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ISO RECOMMENDATION

R 1218

PLASTICS

DETERMINATION OF THE "MELTING POINT" OF POLYAMIDES

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 1218, *Plastics – Determination of the "melting point" of polyamides*, 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. 1218 which was circulated to all the ISO Member Bodies for enquiry in April 1967. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

The following Member Body opposed the approval of the Draft :

United Kingdom

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

ISO Recommendation | R 1218 | April 1970

PLASTICS

DETERMINATION OF THE "MELTING POINT" OF POLYAMIDES

1. SCOPE

1.1 This ISO Recommendation describes two methods for determining the "melting point" of polyamides.

1.2 The "melting point" is an arbitrarily determined temperature which, in fact, lies in a range of temperature which, for the homopolymers, covers a few degrees only.

1.3 The determination of the "melting point", however, is useful for the characterization of polyamide homopolymers. It is of less use for copolymers, which may have a much wider melting range.

1.4 The two methods of test described in this ISO Recommendation give results in close agreement in any one test, and quite good agreement between the two methods.

1.5 Methods different from the two described in this document may give very different results.

METHOD A – HEATING CHAMBER WITH CAPILLARY TUBE

2. PRINCIPLE

Determination of the temperature at which is observed, visually, the change in state of a test specimen of polyamide in a capillary tube enclosed in a heating chamber.

3. MATERIALS

Standards :

3.1 *Bismuth*, analytical quality, melting point 271.3 °C.

3.2 *Tin*, analytical quality, melting point 231.9 °C.

3.3 *Suitable pure compounds* of known melting point.

4. APPARATUS

4.1 *Melting apparatus* consisting of the following items (see Fig. 1) :

(*a*) *cylindrical metal block*, the upper part of which is hollow and forms a chamber;

(*b*) *metal plug*, with two or more holes, which allows a thermometer and one or more capillary tubes to be mounted into the metal block (*a*);

(*c*) *heating system for the metal block* (*a*), provided, for example, by an electrical resistance enclosed in the block;

(*d*) *rheostat* for regulation of the power input, if electrical heating is used;

(*e*) *four windows* of heat-resistant glass on the lateral walls of the chamber, diametrically disposed at right angles to each other. In front of one of these windows is mounted an eye-piece for observing the capillary tube. The other three windows are used for illuminating the inside of the enclosure by means of lamps.

4.2 *Capillary tube* of heat-resistant glass, closed at one end, and of maximum external diameter 2 mm.

4.3 *Precision thermometer* graduated from 20 to 300 °C, in 1 °C intervals.

5. TEST SPECIMEN

5.1 Using a razor blade, take a thin section 5 mm long from the polyamide resin and place it in the capillary tube.

5.2 For a given polyamide two test specimens are generally sufficient.

6. PROCEDURE

6.1 Before using the apparatus for the first time, and whenever the thermometer is changed, calibrate the apparatus following the procedure given in clauses 6.2 to 6.7, but using, instead of the polyamide specimen, one or more of the appropriate standards from section 3 melting close to or covering the range of the melting points to be measured.

6.2 Insert the thermometer in position in the apparatus.

6.3 Insert the capillary tube containing the test specimen in the apparatus and heat with maximum power.

6.4 When the temperature is about 10 °C below the expected melting point, reset the rheostat to reduce the rate of temperature rise to 2 ± 0.5 °C per minute.

6.5 Switch on the illuminating lamps of the apparatus.

6.6 Observe the specimen and record the temperature, indicated by the thermometer, at which the specimen melts. The melting point corresponds to that temperature at which the sharp edges of the specimen disappear.

6.7 Repeat the operations described above using a second test specimen. If there is a difference of more than 5 °C between the two determinations, these results should be disregarded and two additional specimens tested.

7. EXPRESSION OF RESULTS

7.1 Record as "melting point" the arithmetic mean of the temperatures observed in accordance with clause 6.6 for the two test specimens.

7.2 If, when calibrating (with tin and bismuth or another standard of known melting point), a difference of more than 1 °C has been found from the theoretical melting point of the standard, apply a similar correction to the reading recorded for the test specimens.

NOTE. – As the "melting point" of polyamides is a conventional temperature which it is sufficient to know to the nearest 0.5 °C, a correction will not be necessary unless the readings are markedly different. However, if the values for the standard differ by more than 5 °C from the theoretical values, it is necessary to correct the apparatus..

