INTERNATIONAL STANDARD

ISO 11235

Third edition
2023-07

Rubber compounding ingredients — Sulfenamide accelerators — Test methods

Ingrédients de mélange du caoutchouc — Accélérateurs de type sulfénamide — Méthodes d'essai

Reference number
ISO 11235:2023(E)

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Published in Switzerland

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Contents

Page

Foreword ..... **v**

1 Scope **1**

2 Normative references **1**

3 Terms and definitions **2**

4 Determination of physical and chemical properties **2**
4.1 Sampling 2
4.2 Test methods 2
4.3 Limit of acceptance 2

5 Test methods for purity **3**
5.1 Method to determine purity by reduction with MBT and titration 3
5.1.1 Purpose 3
5.1.2 Principle 3
5.1.3 Reagents 3
5.1.4 Apparatus 4
5.1.5 Procedure 4
5.1.6 Expression of results (methods A and B) 5
5.2 Method to determine purity by high performance liquid chromatography (HPLC) 6
5.2.1 Purpose 6
5.2.2 Principle 7
5.2.3 Significance and use 7
5.2.4 Interferences 7
5.2.5 Reagents and materials 7
5.2.6 Apparatus 7
5.2.7 Calibration and standardization 8
5.2.8 Procedure 8
5.2.9 Sample analysis 9
5.2.10 Expression of results 9
5.3 Precision 10

6 Test method for insoluble material **10**
6.1 Purpose 10
6.2 Principle 10
6.3 Significance and use 10
6.4 Reagents 11
6.5 Apparatus 11
6.6 Procedure 12
6.7 Expression of results 12

7 Test methods for melting range **12**
7.1 Melting range by capillary tube 12
7.1.1 Purpose 12
7.1.2 Significance and use 12
7.1.3 Limitations 13
7.1.4 Apparatus 13
7.1.5 Preparation of test sample 13
7.1.6 Procedure 13
7.2 Melting range by differential scanning calorimetry (DSC) 14
7.2.1 Purpose 14
7.2.2 Significance and use 14
7.2.3 Limitations 14
7.2.4 Apparatus 14
7.2.5 Preparation of test sample 14
7.2.6 Procedure 14

8 Test method for volatile material **15**

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8.1 Purpose 15
8.2 Principle 15
8.3 Apparatus 15
8.4 Procedure 16
8.5 Expression of results 16

9 Test method for wet sieve analysis 16
9.1 Purpose 16
9.2 Significance and use 16
9.3 Materials 17
9.3.1 Liquid detergent, neutral. 17
9.4 Apparatus 17
9.5 Procedure 17
9.6 Expression of results 17

10 Test method for the determination of ash 18
10.1 Purpose 18
10.2 Principle 18
10.3 Significance and use 18
10.4 Apparatus 18
10.5 Procedure 19
10.6 Expression of results 19

11 Test report 19

Annex A (informative) **Classification and key properties of sulfenamide (class 1) vulcanization accelerators 21**

Annex B (informative) **Precision 24**

Bibliography 26

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Foreword

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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 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This third edition cancels and replaces the second edition (ISO 11235:2016), which has been technically revised.

The main changes are as follows:

— errors in Formula (1) and Formula (2) have been corrected;

— the CAS Registry Number®[1)] has been added for each chemical;

— the usage of auto titrator with electrode has been added in Clause 5;

— Annex A has been changed from “normative” to “informative”.

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.

1) CAS Registry Number® is a trademark of CAS corporation. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

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Rubber compounding ingredients — Sulfenamide accelerators — Test methods

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to determine the applicability of any other restrictions.

1 Scope

This document specifies the methods to be used for the evaluation of sulfenamide accelerators:

— MBTS: benzothiazyl disulphide;

— CBS: *N*-cyclohexylbenzothiazole-2-sulfenamide;

— TBBS: *N*-*tert*-butylbenzothiazole-2-sulfenamide;

— DIBS: *N*,*N*'-diisopropylbenzothiazole-2-sulfenamide;

— DCBS: *N*,*N*'-dicyclohexylbenzothiazole-2-sulfenamide;

— MBS: *N*-oxydiethylenebenzothiazole-2-sulfenamide.

NOTE 1 Although MBTS is not a sulfenamide, it is the primary decomposition product of these accelerators and quantitatively determined by the method specified in 5.2.

The analytical methods are applicable for most commercial sulfenamide accelerators:

— sulfenamides of primary amines (type I);

— sulfenamides of unhindered secondary amines (type II);

— sulfenamides of hindered secondary amines (type III).

NOTE 2 Classification and key properties of sulfenamide accelerators are described in Annex A.

The method (5.2) to determine purity by high performance liquid chromatography (HPLC) is the preferred method.

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 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 1772, *Laboratory crucibles in porcelain and silica*

ISO 3819, *Laboratory glassware — Beakers*

ISO 4788, *Laboratory glassware — Graduated measuring cylinders*

ISO 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*

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ISO 6556, *Laboratory glassware — Filter flasks*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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
lot sample
sample from production representative of a standard production unit, normally referred to as "the sample"

3.2
test portion
actual material, representative of the lot sample, used for a particular determination

4 Determination of physical and chemical properties

4.1 Sampling

The sampling of the product shall be performed in accordance with ISO 15528.

To ensure homogeneity, thoroughly blend at least 250 g of the lot sample before removing the test portion.

4.2 Test methods

The test methods list of sulfenamide accelerators is shown in Table 1.

Table 1 — List of the test methods

Property	Clause or subclause of this document
Purity by reduction with MBT and titration	5.1
Purity by high performance liquid chromatography (HPLC)	5.2
Insoluble material	6
Melting range by capillary tube	7.1
Melting range by differential scanning calorimetry (DSC)	7.2
Volatile material	8
Wet sieve analysis	9
Ash	10

4.3 Limit of acceptance

The difference between the results of duplicate determinations shall not exceed the repeatability of the test, if it is defined. Otherwise, it is necessary to repeat the test. When the repeatability is not defined, the results of both determinations shall be reported.

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