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Hot-melt adhesives — Determination of thermal stability

Adhésifs thermofusibles — Détermination de la stabilité thermique



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Foreword

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International Standard ISO 10363 was prepared by Technical Committee ISO/TC 61, *Plastics*, Sub-Committee SC 11, *Products*.

Annex A of this International Standard is for information only.

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Hot-melt adhesives — Determination of thermal stability

1 Scope

This International Standard specifies a method for determining the thermal stability of non-reactive hot-melt adhesives at temperatures up to 260 °C.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1770:1981, *Solid-stem general purpose thermometers*.

ISO 2555:1989, *Plastics — Resins in the liquid state or as emulsions or dispersions — Determination of apparent viscosity by the Brookfield Test method*.

ISO 4625:1980, *Binders for paints and varnishes — Determination of softening point — Ring-and-ball method*.

3 Principle

A quantity of hot-melt adhesive is heated under specified conditions. Samples are taken at regular time intervals and any changes in viscosity and in softening point (determined by the “ring-and-ball” method) during heating are noted. The maximum temperature and the duration of the test are fixed by agreement between the adhesive user and manufacturer.

4 Apparatus

4.1 Stainless-steel or glass vessel, external diameter 65 mm, height 95 mm, equipped with a loose-fitting lid.

4.2 Oil bath, stirred and capable of being maintained at the operating temperature recommended by the manufacturer of the hot-melt adhesive. Alternatively, a ventilated oven may be used instead of the oil bath. The oven or bath shall be capable of heating the sample to within ± 2 °C of the required temperature.

4.3 Glass stirring rod.

4.4 Apparatus for measuring the softening point by the “ring-and-ball” method as specified in ISO 4625.

4.5 Apparatus for measuring viscosity, in accordance with ISO 2555.

4.6 Thermometer, complying with type T of ISO 1770.

4.7 Balance, capable of weighing to the nearest 0,1 g.

5 Procedure

WARNING — For reasons of health and safety, conduct the tests in a ventilated space with a fume extraction system.

5.1 Place the stainless-steel or glass vessel (4.1) in the oil bath or oven (4.2) regulated to heat the sample to within ± 2 °C of the operating temperature recommended by the manufacturer of the hot-melt adhesive.

5.2 Add a sufficient quantity of the hot-melt adhesive under test to the vessel. Mix well with the glass stirring rod (4.3) until the sample has melted completely. Measure the temperature by inserting the thermometer (4.7) into the sample. Start the timing from this point. Continue to heat the adhesive for 2 h at the operating temperature ± 2 °C to establish thermal equilibrium.

5.3 Measure, at the operating temperature ± 2 °C, the viscosity in accordance with ISO 2555, making the measurement directly in the stainless-steel or glass vessel, or with the aid of the rotary viscosimeter for measuring viscosity in the molten state. Using the appropriate amount of adhesive, measure the softening point by the "ring-and-ball" method as specified in ISO 4625.

5.4 Repeat all the operations described in 5.3 at regular time intervals of between 4 h and 6 h, until the time, based on pre-determined criteria, for stopping the test is reached. Record the criteria used and the duration of the test in the test report. If skin formation is observed on the surface of the hot-melt adhesive, the skin shall be removed before each subsequent viscosity measurement.

If it is not possible to carry out the tests every 4 h to 6 h, care shall be taken to ensure that any gaps in the timing do not occur towards the end of the useful life of the adhesive.

5.5 At the time that each measurement is taken, observe and record whether or not

- a skin has formed on the surface of the adhesive;
- fumes are being given off;
- phase separation has occurred;
- gelation has occurred;
- sedimentation has occurred;
- any change in colour has occurred.

6 Expression of results

Tabulate the viscosity in pascal seconds and the softening point in degrees Celsius measured after each of a series of heating intervals (in hours) chosen depending on the nature of the hot-melt adhesive and the manufacturer's instructions for use.

An example of a convenient form of expression of results is shown in annex A.

7 Precision

The precision of this test method is not known because inter-laboratory data are not available. When inter-laboratory data are obtained, a precision statement will be added at a subsequent revision.

8 Test report

The test report shall include the following information:

- a) a reference to this International Standard;
- b) all details necessary for complete identification of the hot-melt adhesive tested;
- c) information about sample conditioning;
- d) the test conditions, test temperature, time intervals between measurements, and conditions of measurement of the viscosity (type of spindle and speed) and of the "ring-and-ball" softening point;
- e) the criterion (or criteria) selected to indicate when to stop the test;
- f) the duration of the test;
- g) the tabulated test results as specified in clause 6;
- h) all procedural details that are optional or not covered by this standard, as well as any incidents that may have affected the results.

Annex A

(informative)

Example of expression of results

Identification of the hot-melt adhesive:			
Heating temperature: °C			
Test date:			
Heating time (h)	Viscosity (type of spindle and speed) (Pa·s) ISO 2555	Softening point ("ring- and-ball") (°C) ISO 4625	Remarks in accordance with 5.5

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