
**Rubber and rubber
products — Determination of
2-mercaptobenzothiazole content
by high performance liquid
chromatography (HPLC)**

*Caoutchouc et produits à base de caoutchouc — Détermination de la
teneur en 2-mercaptobenzothiazole par chromatographie en phase
liquide haute performance*



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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 ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

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.

Introduction

2-Mercaptobenzothiazole (sometimes also referred to as: MBT; 2-MBT; 2-benzothiazolethione, or BTSH) is used in the rubber industry as a curing agent. MBT is in the group of thiazoles and is considered as scorch fast when used as a primary accelerator.

2-Mercaptobenzothiazole as the acidic sulfur accelerator is widely used in rubber materials because of its good characteristics: stable sulfides, good vulcanization, and a low critical temperature to accelerate vulcanization so that the rubber product can reach higher tensile strength and hardness levels.

Measuring 2-mercaptobenzothiazole concentration in rubber compounds at different stages of curing the rubber product is an excellent means to define the optimal curing conditions of temperature and time in order to obtain the right properties for the products at the best cost.

During the curing of rubber compounds sulfenamides are used as accelerators, which chemically react at an early stage of the curing to produce 2-mercaptobenzothiazole and other species. 2-Mercaptobenzothiazole then contributes to the initiation of the mechanism which creates the sulfur crosslinks between the rubber macromolecules at the end (an example is given in [Figure A.1](#)). To ensure continuous progress, it is important to know the chemical mechanisms involved at each stage. Thus, it is necessary to quantify the content of 2-mercaptobenzothiazole during the decomposition of the sulfenamide and to know whether 2-mercaptobenzothiazole has disappeared in any further chemical reactions.

Rubber and rubber products — Determination of 2-mercaptobenzothiazole content by high performance liquid chromatography (HPLC)

1 Scope

This document specifies a quantitative test method to determine the 2-mercaptobenzothiazole content in rubber and rubber products by high performance liquid chromatography (HPLC).

This document delivers a method for quantifying 2-mercaptobenzothiazole in rubber products for a better selection of curing conditions.

This document provides a method to follow the curing of rubber with sulfur- and benzothiazole-based accelerators using a chemical measurement which is complementary to the classical rheometric technique.

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 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 4661-2, *Rubber, vulcanized — Preparation of samples and test pieces — Part 2: Chemical tests*

3 Terms and definitions

No terms and definitions are listed in this document.

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/>

4 Principle

The 2-mercaptobenzothiazole in rubber is ultrasonically extracted with a chloroform-methanol solution determined and confirmed with HPLC-DAD (high performance liquid chromatography equipped with a diode-array detector).

Note There is a risk of neo-formed MBT from MBTs if a thiazole accelerator is used in the formulation of the sample.

5 Reagents and materials

Unless otherwise specified, analytical grade chemicals should be used. Water shall be distilled or deionized to fulfil grade 3 in accordance with ISO 3696.

5.1 Methanol, of analytical grade.