
**Rubber, vulcanized — Preparation of
samples and test pieces —**

**Part 2:
Chemical tests**

*Caoutchouc vulcanisé — Préparation des échantillons et
éprouvettes —*

Partie 2: Essais chimiques

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

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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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This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This second edition cancels and replaces the first edition (ISO 4661-2:1987), which has been technically revised.

The main changes compared to the previous edition are as follows:

- updating of the format of the document in regard to [Clauses 2](#) and [3](#);
- replacement of chlorinated solvents with less hazardous solvents in [4.3](#).

A list of all parts in the ISO 4661 series can be found on the ISO website.

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

When preparing samples of vulcanized rubber for chemical testing, care should be taken that any test portion is representative of the sample with respect to the property or constituent to be determined. Thus, if it is desired to deduce the composition of the original mix, any surface bloom should be incorporated, but if the final bulk composition is required, bloom should be removed preferably by using mechanical means. In the case of tests carried out with test pieces taken from a manufacturer's product, it might be necessary first to separate the desired vulcanized rubber from other possible components of the manufactured products, such as adjacent rubber compounds of different composition, metals, threads, tapes, and fabrics as well as coatings or veneers that cover it. This separation should be made using, whenever possible, mechanical means — blades, abrasive wheels, files, etc. — and avoiding any heat generation.

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Rubber, vulcanized — Preparation of samples and test pieces —

Part 2: Chemical tests

1 Scope

This document specifies a method of preparing samples from vulcanized rubber for use in chemical tests.

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 1407:2011, *Rubber — Determination of solvent extract*

3 Terms and definitions

No terms and definitions are listed in this document.

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 <http://www.electropedia.org/>

4 Preparation of samples

4.1 Soft vulcanized rubber

The sample shall be comminuted with scissors, a rotating rasp, a suitable grinder or by cryogenic crushing to pass a sieve with an approximately 1,7 mm opening. Alternatively, it shall be sheeted to a thickness not exceeding 0,5 mm by passing between cold, tightly closed rolls of a laboratory mill. The type of grinder or mill used is immaterial, provided that the sample does not become contaminated or unduly heated.

4.2 Ebonite

The sample shall be rasped to powder which will pass a sieve with an approximately 400 µm opening. The powder shall be treated with a magnet to remove any iron particles.

4.3 Rubberized composites

Where it is not possible to separate the rubber mechanically, the following procedure shall be carried out.

The rubber shall be separated by exposing it to the vapour only of a suitable solvent. For composites based on NR, SBR and BR, acetone or ETA (ethanol-toluene azeotrope) will be suitable solvents. For other types of rubber, refer to ISO 1407:2011 (Annex A) for suitable solvents.

The time of exposure should be kept as short as possible to avoid any possibility of extracting plasticizers from the rubber compound.

Afterwards, the swollen rubber shall have the solvent completely removed in air at room temperature and treated as described in [4.1](#).

In cases where the rubber is chemically bonded to the substrate, it should be noted that the composition of the rubber in the region of the bond can be substantially different from that in the bulk of the material.

In all cases, sampling shall be appropriate to the test being carried out. Even if the substrate can be removed "clean", the remaining rubber might consist of more than one formulation, and the blending carried out subsequently in accordance with [4.1](#) can result in a sample for analysis which is not representative of any one original formulation. Such composite layers can be revealed by microscopic examination of a series of cross-sections of the material.

It will then be possible, by careful buffing or cutting, to produce a sample of one or more rubber components for individual analysis.

Where it is impossible to separate the rubber from the substrate, the material shall be cut into small cubes to pass a sieve with 2 mm openings, and analysed as a whole.

In this case, the proportion by mass of rubber in the composite can be determined by disintegration of the rubber in a hot high boiling solvent, and weighing before and after the treatment. It should be noted that there might be partial dissolution of any organic material in the composite (for example, fabric), and the results should be interpreted with caution.

Where available, a sample of the non-rubber portion of the composite should also be analysed similarly.

In reporting the results of any analysis, the method of separation used shall be specified. If separation was impossible, it shall be made clear that the sample analysed was a heterogeneous blend of rubber and substrate and the possibility of error due to inhomogeneity or inadequate separation of materials should be pointed out.