SLOVENSKI STANDARD
SIST EN ISO 11358:1999

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Polimerni materiali – Termogravimetrija polimerov – Splošna načela (ISO 11358:1997)

Plastics - Thermogravimetry (TG) of polymers - General principles (ISO 11358:1997)

Kunststoffe - Thermogravimetrie (TG) von Polymeren - Allgemeine Grundlagen (ISO 11358:1997)

Plastiques - Thermogravimétrie (TG) des polymères - Principes généraux (ISO 11358:1997)

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart,36 B-1050 Brussels

Foreword

The text of the International Standard ISO 11358:1997 has been prepared by Technical Committee ISO/TC 61 "Plastics" in collaboration with Technical Committee CEN/TC 249 "Plastics", the secretariat of which is held by IBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 1997, and conflicting national standards shall be withdrawn at the latest by October 1997.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 11358:1997 was approved by CEN as a European Standard without any modification.

INTERNATIONAL STANDARD

ISO 11358

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Plastics — Thermogravimetry (TG) of polymers — General principles

Plastiques — Thermogravimétrie (TG) des polymères — Principes généraux

Reference number
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Foreword

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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.

International Standard ISO 11358 was prepared by Technical Committee ISO/TC 61, *Plastics,* Subcommittee SC 5, *Physical-chemical properties.*

Annex A of this International Standard is for information only.

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Plastics — Thermogravimetry (TG) of polymers — General principles

1 Scope

1.1 This International Standard specifies the general conditions for the analysis of polymers using thermogravimetric techniques.

1.2 It is applicable to liquids or solids. Solid materials may be in the form of pellets, granules or powders. Fabricated shapes reduced to appropriate specimen size may also be analysed by this method.

1.3 Thermogravimetry can be used to determine the temperature(s) and rate(s) of decomposition of polymers, and to measure at the same time the amounts of volatile matter, additives and/or fillers they contain.

1.4 The thermogravimetric measurements may be carried out in a dynamic mode (mass change versus temperature or time under programmed conditions) or an isothermal mode (mass change versus time at constant temperature).

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was 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 edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 291:—[1], *Plastics — Standard atmospheres for conditioning and testing.*

3 Definitions

For the purposes of this International Standard, the following definitions apply:

3.1 thermogravimetry (TG): A technique in which the mass of a test specimen is measured as a function of temperature or time, while the test specimen is subjected to a controlled temperature programme.

3.2 dynamic mass-change determination: A technique for obtaining a record of the variation of the mass of a test specimen with temperature T which is changing at a programmed rate.

3.3 isothermal mass-change determination: A technique for obtaining a record of the variation of the mass of a test specimen with time t at constant temperature T.

1) To be published. (Revision of ISO 291:1977)

3.4 TG curve: A curve drawn in thermogravimetry by plotting the mass of a test specimen as the ordinate (y-axis) and the temperature T, or time t, as the abscissa (x-axis).

3.5 differential scanning calorimetry (DSC): A technique in which the difference in heat flux (power) into a test specimen and a reference specimen is measured as a function of temperature and/or time while the test specimen and the reference specimen are subjected to a controlled temperature programme.

3.6 differential thermal analysis (DTA): A technique in which the difference in temperature between a test specimen and a reference specimen is measured as a function of temperature and/or time while the test specimen and the reference specimen are subjected to a controlled temperature programme.

3.7 Curie temperature: The temperature at which a ferromagnetic material passes from the ferromagnetic state to the paramagnetic state or *vice versa*.

3.8 sample: A small part or portion of a bulk material or batch of products intended to be representative of the whole.

3.9 test specimen: A complete product or single piece taken from a sample and used to carry out a test. In the case of bulk materials such as pellets, powders and granules: a portion taken from a sample and used to carry out a test.

4 Principle

4.1 A test specimen is heated at a constant rate with a controlled temperature programme, and the change in mass is measured as a function of temperature. Alternatively, the specimen is kept at a given constant temperature and the change in mass is measured as a function of time over a given period.

In general, the reactions which cause the mass of a test specimen to change are decomposition or oxidation reactions or the volatilization of a component. The change in mass is recorded as a TG curve.

4.2 The change in mass of a material as a function of temperature and the extent of this change are indicators of the thermal stability of the material. TG data can therefore be used to evaluate the relative thermal stability of polymers of the same generic family and polymer-polymer or polymer-additive interactions, using measurements made under the same test conditions.

4.3 TG data may be used for process control, process development and material evaluation. Long-term thermal stability is a complex function of service and environmental conditions. TG data alone will not describe the long-term thermal stability of a polymer.

5 Apparatus

A number of commercial instruments suitable for thermogravimetric measurements are available. The basic apparatus components consist of the following:

5.1 Thermobalance, of the null or deflection type. Where the mass of the test specimen is less than 50 mg, the thermobalance shall be capable of measuring the mass with an accuracy of ± 0,020 mg. The thermobalance shall be constructed so that gas flows around the test specimen and permits heat transfer to it at a constant rate.

5.2 Furnace, with a housing of low thermal mass to allow rapid or slow heating and cooling (generally at least 50 °C/min) over a temperature range of ambient to about 1 000 °C.

5.3 Temperature sensor, capable of measuring the temperature of the test specimen. It shall be located as close as possible to the test specimen.

5.4 Temperature programmer, capable of providing a linear rate of scanning over a predetermined temperature range.

5.5 Recording device, capable of recording the specimen mass and temperature and/or time in a way that the relation between mass loss and temperature or time is illustrated. An X-Y recorder is suitable for this purpose.

5.6 Specimen holder, of shape and dimensions sufficient for a mass of at least 5 mg and made of a material capable of withstanding the maximum temperature to be used.

5.7 Purge gas: dry air or oxygen (oxidizing conditions) or a suitable inert gas with an oxygen content of 0,001 % (*V*/*V*) or less (non-oxidizing conditions). In either case, the water content of the purge gas shall be less than 0,001 % (*m*/*m*).

5.8 Flowmeter, capable of measuring gas-flow rates of 50 ml/min to 150 ml/min.

5.9 Balance, capable of measuring the initial mass of the specimen with an accuracy of 0,01 mg.

6 Test specimen preparation

Test specimens may be liquids or solids. The latter may be in the form of powders, pellets, granules or cut pieces. For finished products, the test specimen shall be in the form normally found in use.

6.1 Test specimens from finished products

Cut the test specimen to an appropriate size for the specimen holder. Microtomes or razor blades are suitable for this purpose.

NOTE — Test specimen size and shape will generally be dependent on the sample holder. Surface area will affect the overall results. For instance, in comparing a test specimen of large surface area with a test specimen of smaller surface area, both having the same mass, the smaller surface area test specimen normally changes at a slower rate.

6.2 Test specimen conditioning

Unless otherwise specified, test specimens shall be conditioned, prior to measurement, at 23 °C ± 2 °C and (50 ± 5) % relative humidity in accordance with ISO 291, or by any other method specified by agreement between the interested parties.

6.3 Test specimen mass

The mass of the test specimen shall be greater than 10 mg unless only smaller quantities of material are available.

7 Calibration

7.1 Mass calibration

Without any gas flow through the thermobalance (to prevent any disturbance through buoyancy and/or convection effects), calibrate the thermobalance as follows, using calibrated weights in the range 10 mg to 100 mg:

Zero the thermobalance. Place the calibration weight on the thermobalance and measure the corresponding mass change. If necessary, adjust the thermobalance so that the measured mass is equal to the mass of the calibration weight.