
**Rubber compounding ingredients —
Precipitated silica — Determination
of aggregate size distribution by disc
centrifuge**

*Ingrédients de mélange du caoutchouc — Silice précipitée —
Détermination de la distribution dimensionnelle par à disque
centrifuge*



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Published in Switzerland

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

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

The determination of the aggregate size distribution (ASD) of silica by disc centrifuge photosedimentometry can be used for characterizing and specifying these products. It is well accepted that the aggregate size distribution of silica could have an influence on the performance of these materials used in different applications. Therefore, a standardized procedure regarding the sampling preparation and the testing of the aggregate size distribution seems to be necessary in order to compare, discuss and interpret received results between the laboratories using this method.

See [Annex A](#) for physical principles of measurement.

Rubber compounding ingredients — Precipitated silica — Determination of aggregate size distribution by disc centrifuge

1 Scope

This document specifies a general method for determining the aggregate size distribution (ASD) of silica by using a disc centrifuge according to the principle of sedimentation. As pre-stage the silica is de-agglomerated in water using strong ultrasonic power treatment.

The method is used for precipitated silica.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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 <http://www.electropedia.org/>

3.1

silica aggregate

discrete, rigid colloidal entity that is the smallest dispersible unit in a suspension

Note 1 to entry: In comparison to carbon black^[3] the term silica aggregate is less defined and has to be seen always in context with the silica treatment (i.e. ultrasonic power in a silica suspension in water). The references apply to carbon black but are also broadly used by the rubber industry for precipitated silica.

4 Significance and use

A disc centrifuge is used for measuring the aggregate size distribution (ASD) of precipitated silica. As a function of test time and rotational speed, aggregate sizes in the range of approximately 5 nm to 100 µm can be analysed according to the principle of sedimentation. Firstly, the silica sample is dispersed in an aqueous medium by using ultrasonic power treatment. Afterwards, the suspension is transferred to the disc centrifuge and separated according to its aggregate size. The sedimentation is accelerated by centrifugal forces generated by the rotation of the centrifuge. By using a density gradient of sucrose solution the sedimentation can be stabilised. Over the course of the experiment, a separation in different silica aggregate sizes is possible and can be evaluated.

For investigations between different laboratories, it is recommended to use the IRM 100 silica standard¹⁾ according to ASTM D5900 (see [Clause 7](#)).

1) IRM 100 silica standard is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product. The IRM 100 silica standard can be provided from Balentine Enterprises, INC. (www.irmsilicastandard.com).