BS ISO 19242:2015

BSI Standards Publication

Rubber — Determination of total sulfur content by ion chromatography

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National foreword

This British Standard is the UK implementation of ISO 19242:2015.

The UK participation in its preparation was entrusted to Technical Committee PRI/23, Test methods for rubber and non-black compounding ingredients.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Rubber — Determination of total sulfur content by ion chromatography

Caoutchouc — Détermination de la teneur en soufre total par chromatographie ionique

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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 2, *Testing and analysis*.

Introduction

Titration methods using thorin, a toxic arsenic compound as a reagent, have been used to determine the total sulfur content in raw rubber and rubber compounds. In this International Standard the sulfur in a test piece is burnt and oxidized to sulfur dioxide, which is then absorbed into a hydrogen peroxide solution and converted to sulfuric acid for determination by ion chromatography.

No hazardous reagents are required, thus improving the working environment as well as being eco-friendly. In addition, ion chromatography is used worldwide for its simplicity and high accuracy.

Rubber — Determination of total sulfur content by ion chromatography

WARNING 1 — 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.

WARNING 2 — The use of this International Standard pre-supposes sufficient working knowledge of the principles and techniques of ion chromatography for the analyst to perform the operations described and interpret the results correctly.

CAUTION — Certain procedures specified in this International Standard may involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This International Standard specifies a method for the determination of the total sulfur content of raw rubber and unvulcanized or vulcanized rubber compounds by ion chromatography following the preparation of a sample solution using either a tubular furnace combustion or an oxygen combustion flask method.

The tubular furnace combustion method is applicable for rubbers with sulfur content less than 0,1 % as it is possible that the oxygen combustion flask method will not give sufficiently accurate results.

The oxygen combustion flask method is not applicable to rubbers containing a metal salt that forms an insoluble metal sulfate, such as barium sulfate.

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 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 1795, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*

ISO 4661-2, *Rubber, vulcanized — Preparation of samples and test pieces — Part 2: Chemical tests*

3 Principle

3.1 Combustion

3.1.1 Tubular furnace combustion method

A test piece is burnt in an oxygen stream in a tubular furnace with an electrical heater. The sulfur in the test piece is oxidized to sulfur dioxide, which is absorbed into hydrogen peroxide solution and converted to sulfuric acid.

3.1.2 Oxygen combustion flask method

A test piece is burnt in the presence of oxygen in a flask. The sulfur in the test piece is oxidized to sulfur dioxide, which is absorbed into hydrogen peroxide solution and converted to sulfuric acid.

3.2 Determination

The sulfate from either the tubular furnace combustion or the oxygen combustion flask method is quantitatively analysed by ion chromatography, and the result is converted to the total sulfur content of the original rubber sample.

4 Reagents

Use only reagents of recognized analytical grade and only water as specified in 4.1. The reagents for the tubular furnace combustion method are given in A.2. The reagents for the oxygen combustion flask method are given in B.2.

4.1 Water, of grade 1 or higher as specified in ISO 3696.

4.2 Sulfate stock standard solution, of a commercial standard solution with a certified sulfate concentration, e.g. 1 000 mg/dm^3 of sulfate, traceable to national standards.

Observe the manufacturer's expiry date or recommended shelf-life.

4.3 Sulfate calibration solutions, prepared by dilution of the sulfate stock standard solution (4.2) with water (4.1).

Prepare at least four solutions of different concentration of sulfate covering the expected concentration from the sample. The solutions shall be prepared every analytical day.

4.4 Eluent solution, capable of eluting sulfate to a proper retention time in selected column.

Follow the column manufacturer's instructions. Some examples of eluent are shown in Annex C.

5 Apparatus

5.1 Balance, of accuracy to the nearest 0,1 mg.

5.2 Apparatus for the tubular furnace combustion method, as specified in A.3.

5.3 Apparatus for the oxygen combustion flask method, as specified in B.3.

5.4 Ion chromatograph, consisting of the pump to supply eluent, injection valve, column and conductivity detector specified as follows:

- Pump: capable of delivering a constant flow within the range of 0,1 cm^3/min to 2,0 cm^3/min;
- Injection valve: capable of injecting a constant volume of solution;
- Column: filled with anion exchange resin suitable for resolving sulfates from other inorganic anions. Some examples are shown in Annex C;
- Suppressor: used for reducing the conductivity of high ionic eluent;
- Detector: for detecting conductivity.