8. TEST REPORT

The test report should include the following information :

(*a*) complete identification of the sample, i.e. type, source of supply, manufacturer's code number, trade name, and any other information which is considered necessary;

(*b*) reference of the method of test used; i.e. Method A of this ISO Recommendation;

(*c*) "melting point", in accordance with section 7;

(*d*) observations of any circumstances which may have affected the result;

(*e*) date of test.

METHOD B – HEATING BLOCK

9. PRINCIPLE

Determination of the temperature at which is observed, visually, the displacement of a meniscus of silicone-oil enclosed between a hot stage and a cover-glass supported by the polyamide test specimen.

10. MATERIALS

Standards :

10.1 *Bismuth,* analytical quality, melting point 271.3 °C.

10.2 *Tin,* analytical quality, melting point 231.9 °C.

10.3 *Suitable pure compounds* of known melting point.

Oil :

10.4 *Silicone-oil* (for example, Dow 510 or any other equivalent grade).

11. APPARATUS

11.1 *Melting apparatus* consisting of the following items (see Fig. 2) :

(*a*) *aluminium block* with a horizontal stage and with a cylindrical horizontal hole allowing a closely fitting thermometer to be positioned so that the bulb of the thermometer is below and close to the stage;

(*b*) *electrical heating system* enclosed in the aluminium block (*a*);

(*c*) *rheostat* for regulation of the power input;

(*d*) *lens* with magnification 3 X to 5 X;

(*e*) *illuminating lamp* with collimator;

(*f*) with certain apparatus, *an additional transparent glass cover* may be used to minimize heat loss.

11.2 *Precision thermometer* graduated from 20 to 300 °C, in 1 °C intervals.

11.3 *Silver wool.*

11.4 *Circular cover-glasses* of about 18 mm diameter.

11.5 *Microtome* or any other means of obtaining thin sections; for example, a razor blade.

11.6 *Sieve* of 800 µm mesh opening.

11.7 *Sieve* of 630 µm mesh opening.

11.8 *Punch* to cut disks of about 1.6 mm diameter (see Fig. 3).

11.9 *Bent dissection needle.*

12. TEST SPECIMEN

12.1 Because the results are markedly affected by the granule size of the polyamide under test, always use specimens of approximately constant size.

12.2 Obtain the specimen by sieving the powdery portion which is almost always present in polyamides; use a granule passing through the 800 μm sieve (11.6) but retained on the 630 μm sieve (11.7).

12.3 If the polyamide sample is not available in the form of powder, cut, by means of the microtome (11.5), a slice of about 0.1 mm thickness. From this slice cut, using the punch (11.8), some disks of about 1.6 mm diameter and divide each disk into four parts by means of the razor blade (11.5). Use one of those quarters as test specimen.

12.4 For a given polyamide sample, use two test specimens.

13. PROCEDURE

13.1 Before using the apparatus for the first time, and whenever the thermometer is changed, calibrate the apparatus following the procedure given in clauses 13.2 to 13.10, but using, instead of the polyamide specimen, one or more of the appropriate standards from section 10 melting close to or covering the range of the melting points to be measured. Care should be taken to interpose a cover-glass (11.4) between the metallic grain and the hot stage when metallic standards are used. This precaution is not necessary with the polyamide specimens since melting temperatures obtained with or without this cover-glass are practically the same.

Carry out one determination for each standard.

13.2 Insert the thermometer (11.2), filling any empty space between the bulb and the walls of the hole with silver wool.

13.3 Place three drops of silicone-oil (10.4) upon the heating stage of the aluminium block (11.1 (*a*)).

13.4 Place the test specimen in the oil.

13.5 Place a cover-glass on the test specimen so that it is supported by the specimen itself and held in a slightly inclined position (see Fig. 4). The oil must not be in contact with the entire surface of the cover-glass but must form a meniscus slightly ahead of the specimen.

NOTE. – When the additional glass cover (11.1 (*f*)) is used, cover the assembly with it.

13.6 Heat the block (11.1 (*a*)) by regulating the rheostat (11.1 (*c*)) to produce a rapid rise in the temperature.

13.7 When the temperature is about 20 °C below the expected melting point, reset the rheostat to reduce the rate of temperature rise to 2 ± 1 °C per minute.

