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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

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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.

The use of 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 CMR substances in different countries by the legal authorities.

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 have 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 cause of evaluated absorption numbers close to DBP measurement.

Rubber compounding ingredients — Silica — Oil absorption of precipitated silica

1 Scope

This International Standard specifies a general method for determining the liquid absorption capacity of a pigment and extender by using Dioctyladipate, Di-(2-ethylhexyl) adipate (DOA). 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, preferably 12,5 g, shall be transferred to the kneader chamber of the absorptometer.

Under permanent kneading at 125 rpm, DOA shall be added with a constant rate of 4 ml/min by means of a burette. 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.

After reaching the maximum torque – depending on the setting in the program – the burette and the kneaders shall stop automatically.

On basis of the raw data torque curve and the settings a polynom 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 based on original substance in ml/100 g.

The most important factors which affect the determination shall be pointed out.

a) Pore volume: the porosity of the material is the real cause for the absorption of liquid.

b) Moisture content: as the moisture content increases, the absorptive capacity decreases.

c) Particle size: at the same material family, but different degree of milling,the particle size can influence the DOA absorption. This is to take into account for comparison. In case of extremely fine milled samples an overload with DOA in connection with inhomogeneity of the mixture can occur that results in incorrect values.

d) Sample weight: with increasing sample weight the specific DOA absorption number decreases.

4 Materials

4.1 Dioctyladipate, Di-(2-ethylhexyl)adipate (DOA), which density is approximately 0,9255 g/cm^3 at 20 °C and which refractive index n(D, 20°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.

5 Equipment

5.1 Absorptometer with burette and system for measurement, storage and evaluation of torque data of the kneader.

NOTE The following pieces of equipment can be used:

— Absorptometer E, Fa. Brabender, Duisburg; equiped with extended functionality/evaluation unit), Brabender, Duisburg;

— Absorptometer C, Fa. Brabender, Duisburg;

— Hitec DBP-Absorptometer, Fa. Hitec, Luxembourg;

5.2 Beaker.

5.3 Sieves, one with mesh width of 1,0 mm, another one with mesh width of 3,15 mm.

5.4 Sieve pan.

5.5 Plastic or soft metal spatula and brush for cleaning the kneading chamber.

5.6 Precision balance, accuracy of 0,01 g.

5.7 Oven, capable of being maintained at 105 °C ± 2 °C.

6 Sampling

Take a representative sample of the material to be tested according to ISO 15528.

7 Procedure

7.1 Preliminary note

7.1.1 Carry out the determination in duplicate.

7.1.2 To avoid wrong measurement control the feeding pipe shall be free of air bubble before starting the measurement. If necessary the pipe shall be purged and the burette refilled.

7.1.3 The following settings should be used in the program.

7.1.3.1 Measurement settings

— Dosing rate (burette): 4,0 ml/min

— Rotation speed (kneader blades): 125 min^{-1}

— Temperature: 23 °C

The temperature of 23 °C in the settings should be used as a target value. Actually the temperature during the measurement should be in a range of 23 °C ± 5 °C.

7.1.3.2 Evaluation

a) Test end

— Threshold: 100 mNm

— End time: 40 s after max.

— Torque limit: 10 000 mNm

b) Polynom

— Start percent: 50 % of max torque

— End time: 20 s after max.

7.1.4 The evaluation of the aging status of the absorptometer shall be done according to Annex A.

7.2 Determination

7.2.1 Method A (powder sample, micro-perls)

7.2.1.1 The loss on drying of the sample material should not exceed 10 %. Material with a higher content of moisture should be dried to a lower value.

7.2.1.2 Weigh 12,50 g ± 0,02 g of the sample material by means of a precision balance (5.7) into a beaker (5.2), transfer to the kneader chamber and enter the sample weight into the program.

A sample weight of 12,5 g represents an optimum for most silica or silicates. It is advisable to use an integrated or separated funnel for filling the kneader chamber, during the operation of the absorptometer respectively that allows also in case of material with lower tamped density (< 150 g/l) to add the whole sample amount of 12.5 g at once.

In case of sample material with higher tamped density (> 300 g/l) it is recommended to use a higher sample amount (preferably 20,0 g) to achieve a sufficient filling of the kneader chamber and repeatable measurements.

The tamped density shall be measured according to ISO 787-11.

Ideally the sample amount should be designed to fill the kneader chamber sufficiently. It should neither be worked with to low sample amount (there is no sufficient increase of torque), nor the chamber should be overfilled, that does not ensure a sufficient mixing of the sample. Any deviation of the sample weight of 12,5 g shall be documented in the test report.

7.2.1.3 Close the safety device of the equipment, bring the feeding pipe into position and start the determination.

7.2.1.4 After the determination is ended, read the DOA absorption (based on original substance) as expressed as the 70 % value of the maximum torque from the measurement report.

During the determination, however in the range of the maximum DOA absorption, the observable silica-DOA-mixture forms a paste, that is indicated in the increase of the torque. After reaching the torque maximum, the torque decreases to lower values. The DOA absorption number based on original substance in ml/100g is the consumption of DOA related to the sample amount at 70 % of the maximum torque of the polynom. This polynom curve is calculated automatically at the end of the determination based on the settings (see 7.1.3) using the raw data of the measured torque curve.

7.2.1.5 Refill the burette, if there is no automatic refilling mode and clean the mixing chamber and kneader blades carefully. The DOA sample mixture is disposed conveniently, considering legal restrictions.

The DOA-sample mixture may cause difficulties during cleaning. In this case it is recommended to add some portion of a silica powder, switch on the kneader only for a short time and then dismount the mixing chamber for cleaning.

7.2.2 Method B (granulated samples)

7.2.2.1 Prepare a granular size fraction of between 1,0 mm and 3,15 mm by means of the appropriate sieves (5.3).

7.2.2.2 The further determination shall be carried out according to method A.

7.3 Evaluation

7.3.1 Evaluation for powder and micro-perl materials

The result of the determination can be given as DOA absorption **based on original substance** or optional as moisture corrected DOA absorption, as **based on dried substance**. In the test report the value that is reported shall be specified.

The calculation of the DOA absorption number based on dried substance can be calculated on the basis of the following formula and is given without decimal place.

$$DOA_{\text{orig}} = DOA_{\text{dry}} \times p \tag{1}$$

and

$$p = \frac{100}{100 - LOD} \tag{2}$$

where

DOA_{orig} is the DOA absorption based on original substance, in ml/100g;

DOA_{dry} is the DOA absorption number based on dry substance, in ml/100g;

p is the correction factor;

LOD is the loss on drying (2 h at 105 °C), in %.

The loss on drying (2 h at 105 °C) shall be determined separately according to ISO 787-2.

