INTERNATIONAL STANDARD

ISO 8312

Second edition
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Rubber compounding ingredients — Stearic acid — Definition and test methods

Ingrédients de mélange du caoutchouc — Acide stéarique — Définition et méthodes d'essai

Reference number
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Foreword

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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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

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*.

This second edition cancels and replaces the first edition (ISO 8312:1999), of which it constitutes a minor revision with the following changes:

— atomic absorption spectrometric method is stated as the preferred method in the Scope;

— Normative references in Clause 2 were updated and a Bibliography was added.

Rubber compounding ingredients — Stearic acid — Definition and test methods

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard defines stearic acid (including blends of stearic and palmitic acid) for use as a compounding ingredient in the rubber industry and specifies the test methods for describing its properties.

Classification of stearic acid and stearic acid/palmitic acid blends according to iodine value and typical chemical and physical properties for such materials for use in the rubber industry are given in Annex L. Annex L is given for information only.

In this International Standard, the atomic absorption spectrometric method is the preferred method.

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 660, *Animal and vegetable fats and oils — Determination of acid value and acidity*

ISO 662, *Animal and vegetable fats and oils — Determination of moisture and volatile matter content*

ISO 935, *Animal and vegetable fats and oils — Determination of titre*

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks*

ISO 3596, *Animal and vegetable fats and oils — Determination of unsaponifiable matter — Method using diethyl ether extraction*

ISO 3657, *Animal and vegetable fats and oils — Determination of saponification value*

ISO 3961, *Animal and vegetable fats and oils — Determination of iodine value*

ISO 4058, *Magnesium and its alloys — Determination of nickel — Photometric method using dimethylglyoxime*

ISO 5508, *Animal and vegetable fats and oils — Analysis by gas chromatography of methyl esters of fatty acids*

ISO 6685, *Chemical products for industrial use — General method for determination of iron content — 1,10-Phenanthroline spectrophotometric method*

ISO 7780:1998, *Rubbers and rubber latices — Determination of manganese content — Sodium periodate photometric methods*

ISO 8053, *Rubber and latex — Determination of copper content — Photometric method*

ISO 12966-2, *Animal and vegetable fats and oils — Gas chromatography of fatty acid methyl esters — Part 2: Preparation of methyl esters of fatty acids*

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
stearic acid (for use in the rubber industry)
mixture of straight-chain saturated fatty acids composed substantially of stearic acid in the form $C_{17}H_{35}COOH$ and palmitic acid in the form $C_{15}H_{31}COOH$

4 Sampling

Sampling shall be carried out in accordance with ISO 15528, using a stainless-steel sampling device.

5 Physical and chemical properties

The physical and chemical properties shall be determined by the methods of test listed in Table 1.

Table 1 — List of physical and chemical properties of stearic acid and the methods used for their determination

Property	Test method
Acid value, mg KOH/g	ISO 660
Saponification value, mg KOH/g	ISO 3657
Titre value, °C	ISO 935
Fatty acids, C_{16} to C_{18}, including unsaturates, percentage (mass fraction)	ISO 5508 and ISO 12966-2
Total matter volatile at 105 °C ± 3 °C, percentage (mass fraction)	ISO 662, oven method
Ash at 550 °C ± 25 °C, percentage (mass fraction)	Annex A
Iodine value, g/100 g	ISO 3961
Mineral acidity, $cm^3/100$ g	Annex F
Copper, mg/kg	Annex B[a] or G
Manganese, mg/kg	Annex C[a] or H
Iron, mg/kg	Annex D[a] or J
Unsaponifiable matter, percentage (mass fraction)	ISO 3596
Nickel, mg/kg	Annex E[a] or K
Note Where an atomic absorption spectrometer is not available, the molecular absorption spectrometric methods given in Annexes G, H, J and K may be used.	
[a] For speed and simplicity, the methods given in Annexes B, C, D and E are recommended.	

6 Test report

The test report shall include the following information:

a) all details necessary for complete identification of the product tested;

b) a reference to this International Standard, i.e. ISO 8312;

c) the results obtained:

— percentage ash w_A, from A.4;

— copper content w_{Cu}, from B.6 or from Annex G (state the method used);

— manganese content w_{Mn}, from C.6 or from Annex H (state the method used);

— iron content w_{Fe}, from D.6 or from Annex J (state the method used);

— nickel content, w_{Ni}, from E.6 or from Annex K (state the method used);

— mineral acidity, N_{ma}, from F.5;

— the results of other tests which may have been performed (see Table 1);

d) any unusual features noted during the determinations;

e) any operations not included in this International Standard, or in other International Standards cited, which might have affected the results;

f) the dates of the tests.

Annex A
(normative)

Determination of ash at 550 °C ± 25 °C

A.1 Principle

A weighed test portion is carefully volatilized without ignition, and the residue is ashed in a furnace at 550 °C ± 25 °C. The mass of ash is determined as a percentage of the mass of the original test portion.

A.2 Apparatus

Ordinary laboratory apparatus, plus the following:

A.2.1 Silica crucible.