An ion chromatograph fitted with a suppressor is suitable for sensitive analysis (see Figure 1). The suppressor is attached between the column and the detector. However, an ion chromatograph without a suppressor may also be selected (see Figure 2). For the conditions of these methods, see Annex C.

Figure 1 — Example of basic ion chromatography system with suppressor

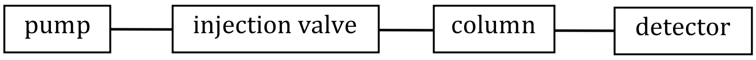


Figure 2 — Example of basic ion chromatography system without suppressor

6 Procedure

6.1 Preparation of sample solutions

6.1.1 Tubular furnace combustion method

Follow the method specified in Annex A. Repeat the procedure to create two solutions for each sample.

6.1.2 Oxygen combustion flask method

Follow the method specified in Annex B. Repeat the procedure to create two solutions for each sample.

6.2 Determination of sulfate by ion chromatography

6.2.1 Setting up the ion chromatograph

Set up the ion chromatograph in accordance with the manufacturer's instructions.

6.2.2 Measuring calibration solutions

Inject the calibration solutions in order of increasing concentration and measure the conductivity of sulfate peak area in each solution. Determine the correlation formula (calibration curve) by plotting the peak area as a function of concentration by means of linear regression.

6.2.3 Measuring blank solutions and sample solutions

Inject the blank solutions and sample solutions (created in 6.1) to measure the peak area. Then determine the sulfate concentration of the blank C_B and that of the individual sample solution C_S by using the calibration curve determined in 6.2.2.

7 Calculations

Calculate the sulfur content of the sample in percentage from each value determined in 6.2.3. Average the two content values and round to the second decimal place, using the following formula:

$$\frac{(C_S - C_B)\times V}{W\times 1\,000}\times\frac{32{,}06}{96{,}06}\times 100$$

where

W is the mass of the test piece, in mg;

C_S is the sulfate concentration of the sample solution, in mg/dm^3;

C_B is the sulfate concentration of the blank solution, in mg/dm^3;

V is the volume of the volumetric flask, in cm^3;

32,06 is the atomic weight of sulfur;

96,06 is the molecular mass of sulfate.

8 Precision

See Annex D.

9 Test report

The test report shall include the following particulars:

a) sample details:
   1) a full description of sample;
   2) if appropriate, the brief preparation procedure of the test piece from the sample;
b) test method:
   1) a reference to this International Standard (i.e. ISO 19242);
   2) the combustion method used: “tubular furnace combustion method” or “oxygen combustion flask method”;
   3) the method of ion chromatography used: suppressor or non-suppressor method;
   4) the column and eluent used;
c) details of any procedures not specified in this International Standard;
d) the test results;
e) the date(s) of the test.

Annex A
(normative)

Preparation of sample solutions by the tubular furnace combustion method

A.1 General

This annex specifies the preparation procedure of sample solutions by the tubular furnace combustion method.

A.2 Reagents and materials

A.2.1 Hydrogen peroxide solution of 500 µg/g, prepared by diluting hydrogen peroxide of 30 % mass fraction solution with water to 500 µg/g.

A.2.2 Oxygen, of mass fraction > 99,7 %.

A.2.3 Argon, of mass fraction > 99,98 %.

A.2.4 Air, of certified quality in contamination and purity.

NOTE Air is an alternative to the combustion gas of oxygen mixed with argon. The certification of the air quality can be judged by individual laboratories in so far as it does not affect the test result.

A.3 Apparatus

The apparatus consists of combustion furnace, quartz tube, and absorbing vessel as shown in Figure A.1.

A.3.1 Combustion furnace, with an electric heater capable of heating at a temperature of 1 000 °C and maintaining the temperature for a certain period of time.

A.3.2 Quartz tube, having heat-resistance enough to be heated at the maximum temperature (e.g. 1 000 °C) by the electric heater and capable of holding a sample boat inside.

