel, with a lid.

3.1.3 Balance, capable of weighing to 0,001 g.

3.1.4 Desiccator, containing a suitable desiccant.

3.2 Method B (using an automatic thermobalance)

3.2.1 Oven, capable of being controlled at 110 °C ± 2 °C.

3.2.2 Automatic thermobalance, consisting of a precision balance and an IR or halogen oven. The thermobalance automatically evaporates the volatile matter to constant mass by checking the mass readings.

3.2.3 Weighing dish, about 100 mm in diameter and more than 5 mm in height, made of aluminium.

3.2.4 Balance, capable of weighing to 0,001 g.

3.2.5 Desiccator, containing a suitable desiccant.

4 Procedure

4.1 Method A

Bring the oven (3.1.1) to 110 °C ± 2 °C. Heat the dish (3.1.2), with its lid, in the oven for about 1 h. Remove and allow to cool in the desiccator (3.1.4) to room temperature. Weigh the dish and lid to the nearest 0,005 g.

Spread about 5 g of the test sample evenly over the bottom of the dish. Replace the lid and weigh to the nearest 0,005 g.

Place the assembly in the oven at 110 °C ± 2 °C. Remove the lid — but leave it in the oven — and continue heating for about 1 h.

Remove the assembly from the oven. Replace the lid on the dish. Allow to cool in the desiccator and weigh to the nearest 0,005 g.

Following the same procedure, heat in the oven for further periods of 30 min until the difference between two successive weighings does not exceed 0,005 g.

NOTE Prolonged heating at 110 °C ± 2 °C may result in the thermal degradation of some resins. In such circumstances, it is recommended that the evaporation procedure be conducted at 105 °C ± 2 °C.

Carry out two determinations on each test sample.

4.2 Method B

Bring the oven (3.2.1) to 110 °C ± 2 °C. Heat the aluminium dish (3.2.3) for about 1 h. Remove and allow to cool in the desiccator (3.2.5) to room temperature.

Place the dish in the automatic thermobalance (3.2.2) and tare it.

Spread 5 g to 15 g, depending on the type of resin, of the test sample evenly over the bottom of the dish and weigh it to the nearest 0,005 g.

Set the thermobalance test temperature to the value appropriate to the resin.

Switch on the thermobalance heating system and heat until the mass loss per second over a period of 2 min is less than 0,02 mg.

NOTE These operating conditions have been selected to minimize the effect of thermal degradation.

Carry out two determinations on each test sample.

5 Expression of results

5.1 Method A

For each determination, calculate the percentage of volatile matter (including water) to two decimal places from the formula:

$$\frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where

m_1 is the mass, in grams, of the empty dish and lid (after heating and cooling);

m_2 is the mass, in grams, of the dish, lid and test portion before heating;

m_3 is the mass, in grams, of the dish, lid and test portion after heating.

If the values of the percentage volatile matter obtained in the two determinations on the test sample differ by less than 0,10 % (absolute), use these values to calculate the mean percentage volatile matter of the test sample, expressing the mean to the nearest 0,01 % (absolute).

If this is not the case, carry out further determinations until two values satisfying this requirement are obtained. However, if the two values obtained for the percentage volatile matter are each less than 0,30 %, use these values to calculate the mean value for the test sample irrespective of the absolute difference between them.

NOTE For many purposes, for example the designation of a resin, the expression of the mean value of the percentage volatile matter to one decimal place is sufficient.

5.2 Method B

For each determination, the result is calculated automatically by the automatic thermobalance. It is expressed as a percentage to two decimal places.

If the values of the percentage volatile matter obtained in the two determinations on the test sample differ by less than 0,10 % (absolute), use these values to calculate the mean percentage volatile matter of the test sample, expressing the mean to the nearest 0,01 % (absolute).

If this is not the case, carry out further determinations until two values satisfying this requirement are obtained. However, if the two values obtained for the percentage volatile matter are each less than 0,30 %, use these values to calculate the mean value for the test sample irrespective of the absolute difference between them.

NOTE For many purposes, for example the designation of a resin, the expression of the mean value of the percentage volatile matter to one decimal place is sufficient.

6 Precision

— **Method A**: An interlaboratory test programme has given a reproducibility of ± 0,10 % (absolute).

— **Method B**: An interlaboratory test programme, carried out within one company, has given a reproducibility of ± 0,10 % (absolute).

7 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) all details necessary for complete identification of the product tested;

c) the method used, i.e. method A or method B;

d) the temperature used to heat the test portion in method A;

e) the mass of the test portion, the temperature and the test duration in method B;

f) the result, expressed in accordance with 5.1 or 5.2;

g) details of any circumstances which may have affected the result.

ICS 83.080.20

Price based on 3 pages