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

**Part 2 :**  
**Chemical tests** **STANDARD PREVIEW**  
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*Caoutchouc vulcanisé — Préparation des échantillons et éprouvettes —*

[ISO 4661-2:1987](#)

*Partie 2 : Essais chimiques*

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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4661-2 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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

## Part 2 : Chemical tests

### 0 Introduction

International Standard ISO 4661 for the preparation of samples and test pieces for rubber testing comprises two parts :

- Part 1 : Physical tests.
- Part 2 : Chemical tests.

This part of ISO 4661 includes a number of factors of importance in the preparation of samples for chemical testing, in order to ensure the best use of the relevant ISO methods of test.

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 manufacturer's products, it may be necessary first to separate vulcanized rubber from other possible components of the manufactured products, such as metals, threads, tapes, fabrics as well as coatings or veneers that may cover it. This separation should be made using whenever possible mechanical means — blades, abrasive wheels, files, etc. — and avoiding any heat generation.

### 1 Scope and field of application

This part of ISO 4661 specifies a method of preparing samples from vulcanized rubber for use in chemical tests.

### 2 Preparation of samples

#### 2.1 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.

#### 2.2 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.

#### 2.3 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, methylene chloride or 1,1,1-trichloroethane are suitable solvents.

NOTE — 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 2.1.

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 may 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 may consist of more than one formulation, and the blending carried out subsequently in accordance with 2.1 may result in a sample for analysis which is not representative of any one original formulation. Such composite layers may be revealed by microscopic examination of a series of cross-sections of the material.

It may 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 a 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 may 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 may 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.

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