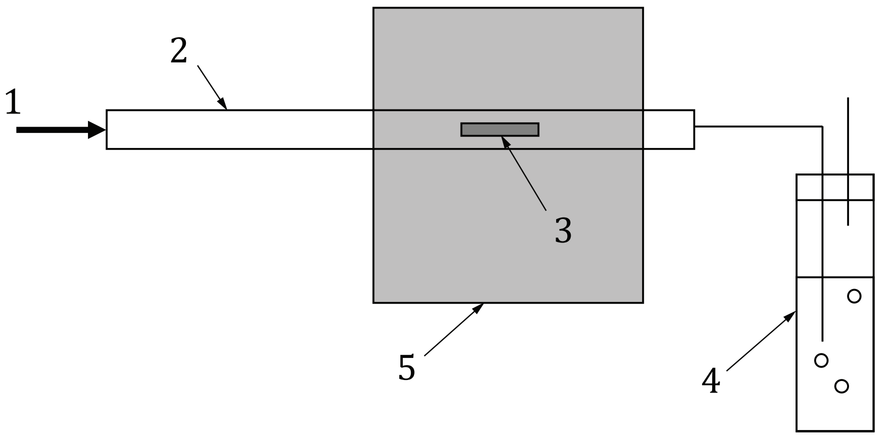
A gas consisting of oxygen mixed with argon, or air which is supplied by the system, passes through the tube.

A.3.3 Sample boat, made of quartz, ceramic, or platinum and capable of carrying a test piece.

It shall have heat-resistance enough to be heated by the electric heater at the maximum temperature (e.g. 1 000 °C).

A.3.4 Absorption vessel, made of borosilicate glass with sufficient volume to allow the gas to bubble through the absorbent solution in it when it is filled at a certain level.

Other types of apparatus than those specified in A.3.1 through A.3.4 may be used when they are proven to give the same result.



Key

1	combustion gas
2	quartz tube
3	sample boat
4	absorption vessel
5	combustion furnace

Figure A.1 — An example of the tubular combustion furnace

A.4 Sample preparation

In the case of raw rubber and rubber compounds, carry out sampling in accordance with ISO 1795 or ISO 4661-2, respectively.

A.5 Procedure

A.5.1 Ignite the sample boat at the combustion maximum temperature and weigh it to the nearest 0,1 mg after cooling down.

A.5.2 Weigh out a test piece of about 10 mg taken from the test sample prepared in A.4 and place it in the sample boat. Weigh the boat with the test piece to the nearest 0,1 mg and determine the mass of the test piece W by subtracting the mass measured in A.5.1.

A.5.3 Follow procedure a) through c) below, unless the manufacturer‘s instructions are available:

a) Insert the sample boat into the combustion furnace.

b) Put the absorbent solution in the absorption vessel and connect it to the combustion furnace.

c) Start the flow of the argon and oxygen gas, or air, and turn on the combustion furnace to burn the test piece completely. An example of suitable conditions for the combustion is as follows:

- temperature: 1 000 °C;
- flow rate of argon gas: 200 cm^3/min;
- flow rate of oxygen gas: 400 cm^3/min;
- amount of absorbent solution [H_2O_2, 500 µg/g (A.2.1)]: 10 cm^3.

A.5.4 After the completion of burning, wash through the whole tube and collect the washes into the absorption vessel together with the absorbent solution to use as a sample solution. Adjust the amount of the sample solution to 25 cm^3 (V) for ion chromatography analysis.

When it is not possible to adjust the solution amount to 25 cm^3 due to the system specification, add a proper internal standard to the absorbent beforehand and write a calibration curve using the concentration ratio of the sulfate to the internal standard.

A.5.5 Prepare a blank solution by repeating the procedures from A.5.1 through A.5.4 without placing a test piece in the sample boat. Only one blank determination is necessary.

Annex B
(normative)

Preparation of sample solutions by the oxygen combustion flask method

B.1 General

This annex specifies the preparation procedure of sample solutions by the oxygen combustion flask method.

B.2 Reagents and materials

B.2.1 Hydrogen peroxide solution 0,3 % (m/m), prepared by diluting hydrogen peroxide of 30 % mass fraction solution with water to 0,3 %.

B.2.2 Oxygen, of mass fraction > 99,7 %.

B.2.3 Ashless filter paper, for wrapping a test piece (see Figure B.1 for examples).

B.2.4 Membranous filter, of pore size 0,45 µm.

