INTERNATIONAL STANDARD

ISO 11358-1

Second edition
2022-03

Plastics — Thermogravimetry (TG) of polymers —

Part 1: General principles

Plastiques — Thermogravimétrie (TG) des polymères —

Partie 1: Principes généraux

Reference number
ISO 11358-1:2022(E)

© ISO 2022



COPYRIGHT PROTECTED DOCUMENT

© ISO 2022

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

© ISO 2022 – All rights reserved

Contents

Page

Foreword ... **iv**

1 Scope ... **1**

2 Normative references ... **1**

3 Terms and definitions ... **1**

4 Principle ... **2**

5 Apparatus ... **2**

6 Test specimen preparation ... **3**
6.1 General ... 3
6.2 Test specimens from finished products ... 3
6.3 Test specimen conditioning ... 3
6.4 Test specimen mass ... 3

7 Calibration ... **3**
7.1 Mass calibration ... 3
7.2 Temperature calibration ... 3

8 Procedure ... **5**
8.1 General ... 5
8.2 Temperature scanning mode ... 5
8.3 Isothermal mode ... 6
8.4 Buoyancy correction ... 6

9 Expression of results ... **6**
9.1 Graphical representation ... 6
9.2 Determination of increase in mass ... 6
9.3 Determination of loss in mass ... 7
9.3.1 Single-stage decrease in mass ... 7
9.3.2 Multi-stage decrease in mass ... 8
9.3.3 Determination of residue ... 11

10 Test report ... **11**

Annex A (informative) **Calibration materials for Curie-point temperature calibration** ... **12**

Bibliography ... **13**

© ISO 2022 – All rights reserved

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.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical chemical properties,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, *Plastics,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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:

— a normative reference to ISO 472 has been added;

— definitions specified in ISO 472 have been removed;

— measurement under reactive atmosphere has been added;

— the apparatus specifications have been changed;

— the calibration procedures have been revised;

— buoyancy correction has been added;

— use of a differential thermogravimetric curve has been added.

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.

© ISO 2022 – All rights reserved

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 can be in the form of pellets, granules or powders. Fabricated shapes reduced to appropriate specimen size can 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.

This document 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.

This document is applicable to measurements in dynamic mode (mass change versus temperature or time under programmed temperature conditions) or isothermal mode (mass change versus time at constant temperature).

This document is applicable to measurements at different testing atmospheres, such as separation of 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*

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

© ISO 2022 – All rights reserved

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 (dynamic mass-change determination). 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 (isothermal mass-change determination).

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 may 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.

© ISO 2022 – All rights reserved