13.8 Switch on the illuminating lamp (11.1 (*e*)) of the apparatus.

13.9 Since at the melting temperature the cover-glass is no longer supported by the specimen, the meniscus of silicone-oil begins to move across the cover-glass.

NOTE. – An optional procedure may be used, in which, as the estimated melting point is approached, and at intervals of 1 °C, very gentle pressure is applied to the cover-glass, by means of the needle (11.9), while observation of the specimen is continued by means of the lens (11.1 (*d*)).

13.10 Read the temperature at which this movement occurs, to an accuracy of ± 0.5 °C.

13.11 Repeat the operations described above using a second test specimen. If there is a difference of more than 5 °C between the two determinations, these results should be disregarded and two additional specimens tested.

14. EXPRESSION OF RESULTS

14.1 Record as "melting point" the arithmetic mean of the temperatures observed in accordance with clause 13.10 for the two test specimens.

14.2 If, when calibrating according to clause 13.1, a difference of more than 1 °C has been found from the theoretical melting point of the standards, apply a similar correction to the reading recorded for the test specimens.

NOTE. – As the "melting point" of polyamides is a conventional temperature which it is sufficient to know to the nearest 0.5 °C, a correction will not be necessary unless the readings are markedly different. However, if the values for the standard differ by more than 5 °C from the theoretical values, it is necessary to correct the apparatus.

15. TEST REPORT

The test report should include the following information :

(*a*) complete identification of the sample i.e. type, source of supply, manufacturer's code number, trade name, and any other information which is considered necessary;

(*b*) reference of the method of test used; i.e. Method B of this ISO Recommendation;

(*c*) "melting point", in accordance with section 14;

(*d*) observations of any circumstances which may have affected the result;

(*e*) date of test.

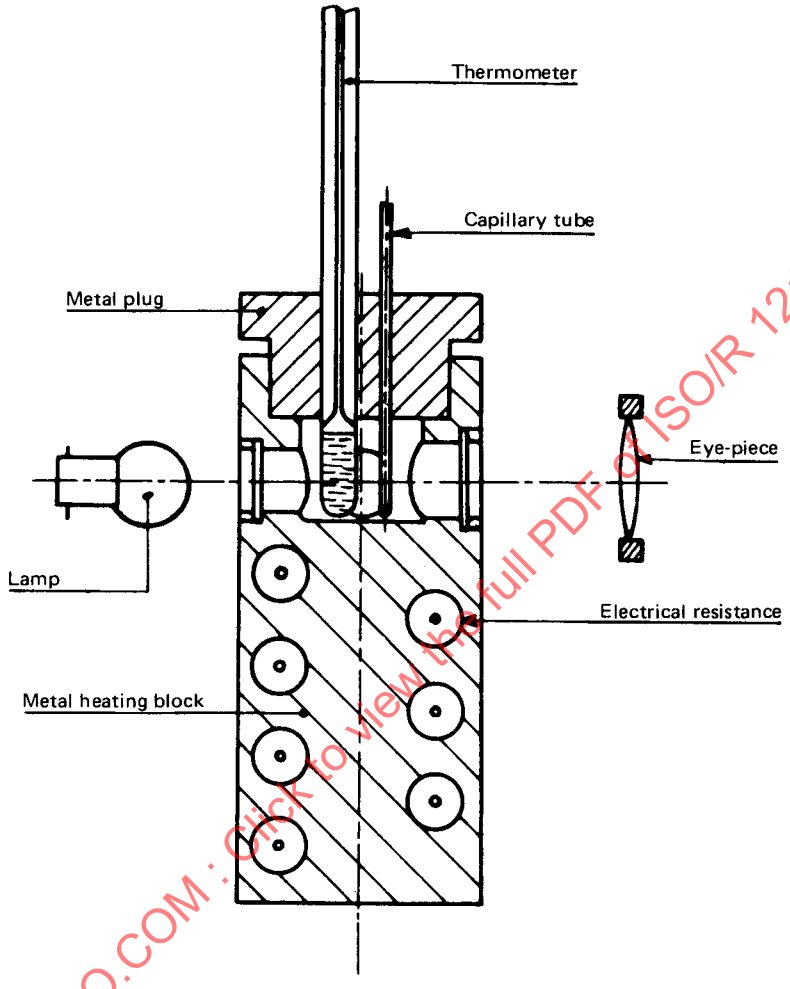

FIG. 1 – Melting apparatus with capillary tube (Method A)