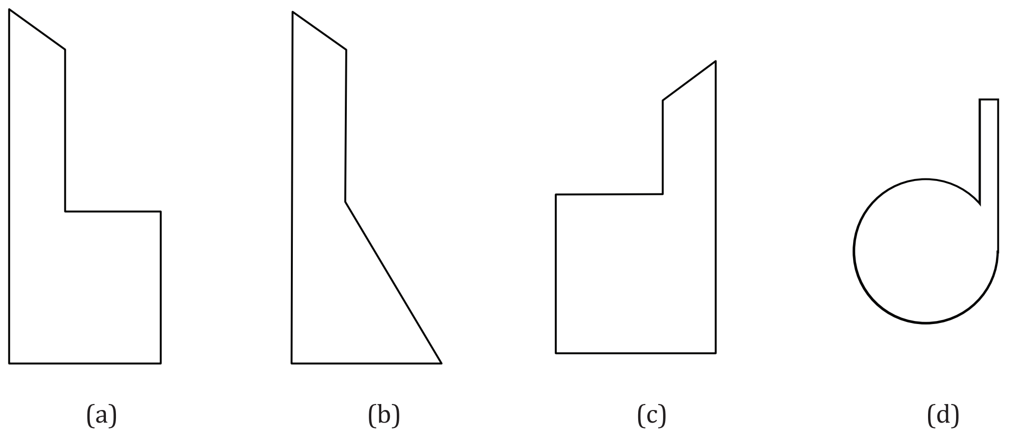


Figure B.1 — Examples of filter paper form for use with platinum sample baskets

B.3 Apparatus

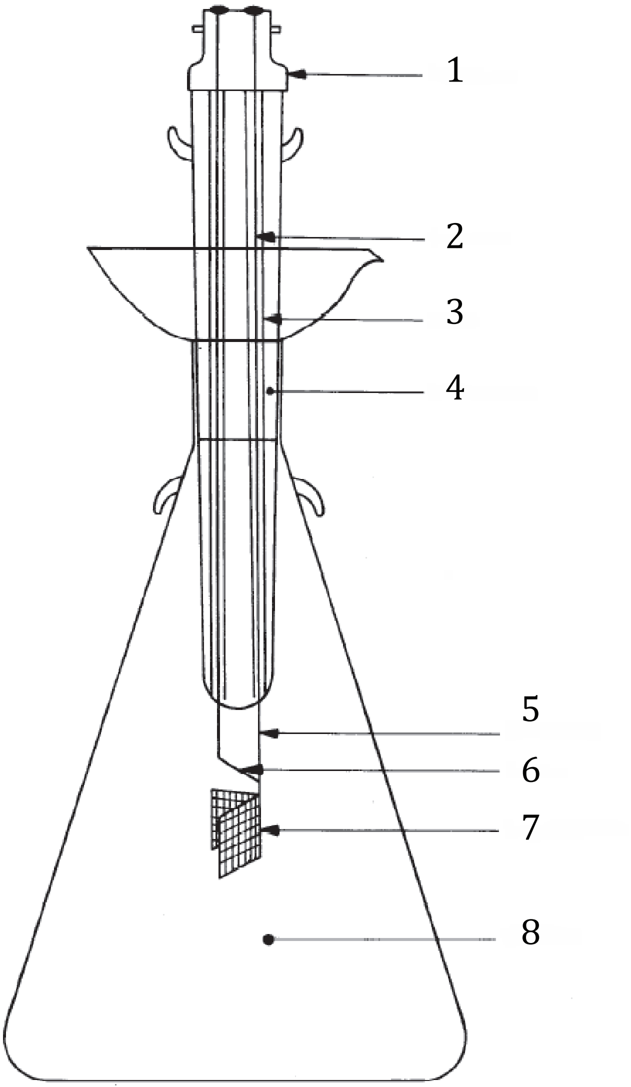
B.3.1 Oxygen combustion flask, thick-walled, of capacity 300 cm^3 to 1 dm^3, with a platinum sample basket as shown in Figure B.2.

The flask and the glass tube for platinum ignition wire shall be inspected for cracks before using in order to avoid a risk of explosion due to the rapid combustion in the flask.

A flask of a different shape may be used when it is proven to give the same result.

B.3.2 Combustion device, of infrared or electrical ignition.

B.3.3 Magnetic stirrer.



Key

1	bayonet cap
2	copper wire
3	sheath
4	ISO 24/29 joint
5	platinum wire
6	fuse bar
7	platinum sample basket
8	1 dm^3 flask

Figure B.2 — An example of a combustion flask

B.4 Sample preparation

In the case of raw rubber and rubber compounds, carry out sampling in accordance with ISO 1795 or ISO 4661-2, respectively.

