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Plastics — Differential scanning calorimetry (DSC) or thermogravimetric analysis (TGA) — Model-free kinetics based on the non-linear incremental isoconversional method

Plastiques — Analyse calorimétrique différentielle (DSC) ou analyse thermogravimétrique (TGA) — Analyse cinétique sans modèle basée sur la méthode isoconversionnelle incrémentale non-linéaire

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Foreword

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Introduction

Conventional thermoanalytical methods for the characterisation of reaction kinetics, such as those described in ISO 11357-7, ISO 11358-2[1] or ISO 11358-3[2], are applicable only for constant values of the activation energy. Furthermore, a reaction model has to be assumed. In case of, for example, competing or side reactions, the assumption of a constant activation energy is not applicable. Furthermore, the description of a chemical reaction can also include processes, such as diffusion or evaporation.

Another method is specified in IEC/TS 60216-7-1[3] in which the thermal endurance of electric insulating materials is predicted based on thermal analysis methods for the evaluation of the activation energy and a lifetime test. However, this method is limited to ageing reactions where one single reaction is predominant.

To cope with these fundamental limitations, model-free kinetics have been proposed. Instead of a reaction model and a constant activation energy, it makes use of an activation energy which depends on the extent of conversion. This conversion-dependent activation energy is not an activation energy in the traditional sense but rather an apparent activation energy describing the chemical reaction(s) as well as any transport processes. This approach is based on the isoconversional principle which assumes that the reaction rate at a certain conversion depends only on the temperature[4][5][6]. For the temperature dependence of the reaction rate the Arrhenius approach can be applied. Thus, the activation energy as a function of the conversion can be calculated from several DSC or TGA measurements of a reaction done at different temperature programs. The determination of the apparent activation energy as a function of the conversion is sufficient to make predictions, i.e. there is no need for an explicit reaction model[6].

More advanced methods of the model-free kinetics approach can be applied to any temperature program, i.e. constant scan rate, isothermal or arbitrary temperature variation measurements[4][8][9].

However, model-free kinetics is not an all-purpose tool. Misleading results can be obtained especially for more complex reactions. In this case, the results and in particular the predictions should be checked for plausibility and verified.

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Plastics — Differential scanning calorimetry (DSC) or thermogravimetric analysis (TGA) — Model-free kinetics based on the non-linear incremental isoconversional method

1 Scope

This document establishes a model-free approach for the isoconversional kinetic analysis of chemical reactions and phase transitions. The isoconversional principle is based on the assumption that the reaction rate at a given conversion depends solely on the temperature, i.e. is independent on the scan rate.

This document establishes a method for the determination of the activation energy as a function of the conversion of the reaction or transition.

The method is applicable to dynamic and isothermal temperature program measurements done by differential scanning calorimetry (DSC) or thermogravimetry (TG). It can be applicable to other measurement techniques, too, that enable the determination of conversion curves.

The method is applicable to the prediction of the kinetic behaviour of reactions and transitions even in temperature regions that are not experimentally accessible.

The method is not applicable in the glassy state of polymers.

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 472, *Plastics — Vocabulary*

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

ISO 11357-5, *Plastics — Differential scanning calorimetry (DSC) — Part 5: Determination of characteristic reaction-curve temperatures and times, enthalpy of reaction and degree of conversion*

ISO 11357-7, *Plastics — Differential scanning calorimetry (DSC) — Part 7: Determination of crystallization kinetics*

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

ISO 23976, *Plastics — Fast differential scanning calorimetry (FSC) — Chip calorimetry*

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 terminology databases for use in standardization at the following addresses:

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

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

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