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Title: Draft BS ISO 18177 Plastics - Test method for estimation of the short chain branching distribution of semicrystalline ethylene 1-olefin copolymers by differential scanning calorimetry (DSC).

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## Introduction

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For international standards, comments will be reviewed by the relevant UK national committee before sending the consensus UK vote and comments to the international committee, which will then decide appropriate action. If the international standard is approved, it is usual for the text to be published as a British Standard.

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## Template for comments and secretariat observations

|                  |                        |
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| 1  | 2                                          | (3)                          | 4               | 5                                                                                                                                  | (6)                                                                      | 7                                                  |
|----|--------------------------------------------|------------------------------|-----------------|------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------|----------------------------------------------------|
| MB | Clause No./ Subclause No./Annex (e.g. 3.1) | Paragraph/ Figure/Table/Note | Type of comment | Comment (justification for change) by the MB                                                                                       | Proposed change by the MB                                                | Secretariat observations on each comment submitted |
|    | 3.1                                        | Definition 1                 | ed              | Definition is ambiguous and needs clarifying.                                                                                      | Amend to read '...so that the mains connector to which no connection...' |                                                    |
|    | 6.4                                        | Paragraph 2                  | te              | The use of the UV photometer as an alternative cannot be supported as serious problems have been encountered in its use in the UK. | Delete reference to UV photometer.                                       |                                                    |



# DRAFT International Standard

## ISO/DIS 18177

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

ICS: 83.080.20

ISO/TC 61/SC 9

Secretariat: **KATS**

Voting begins on:  
**2024-05-16**

Voting terminates on:  
**2024-08-08**

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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](http://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](http://www.iso.org/iso/foreword.html).

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

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](http://www.iso.org/members.html).

## 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<sup>[1]</sup>. 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<sup>[2]</sup>. Using high performance DSC combined with reduced sample mass scan rates up to 200 K/min could be achieved maintaining good peak resolution. This enabled reduction of SSA fractionation times from several hours to approx. 30 minutes<sup>[3]</sup>. 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<sup>[4]</sup>.

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<sup>[5,6]</sup>. Under the premise of ignoring the co-crystallization of the melt at self-nucleation temperature<sup>[7-13]</sup>, the degree of branching and crystallinity of each fraction as a function of melting temperature are determined from empirically-derived equations <sup>[5,14]</sup>. 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<sup>[5,14,18]</sup>. The main advantages of the SSA thermal fractionation method are simple, fast, eco-friendly and cost-less.



# Plastics — Test method for estimation of the short chain branching distribution of semicrystalline ethylene 1-olefin copolymers by 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. It is also applicable to ethylene-propylene copolymers.

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.

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 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 <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