B.5 Procedure

B.5.1 Weigh out a test piece of about 10 mg taken from the test sample prepared in B.4 to the nearest 0,1 mg (W).

B.5.2 Wrap the test piece with a filter paper (B.2.3) and fold it.

B.5.3 Place the filter paper with the test piece in a platinum sample basket.

B.5.4 Put 10 cm^3 of hydrogen peroxide solution (B.2.1) in a flask and place the stirring bar at the bottom of the flask.

B.5.5 Insert a rubber tube into the flask and purge it with oxygen for 10 s to 60 s. Remove the rubber tube. Place the platinum sample basket (B.5.3) with the filter paper inside the flask as quickly as possible. If a flask equipped with an oxygen inlet tube is used, place the basket before purging.

B.5.6 When using an electrical ignition apparatus, attach the flask to the proper connections to electrify the platinum sample basket. When using an infrared ignition apparatus, place the flask with the paper extension in line with the focal point to ignite it. Complete the combustion.

B.5.7 Leave the flask at the ambient temperature for 30 min or cool down in ice water for 1 min to 3 min after the combustion is complete.

B.5.8 Add 10 cm^3 of water into the flask. Stand the flask for 30 min to allow the gases to be absorbed by the absorbent solution. Use a stirrer if necessary.

B.5.9 Move the solution in the flask to a volumetric flask of 50 cm^3 (V) and adjust the amount to a constant volume for the sample solution for ion chromatography analysis.

B.5.10 If necessary filter out particulate matter in the sample solution (B.5.8) with a membranous filter (B.2.4), before injecting it into the ion chromatograph. To avoid possible contamination from the membranous filter, wash the membranous filter with a small quantity of the sample solution and discard the washes before filtering.

B.5.11 Prepare a blank solution by repeating the procedures from B.5.1 through B.5.10 without wrapping a test piece with the filter paper. Only one blank determination is necessary.

Annex C
(informative)

Examples of conditions for ion chromatography

Examples of conditions for ion chromatography are given in Table C.1 and C.2. Other conditions may be applied when they are proven to give the same results.

Table C.1 — Example of conditions for the suppressor method of ion chromatography

Parameter	**Dionex IonPac AS 22-Fast**[a] **(Length: 150 mm, internal diameter: 4,0 mm)**	**TSKgel SuperIC-Anion HS**[b] **(Length: 100 mm, internal diameter: 4,6 mm)**	**Shim-pack IC-SA2**[c] **(Length: 250 mm, internal diameter: 4,0 mm)**
Eluent	The following agents are dissolved in 1 dm^3 of water: 4,5 mmol Sodium carbonate and 1,4 mmol Sodium hydrogen carbonate	The following agents are dissolved in 1 dm^3 of water: 3,0 mmol Sodium carbonate and 3,8 mmol Sodium hydrogen carbonate	The following agents are dissolved in 1 dm^3 of water: 1,8 mmol Sodium carbonate and 1,7 mmol Sodium hydrogen carbonate
Flow rate	1,2 cm^3/min	1,5 cm^3/min	1,0 cm^3/min
Column temperature	30 °C	40 °C	40 °C
Injection volume	10 mm^3	30 mm^3	50 mm^3

NOTE The products listed in the footnotes to this table are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

[a] Thermo Fisher Scientific Co. LTD.; 3-9 Moriya-cho Kanagawa-ku, Yokohama 221-0022, Japan, Fax +81-45-453-9110, Tel +81-45-453-9100, www.thermoscientific.com

[b] Tosoh Corporation; 3-8-2 Shiba Minato-ku, Tokyo 105-8623, Japan, Fax +81-3-5427-5220, Tel +81-3-5427-5180, www.tosoh.com

[c] Shimadzu Corporation; 1 , Nishinokyo Kuwabara-cho, Nakakyo-ku, Kyoto 604-8511, Japan, www.shimadzu.com

Table C.2 — Example of conditions for the non-suppressor method of ion chromatography

