



Designation: D8471 – 22a

Standard Practice for Silica—Reference Compound¹

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1. Scope

1.1 This practice covers the standard raw materials, formulation, and mixing procedure for the evaluation and quality control of silica and related technologies, such as silanes and functionalized polymers, in a standardized rubber compound.

1.2 *Units*—The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D1566 Terminology Relating to Rubber

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

D5900 Specification for Physical and Chemical Properties of Industry Reference Materials (IRM)

3. Terminology

3.1 The following terms are defined in accordance with Terminology **D1566**.

¹ This practice is under the jurisdiction of ASTM Committee **D11** on Rubber and Rubber-like Materials and is the direct responsibility of Subcommittee **D11.20** on Compounding Materials and Procedures.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.2 Definitions:

3.2.1 *silanization, n*—chemical reaction of organosilane molecules with the silanol groups of synthetic amorphous silica covering its surface and improving its compatibility with rubber leading to an enhanced dispersion.

3.2.2 *silica (as related to rubber technology), n*—nanostructured, synthetic amorphous silicon dioxide (SAS) typically based on primary particles bound to form aggregates and agglomerates as secondary particles, covered by silanols and siloxanes, and produced either by conversion to a solid from a supersaturated solution of silicate salts in water (precipitated SAS), or by a pyrogenic process in a flame (fumed SAS).

4. Summary of Practice

4.1 This practice shall be used for specific procedures used in silica technology for rubber compounds. A reference compound is required for quality control of production, for research and development purposes, and for comparison of different materials.

5. Significance and Use

5.1 This practice may be used to characterize silica or silane in terms of specific properties of the standard compound. This practice is useful for the quality assurance of silica production. It may also be used for the preparation of reference compounds for compounding experiments, to confirm the day-to-day reliability of testing operations used in the rubber industry, for the evaluation of experimental compounds, and for quality control of production compounds.

6. Standard Test Formula

6.1 The standard formula in **Table 1** has been formulated for a silica with a CTAB surface area of 160 m²/g using TESPD silane, and for a S-SBR 2548 oil extended with 37.5 phr TDAE. If different silica grades or silane types are used, adjustments of the recipe might be required.

6.2 NR is needed to increase mixing efficiency for this formula. Both Standard Malaysian Rubber grade L (SMR L) and Standard Thai Rubber grade L (STR L) are recommended to give satisfactory performance. Other sources of rubber should be previously checked to ensure that results are equivalent.

TABLE 1 Standard Formula

Material	Quantity, parts by mass ^E
S-SBR 2548, 37.5 TDAE ^A	123.75
NR	10.00
Silica, precipitated	95.00
ZnO	2.50
Stearic acid	2.50
Antioxidant (6PPD ^B)	2.00
Wax	2.00
Silane	6.84
DPG ^C	2.00
CBS ^D	2.00
Sulfur, soluble	2.00
Total:	250.59

^A Treated Distillate Aromatic Extract.

^B N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylenediamine.

^C 1,3-Diphenylguanidine.

^D N-Cyclohexylbenzothiazol-2-sulphenamide.

^E Weigh rubber and silica to the nearest 1 g, sulfur and accelerator to the nearest 0.02 g, and all other compounding materials to the nearest 0.1 g.

6.3 The use of Industry Reference Materials (IRM) as specified in Specification **D5900** is recommended for standard rubber test formulas.³

³ The current list of Industry Reference Materials for rubber can be found on the webpage of the Committee D11 under the section Additional Information (www.astm.org/COMMITTEE/D11).

7. Mixing Procedure

7.1 The mixing procedure for a Standard Internal Mixer according to Practice **D3182** is described in **Table 2** (times in seconds).

7.2 If internal mixers of other sizes are used, adjustments of batch masses, ram pressure, rotor speeds or mixing times will be required to obtain equivalent results. The use of other mixer sizes shall be recorded with the reported data. For general information regarding mixing procedures see Practice **D3182**.

8. Keywords

8.1 formulation; mixing procedure; recipe; silane; silanization; silica