SLOVENSKI STANDARD
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Polimerni materiali - Termogravimetrija (TG) polimerov - 1. del: Splošna načela (ISO/DIS 11358-1:2020)

Plastics - Thermogravimetry (TG) of polymers - Part 1: General principles (ISO/DIS 11358-1:2020)

Kunststoffe - Thermogravimetrie (TG) von Polymeren - Teil 1: Allgemeine Grundsätze (ISO/DIS 11358-1:2020)

Plastiques - Thermogravimétrie (TG) des polymères - Partie 1: Principes généraux (ISO/DIS 11358-1:2020)

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Plastics — Thermogravimetry (TG) of polymers —

Part 1:
General principles

Plastiques — Thermogravimétrie (TG) des polymères —

Partie 1: Principes généraux

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical chemical properties*.

This second edition cancels and replaces the first edition (ISO 11358-1:2014), which has been technically revised. The main changes compared to the previous edition are as follows:

- addition of ISO 472 to the Normative references clause and removal of definitions specified therein;
- addition of measurement under reactive atmosphere;
- revision of apparatus specifications;
- revision of calibration procedures;
- addition of buoyancy correction;
- addition of using a differential thermogravimetric curve.

A list of all parts in the ISO 11358 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Plastics — Thermogravimetry (TG) of polymers —

Part 1: General principles

1 Scope

This document specifies general conditions for the analysis of polymers using thermogravimetric techniques. 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.

This document establishes methods for the investigation of physical effects and chemical reactions that are associated with changes of mass.

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.

Thermogravimetric measurements can be carried out in dynamic mode (mass change versus temperature or time under programmed temperature conditions) or isothermal mode (mass change versus time at constant temperature).

Thermogravimetric measurements can also be carried out using different testing atmospheres, e.g. to separate decomposition in an inert atmosphere from oxidative degradation.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 472, *Plastics — Vocabulary*

ISO 11357-1, *Plastics — Differential scanning calorimetry (DSC) — Part 1: General principles*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and the following apply:

ISO and IEC maintain terminological databases for use in standardisation at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
dynamic mass-change determination
technique for recording the variation of the mass of a test specimen with temperature T which is changing at a programmed rate

3.2
isothermal mass-change determination
technique for recording the variation of the mass of a test specimen with time *t* at constant temperature *T*

3.3
Curie temperature
temperature at which a ferromagnetic material passes from the ferromagnetic state to the paramagnetic state or vice versa

4 Principle

A test specimen is heated at specified rates 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.

During measurement the test specimen is held in a controlled inert, oxidising or otherwise reactive atmosphere.

In general, the reactions which cause the mass of a test specimen to change are decomposition or oxidation reactions or the volatilisation of a component. In some cases, measurements with special reaction gases may also be carried out.

The change in mass vs. temperature and/or time is recorded as a thermogravimetric (TG) curve.

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.

NOTE TG data can 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 cannot be able to 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 consists of the following.

5.1 Thermobalance, meeting the following requirements:

— capability to generate constant heating and cooling rates suitable for intended measurements;

— capability to maintain the test temperature constant (to within ±0,3 K or less for the duration of measurement);

— capability to maintain a constant purge gas flow rate controllable to within ±10 % over the range of flow rates (e.g. 10 ml/min to 150 ml/min) required for intended measurements;

— temperature and mass range in line with experimental requirements;

— recording device capable of automatically recording the measured curve of mass versus temperature and time;

NOTE Some instruments can also display differential thermogravimetric curves (DTG curves) for improved evaluation of results.

— measurement of temperature signals with an accuracy of ±1 % of the absolute temperature measured in K or better;

— measurement of time with an accuracy of ±1 s or better;

— measurement of mass with an accuracy of ±20 µg or better.

5.2 Purge gas, dry air or oxygen (oxidizing conditions) or a suitable inert gas with an oxygen content of 0,001 % by volume or less (non-oxidizing conditions). In either case, the water content of the purge gas shall be less than 0,001 % by mass.

6 Test specimen preparation

6.1 General

Test specimens may be liquids or solids. Solids 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.

6.2 Test specimens from finished products

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

NOTE Test specimen size and shape are generally dependent on the sample holder. Surface area affects 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.

6.3 Test specimen conditioning

Unless otherwise specified in a material specification or product standard, test specimens shall be conditioned, prior to measurement, at one of the standard atmospheres specified in ISO 291, or by any other method specified by agreement between the interested parties.

