
**Rubber compounding ingredients —
Silica — Oil absorption of
precipitated silica**

*Ingrédients de mélange du caoutchouc — Silice — Absorption d'huile
des silices précipitées*



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Contents

	Page
Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Principle	1
4 Materials	1
5 Equipment	2
6 Sampling	2
7 Procedure	2
7.1 Preliminary note.....	2
7.2 Determination.....	3
7.2.1 Method A (powder sample, micro-perls).....	3
7.2.2 Method B (granulated samples).....	4
7.3 Evaluation.....	4
7.3.1 Evaluation for powder and micro-perl materials.....	4
7.3.2 Evaluation of granulated materials.....	5
8 Precision data	5
9 Test report	5
Annex A (normative) Normalization of the mixer chamber by using reference materials	6
Annex B (informative) Precision data	12
Bibliography	14

Foreword

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The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

Introduction

Due to health and environmental safety precautions, the determination of DOA absorption number has been worked out to substitute the determination of the DBP absorption number.

Dibutylphthalate (DBP) and dioctylphthalate (DOP) were commonly used in the past for determining the absorption capacity of pigments and extenders, like carbon black and silica. In the meantime, both substances have been banned as carcinogenic, mutagenic, reprotoxic substances (CMR) in different countries.

The search of a suitable alternative for DBP and DOP, especially for measuring the absorption capacity of polar pigments and extenders, like silica, calcium silicates and sodium aluminium silicates has been carried out in a task group of the Association of Synthetic Amorphous Silica Producers (ASASP) between 2004-2008. Out of different tested liquids, like linseed oil, paraffinic oil, etc., DOA was found as the most suitable alternative which leads to evaluated absorption numbers close to DBP measurement.

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1 Scope

This International Standard specifies a general method for determining the liquid absorption capacity of a pigment and extender by using di-(2-ethylhexyl) adipate (DOA, CAS 103-23-1). The determination of the DOA absorption number is performed by means of an absorptometer which is equipped with a torque measurement and processing system. The DOA absorption number provides an indication of the void volume formed by the aggregates and agglomerates of the pigments and extenders.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 787-2, *General methods of test for pigments and extenders — Part 2: Determination of matter volatile at 105° C*

ISO 787-11, *General methods of test for pigments and extenders — Part 11: Determination of tamped volume and apparent density after tamping*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Principle

For the determination of the DOA absorption number, a defined amount of pigment or extender shall be transferred to the mixer chamber of the absorptometer.

Under permanent kneading, DOA shall be added with a constant rate. The indication is the torque of the kneaders. While the torque is low at the beginning, it increases rapidly near the point of liquid absorption of the sample and decreases after reaching the maximum torque. The mixture changes from a free-flowing state to one of a pasty consistency.

On basis of the raw data torque curve and the settings, a polynomial shall be calculated. The value for 70 % of the maximum torque of this third order polynomial (smoothed curve) shall be used for the evaluation of the DOA absorption number.

4 Materials

4.1 Di-(2-ethylhexyl)adipate (DOA), which density is approximately 0,925 5 g/cm³ at 20 °C and which refractive index $n_D(20\text{ °C})$ is approximately 1,447.

4.2 Pigment or extender, as powder or micro-perls.

It can be added directly to the absorptometer chamber. In case of testing granulated materials, the determination is performed using a granular size fraction of between 1,0 mm and 3,15 mm, that is received by pre-sieving.