INTERNATIONAL STANDARD

ISO 11358-3

Second edition
2021-01

Plastics — Thermogravimetry (TG) of polymers —

Part 3: Determination of the activation energy using the Ozawa-Friedman plot and analysis of the reaction kinetics

Plastiques — Thermogravimétrie (TG) des polymères —

Partie 3: Détermination de l'énergie d'activation à l'aide du graphique d'Ozawa-Friedman et analyse cinétique de la réaction

Reference number
ISO 11358-3:2021(E)

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Published in Switzerland

Contents

Page

Foreword ..... **iv**
Introduction ..... **v**
1 Scope **1**
2 Normative references **1**
3 Terms and definitions **1**
4 Principle **2**
5 Apparatus **2**
6 Test specimens **2**
7 Mass and temperature calibration **2**
7.1 Mass calibration 2
7.2 Temperature calibration 2
8 Procedure **2**
8.1 General 2
8.2 Non-oxidative reactions 4
8.3 Oxidative reactions 4
9 Expression of results **4**
9.1 Graphical presentation 4
9.2 Determination of activation energy 4
9.3 Master curves 6
10 Precision **9**
11 Test report **9**
Annex A (informative) **Mathematical background** **10**
Bibliography **14**

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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).

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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*.

This second edition cancels and replaces the first edition (ISO 11358-3:2013), which has been technically revised.

The changes compared to the previous edition are as follows:

— the term "conversion" has been deleted;

— a corresponding reference to ISO 11358-2 has been added in Clause 3;

— details of the gas atmosphere in 8.1 and 8.2 have been clarified.

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.

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Introduction

The Ozawa-Friedman plot (logarithm of the rate of mass loss versus the reciprocal of absolute temperature at a given mass loss) is a derivative method that can be applied to data obtained by any mode of temperature change in thermal analysis; e.g. isothermal, constant heating rate, sample-controlled thermal analysis, temperature jump, and repeated temperature scanning.

If controlled rate thermogravimetry (CRTG) is used to study the decomposition of polymers, the Ozawa-Friedman method is typically applied to the analysis of data obtained by CRTG and also to that obtained by the combined use of isothermal thermogravimetry (iso–TG) with conventional linear heating rate thermogravimetry (LHTG), i.e. using a constant heating rate.

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Plastics — Thermogravimetry (TG) of polymers —

Part 3: Determination of the activation energy using the Ozawa-Friedman plot and analysis of the reaction kinetics

1 Scope

This document specifies an analysis method for determining the activation energy using the Ozawa-Friedman plot. It also specifies the preparation of master plots for verification of the reaction kinetics determined by thermogravimetry.

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 11358-1, *Plastics — Thermogravimetry (TG) of polymers — Part 1: General principles*

ISO 11358-2, *Plastics — Thermogravimetry (TG) of polymers — Part 2: Determination of activation energy*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11358-1, ISO 11358-2 and the following apply.

ISO and IEC maintain terminological databases for use in standardization 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
generalized time
t_{gen}

$$t_{gen} = \int_0^t \exp\left(-\frac{E_a}{RT}\right) dt$$

where

E_a is the activation energy, expressed in kJ/mol;

R is the gas constant, expressed as 8,314 J/(mol K);

T is the absolute temperature, expressed in Kelvin;

t is time, expressed in minutes.

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3.2
generalized rate of conversion
dC/dt_{gen}

$$\frac{dC}{dt_{\text{gen}}} = \exp\left(\frac{E_a}{RT}\right)\frac{dC}{dt}$$

Note 1 to entry: For the definition of the degree of conversion, refer to ISO 11358-2.

3.3
master curve
plot that can be used to evaluate the results and investigate the reaction kinetics models

EXAMPLE Conversion versus the generalized time, conversion versus the generalized rate of conversion, generalized time versus the generalized rate of conversion.

4 Principle

Test specimens are heated using any temperature profile and the change in mass is measured as a function of temperature and time. At a given conversion, the logarithm of the rate of conversion is plotted versus the reciprocal of the absolute temperature, and the activation energy is calculated from the slope of the straight line fit to the data thus obtained.

At least two of the master curves enable verification of the reaction kinetics analysis.

5 Apparatus

The apparatus shall be in accordance with ISO 11358-1.

6 Test specimens

Test specimens shall be in the form of powder, pellets, flakes, filaments, or film. The test specimens shall be prepared by cutting the material, as necessary, to a size appropriate for the apparatus (see ISO 11358-1).

7 Mass and temperature calibration

7.1 Mass calibration

The procedure of mass calibration is given in ISO 11358-1.

7.2 Temperature calibration

The procedure of temperature calibration is given in ISO 11358-1.

8 Procedure

8.1 General

The determination of the rate of conversion dC/dt is necessary for the analysis in this document. The rate of conversion versus absolute temperature shall be determined.

See ISO 11358-1 and ISO 11358-2.

Perform tests using either an isothermal run, constant rate heating, sample mass controlled rate thermal analysis, temperature jump, repeated temperature scanning, or any combination of the above,

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