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# Plastics — Test method for estimation of the short chain branching distribution of semicrystalline ethylene 1-olefin copolymers — Differential scanning calorimetry (DSC)

*Plastiques — Méthode d'essai pour l'estimation de la distribution des ramifications à chaînes courtes des copolymères d'éthylène-1-oléfines semi-cristallins — Analyse calorimétrique différentielle (DSC)*

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# Foreword

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

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# Introduction

The short chain branching distribution (SCBD) of polyethylene is a fundamental structural parameter, which, together with the molecular weight distribution, determines the properties of polyethylene materials. It is emphasized that with the same type and amount of 1-olefin comonomer, the physical-mechanical behaviour of the polyethylene can be significantly changed by adjusting the SCBD, and then affects the properties of final products. Therefore, it is very important to obtain information about the SCBD to characterize polyethylene in detail. Separation techniques are successfully applied for quantitative determination of the SCBD of polyethylene, which require specific instrumentation, being sometimes laborious or time consuming.[1] However, these solvent separation techniques cannot distinguish intra-molecule heterogeneity. The thermal fractionation technique of successive self-nucleation and annealing (SSA) can be used to distinguish the polymer chains with different branching degrees from the same molecule or different molecules and was first reported by Muller et al. in 1997 using conventional DSC.[2] Using high performance DSC combined with reduced sample mass scan rates up to 200 K/min can be achieved maintaining good peak resolution. This enabled reduction of SSA fractionation times from several hours to approximately 30 min.[3] More recent developments in chip calorimetry enabled much higher scan rates in the order of thousands of K/s and allow observation of polymer crystallisation at a much shorter time scale with similar resolution compared to conventional DSC. This technique does not only offer a significant reduction of required time but enables also additional information on very early stages and molecular mechanisms of thermal fractionation.[4]

The final differential scanning calorimetry (DSC) heating run of the polymer after SSA treatment shows a series of melting peaks. The multiple melting peaks are derived from the melting of crystallites with different mean lamellar thicknesses, which presumably correspond to the chain segments with different crystallizable sequences or degrees of branching.[5],[6] Under the premise of ignoring the co-crystallization of the melt at self-nucleation temperature,[7],[8],[9],[10] the degree of branching and crystallinity of each fraction as a function of melting temperature are determined from empirically-derived equations.[5],[11] Therefore, the amount of chain segments with different branching degrees can be characterized according to the integral area of multiple melting peaks after considering the degree of crystallinity, and the SCBD can be obtained.[5],[11],[15] SSA establishes a method for the estimation of the chemical composition distribution excluding amorphous highly branched components that do not crystallize up to the minimum crystallization temperature selected for experiments. The main advantages of the SSA thermal fractionation method are simple, fast, eco-friendly and cost-less.

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# Plastics — Test method for estimation of the short chain branching distribution of semicrystalline ethylene 1-olefin copolymers — Differential scanning calorimetry (DSC)

## 1 Scope

This document specifies a test method for the estimation of the short chain branching distribution of semicrystalline ethylene 1-olefin copolymers by successive self-nucleation and annealing (SSA) with conventional, high performance and fast differential scanning calorimetry.

This document is applicable to the estimation of the short chain branching distribution of raw materials of semicrystalline ethylene 1-olefin copolymers and its products.

Quantitative calculation of short chain branching content, degree of crystallinity and short chain branching distribution is applicable to ethylene/1-butene copolymers, ethylene/1-hexene copolymers and ethylene/1-octene copolymers.

## 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 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, ISO 11357-1, ISO 23976 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/

**3.1**
**short chain branching**
oligomeric offshoot branching from the backbone chain of a polymer

**3.2**
**degree of branching**
*F*
number of short chain branches per 1 000 backbone carbon atoms

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