Parameter	**Shim-pack IC-A3**[a] **(Length: 150 mm,** **internal diameter: 4,6 mm)**	**TSKgel IC-Anion-PW$_{XL}$ PEEK**[b] **(Length: 75 mm,** **internal diameter: 4,6 mm)**
Eluent	The following agents are dissolved in 1 dm^3 of water 8,0 mmol p-Hydroxybenzoic acid, 3,2 mmol Bis-Tris, and 50,0 mmol Boric acid	The following agents are dissolved in 1 dm^3 of water 1,3 mmol Potassium gluconate, 1,3 mmol Borax,30 mmol Boric acid, 10 % Acetonitrile, and 0,5 % Glycerine
Flow rate	1,0 cm^3/min	1,0 cm^3/min
Column temperature	40 °C	40 °C
Injection volume	50 mm^3	50 mm^3

NOTE The products listed in the footnotes to this table are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

[a] Shimadzu Corporation; 1 , Nishinokyo Kuwabara-cho, Nakakyo-ku, Kyoto 604-8511, Japan, www.shimadzu.com

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Annex D
(informative)

Precision data from an interlaboratory test programme

D.1 General

The following interlaboratory test programme (ITP) was initially carried out in 2014.

All calculations to provide repeatability and reproducibility values were performed in accordance with ISO/TR 9272. Precision concepts and nomenclature are also given in ISO/TR 9272.

D.2 Precision data from the ITP

D.2.1 Programme details

Cured test pieces were prepared in one laboratory and sent to six laboratories for the tubular furnace combustion method and to seven laboratories for the oxygen combustion flask method.

A total of two compounds were used in the test. The samples were designated as Compounds ISO-1 and ISO-2.

The test samples were provided as sheeted pieces with a thickness of 2 mm.

The number of laboratories on which precision data for each property is based is given in Tables D.1 and D.2. The number of participating laboratories as noted in Tables D.1 and D.2 is the final number after identifying certain laboratory values as outliers.

The ITP testing was conducted over a period of two sequential weeks. On a day in a specified week, a blank test and determination tests for each type of rubber ($n = 2 \times 2$) were performed within a day. One week after day 1, the same set of the blank test and the determination tests was repeated ($n = 2 \times 2$). All analysis was conducted on the basis of these test results.

D.2.2 Precision data

The precision data are listed in Table D.1 for the tubular furnace combustion method and in Table D.2 for the oxygen combustion flask method.

Repeatability: The repeatability, *r*, of the test method has been established as the appropriate value tabulated in Tables D.1 and D.2. Two single test results that differ by more than the value shall be considered suspect and some appropriate investigative action should be taken.

Reproducibility: The reproducibility, *R*, of the test method has been established as the appropriate value tabulated in Tables D.1 and D.2. Two single test results that differ by more than the value shall be considered suspect and some appropriate investigative action should be taken.

The precision results as determined by this ITP should not be applied to acceptance or rejection testing for any group of materials or products without documentation that the results of this precision evaluation actually apply to the products or materials tested.

Table D.1 — Precision data for the tubular furnace combustion method

Compound	**Mean level**	S_r	r	(r)	S_R	R	(R)	**No. of laboratories** [a]
ISO-1	1,23	0,018	0,050	4,09	0,049	0,14	11,24	6
ISO-2	0,90	0,017	0,048	5,38	0,036	0,10	11,26	6
Pool value [b]		0,018	0,049	4,78	0,043	0,12	11,25	

NOTE

s_r = within-laboratory standard deviation (in measurement units);

r = repeatability (in measurement units);

(r) = repeatability (in percent of mean level);

s_R = between-laboratory standard deviation (for total between-laboratory variation in measurement units);

R = reproducibility (in measurement units);

(R) = reproducibility (in percent of mean level).

[a] Number of laboratories after outliers deleted (total number of laboratories in ITP: 6).

[b] Root mean squares are listed for pooled values.

Table D.2 — Precision data for the oxygen combustion flask method

Compound	**Mean level**	S_r	r	(r)	S_R	R	(R)	**No. of laboratories** [a]
ISO-1	1,25	0,041	0,117	9,36	0,063	0,18	14,20	7
ISO-2	0,90	0,035	0,100	11,10	0,048	0,14	15,09	7
Pool value [b]		0,038	0,109	10,26	0,056	0,16	14,65	

NOTE See Table D.1 for an explanation of symbols.

[a] Number of laboratories after outliers deleted (total number of laboratories in ITP: 7).

[b] Root mean squares are listed for pooled values.

Bibliography

[1] ISO/TR 9272, *Rubber and rubber products — Determination of precision for test method standards*

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