6.4 Test specimen mass

The mass of the test specimen shall comply with the intended purpose of measurements and shall match the mass range of the thermobalance.

7 Calibration

7.1 Mass calibration

Calibrate the thermobalance using static purge gas conditions (to prevent any disturbance through buoyancy related to gas flow)as follows, using calibrated masses in the intended range of measurements:

Record the temperature at which the mass calibration was carried out.

Zero the thermobalance. Place the calibration weight on the thermobalance and measure the corresponding mass change. In order to span a broader range, several different calibration weights may be used. If necessary, adjust the thermobalance so that the measured mass is equal to the calibration mass.

If mass calibration is done by procedures included in the instrument control software or by external service providers a valid calibration certificate may be acceptable to demonstrate adequate mass calibration.

7.2 Temperature calibration

Carry out the temperature calibration using the same atmosphere, rate of gas flow and heating rate as shall be used in the procedure specified in Clause 8.

If the thermobalance is not coupled with another thermoanalytical method, use the following procedure[1],[2]:

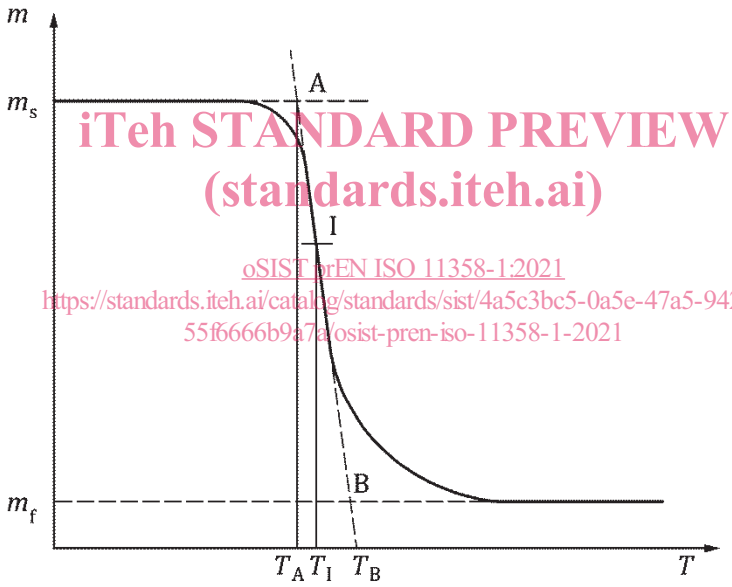
a) Choose two or more calibration materials with a Curie temperature near the temperature range to be examined. If possible, choose the calibration materials in such a way that the temperature range to be examined lies between the Curie temperatures of two of them;

b) Place a suitable magnet above or below the sample as required for the individual thermobalance used. As the strength of the magnetic field can influence the measurement of the Curie temperature, it is recommended to use a low magnetic field strength;

c) Put between 20 mg and 30 mg of calibration material in the sample crucible;

d) Start heating at the same heating rate, same type and material of crucible, and same type and flow of purge gas as will be used in the procedure specified in Clause 8 and carry out a calibration based on the start temperature T_A, inflection-point temperature T_i and end temperature T_B for the Curie temperature transition;

e) Determine the measured Curie temperature from the apparent mass change as shown in Figure 1. If necessary, adjust the thermobalance so that the measured temperature is equal to the declared Curie temperature.



Key

m	mass	A	extrapolated starting point
m_s, m_f	mass of starting point or end point, respectively	B	extrapolated end point
T	temperature	I	inflection point
T_A, T_B, T_I	temperatures of extrapolated starting point (A), extrapolated end point (B) or inflection point (I), respectively		

Figure 1 — Example of Curie temperature calibration

NOTE 1 The Curie point is the temperature at which a ferromagnetic material becomes paramagnetic on heating. The effect is reversible. Applying a magnetic field by placing a magnet below or above the sample exerts a downward or upward force on the ferromagnetic sample. This creates an apparent increase or decrease of weight which is lost upon heating the sample above its Curie temperature. More detailed information on evaluation of the apparent mass change occurring at the Curie point can be found in the literature[3],[4].

NOTE 2 Certified calibration materials traceable to metrology laboratories should be preferably used. Suitable calibration materials may be available via instrument manufacturers or National Metrology Institutes. Examples of suitable magnetic calibration materials are given in Annex A.