A.2.2 Heat-resistant non-conducting (insulating) material in plate form, approximately 150 mm × 150 mm.

A.2.3 Analytical balance, accurate to 0,1 mg.

A.2.4 Muffle furnace, capable of being maintained at a temperature of 550 °C ± 25 °C.

A.3 Procedure

A.3.1 Heat the clean silica crucible (A.2.1) to 600 °C, allow to cool in a desiccator and weigh empty to 0,1 mg.

A.3.2 Place about 10 g of sample in this crucible and re-weigh to 0,1 mg. Place in a hole in the sheet of heat-resistant material (A.2.2).

A.3.3 Heat the crucible and contents gently in order to volatilize the test portion, taking care to ensure that the vapour does not ignite and that hot gases from the burner do not enter the crucible.

A.3.4 When all volatile material has been removed, place the crucible in the muffle furnace (A.2.4), maintained at 550 °C ± 25 °C, and ignite the contents for 30 min.

A.3.5 Place the crucible in a desiccator and allow to cool.

A.3.6 Re-weigh the crucible to the nearest 0,1 mg.

A.3.7 Repeat the operations specified in A.3.3, A.3.4 and A.3.5, until successive mass determinations differ by less than 2 mg.

A.3.8 Retain the ash obtained in A.3.6 if subsequent use can be made in another test.

A.4 Expression of results

Calculate the percentage ash, w_A, in accordance with Formula (A.1):

$$w_A = \frac{m_2 - m_1}{m_0} \times 100 \quad \text{(A.1)}$$

where

w_A is the percentage ash;

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the empty crucible;

m_2 is the mass, in grams, of the crucible and ash.

Annex B
(normative)

Determination of copper content — Atomic absorption spectrometric method

B.1 Principle

Ash made in accordance with Annex A is dissolved in hydrochloric acid and the solution made up to standard volume. The absorbance is measured at 324,7 nm in an atomic absorption spectrometer. The copper content is determined by reference to a calibration graph prepared by measuring the absorbance of standard copper solutions.

B.2 Reagents

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

B.2.1 Hydrochloric acid, a mass fraction of 10 % solution.

B.2.2 Copper, standard solution corresponding to 10 mg of Cu per dm^3.

B.3 Apparatus

Ordinary laboratory apparatus, plus the following:

B.3.1 Atomic absorption spectrometer, fitted with a copper hollow-cathode lamp.

B.3.2 One-mark volumetric flasks, two of capacity 10 cm^3 and six of capacity 50 cm^3, complying with the requirements of ISO 1042:1998, class A.

B.4 Procedure

B.4.1 Obtain a sample of ash by conducting the determination specified in Annex A.

B.4.2 Dissolve the ash so obtained in 5 cm^3 of dilute hydrochloric acid (B.2.1). Transfer the solution quantitatively to a 10 cm^3 one-mark volumetric flask (see B.3.2).

B.4.3 Dilute the digested ash to exactly 10 cm^3 in the one-mark volumetric flask by adding water.

B.4.4 Set the wavelength of the spectrometer (B.3.1) to 324,7 nm and aspirate the test solution into the flame, followed immediately by water, and then blank solution made up from the same reagents and using the same procedure but omitting the test portion.

B.4.5 Repeat this procedure and record the mean values of absorbance of the test solution and the blank test solution.

B.5 Preparation of the calibration graph

B.5.1 Preparation of solutions

Into a series of six 50 cm^3 one-mark volumetric flasks (see B.3.2), transfer the volumes of the standard copper solution (B.2.2) indicated in Table B.1, dilute to the mark with water and mix.

Table B.1 — Standard calibration solutions for determination of copper

Volume of standard copper solution (B.2.2) cm^3	Copper content $\mu g/cm^3$
0, 5	0,1
2,5	0,5
5,0	1,0
10,0	2,0
15,0	3,0
25,0	5,0

B.5.2 Spectrometric measurements

Aspirate each of the standard calibration solutions in turn into the flame of the atomic absorption spectrometer (B.3.1) and record their absorbances at a wavelength of 324,7 nm, following the instructions of the instrument manufacturer.

Aspirate water into the flame after each measurement.

B.5.3 Plotting the calibration graph

Plot a graph having the masses, in micrograms, of copper per cm^3 of the calibration solutions as abscissae and the corresponding values of absorbance as ordinates.

B.6 Expression of results

By reference to the calibration graph prepared as described in B.5.3, determine the copper content corresponding to the absorbances of the test solution and the blank test solution.

The concentration of copper to be determined shall fall within the linear part of the calibration curve.

The total copper content of the sample, w_{Cu}, expressed in milligrams per kilogram, is given in Formula (B.1):

$$w_{Cu} = \frac{10\left(m_3 - m_4\right)}{m_0} \tag{B.1}$$

where

m_0 is the mass, in grams, of the test portion;

m_3 is the copper content, in micrograms per cm^3, of the test solution;

m_4 is the copper content, in micrograms per cm^3, of the blank test solution.

Express the result to the nearest 0,1 mg/kg.