INTERNATIONAL STANDARD

ISO 8054

Second edition
1996-02-01

Rubber, compounded or vulcanized — Determination of sulfide sulfur content — Iodometric method

Caoutchouc vulcanisé ou non vulcanisé — Dosage du soufre sous forme de sulfure — Méthode iodométrique

Reference number
ISO 8054:1996(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 8054 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This second edition cancels and replaces the first edition (ISO 8054:1988), which has been technically revised.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Introduction

In unvulcanized compounded rubbers, any sulfide sulfur present is normally due to metallic sulfides contained in fillers (e.g. lithopone).

In vulcanizates, the sulfide sulfur content increases with the state of cure and can therefore be used as a method of determining the state of cure of products for which this is critical, providing no metallic sulfide fillers are present in large amounts.

This page intentionally left blank

Rubber, compounded or vulcanized — Determination of sulfide sulfur content — Iodometric method

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This 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 specifies a titrimetric method for the determination of sulfide sulfur in unvulcanized compounded rubbers and in vulcanizates.

It is applicable to chlorinated rubbers, nitrile rubbers and hydrocarbon rubbers (including natural rubber).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 383:1976, *Laboratory glassware — Interchangeable conical ground joints.*

ISO 1407:1992, *Rubber — Determination of solvent extract.*

ISO 4793:1980, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation.*

ISO 4799:1978, *Laboratory glassware — Condensers.*

3 Principle

The rubber is extracted with acetone and the extracted rubber is treated with a mixture of hydrochloric and acetic acids to decompose metallic sulfides. The resulting hydrogen sulfide is absorbed in buffered cadmium acetate solution and the cadmium sulfide thus formed is determined iodometrically.

4 Reagents and materials

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

4.1 Acetone.

4.2 Hydrochloric acid/acetic acid, mixture.

Mix together 5 volumes of glacial acetic acid (ρ = 1,05 g/cm^3), 1 volume of concentrated hydrochloric acid [36 % (m/m), ρ = 1,18 g/cm^3] and 1 volume of water.

4.3 Cadmium acetate, buffered solution.

Dissolve 25 g of cadmium acetate dihydrate [$(CH_3COO)_2Cd\cdot 2H_2O$] and 25 g of sodium acetate trihydrate ($CH_3COONa\cdot 3H_2O$) in water, add 100 cm^3 of glacial acetic acid (ρ = 1,05 g/cm^3) and make up to 1 dm^3 with water.

4.4 **Surfactant**, solution.

Make up an approximately 2 % (*m*/*m*) aqueous solution of a surfactant which will not react with iodine. Filter the solution through a No. 1 Whatman[1], or equivalent, filter paper before use.

NOTE 1 Gum ghatti and nonyl phenol polyethoxalate are examples of suitable surfactants.

4.5 **Iodine**, solution, $c(I_2) \approx 0{,}025$ mol/dm^3.

Dissolve 6,35 g of iodine and 40 g of potassium iodide in water, and make up to 1 dm^3.

4.6 **Sodium thiosulfate**, standard volumetric solution, $c(Na_2S_2O_3 \cdot 5H_2O) = 0{,}05$ mol/dm^3.

4.7 **Starch**, solution, 1 g in 100 cm^3 of water.

4.8 **Supply of pure nitrogen gas.**

4.9 **Glycerol.**

5 Apparatus

Ordinary laboratory apparatus, and

5.1 **All-glass extraction apparatus** (see figure 1), as specified in ISO 1407, comprising the elements described in 5.1.1 to 5.1.3.

5.1.1 **Extraction flask**, capacity 150 cm^3, with a 34/35 socket conforming with the requirements of ISO 383 (see also 5.2.1).

5.1.2 **Jacketed Soxhlet extractor**, preferably with a 34/35 socket and 34/35 cone.

5.1.3 **Graham condenser**, coolant-tube length 400 mm, with a 34/35 angle drip cone conforming with the requirements of ISO 4799.

Substitutions may be made if they perform the same function as the item listed.

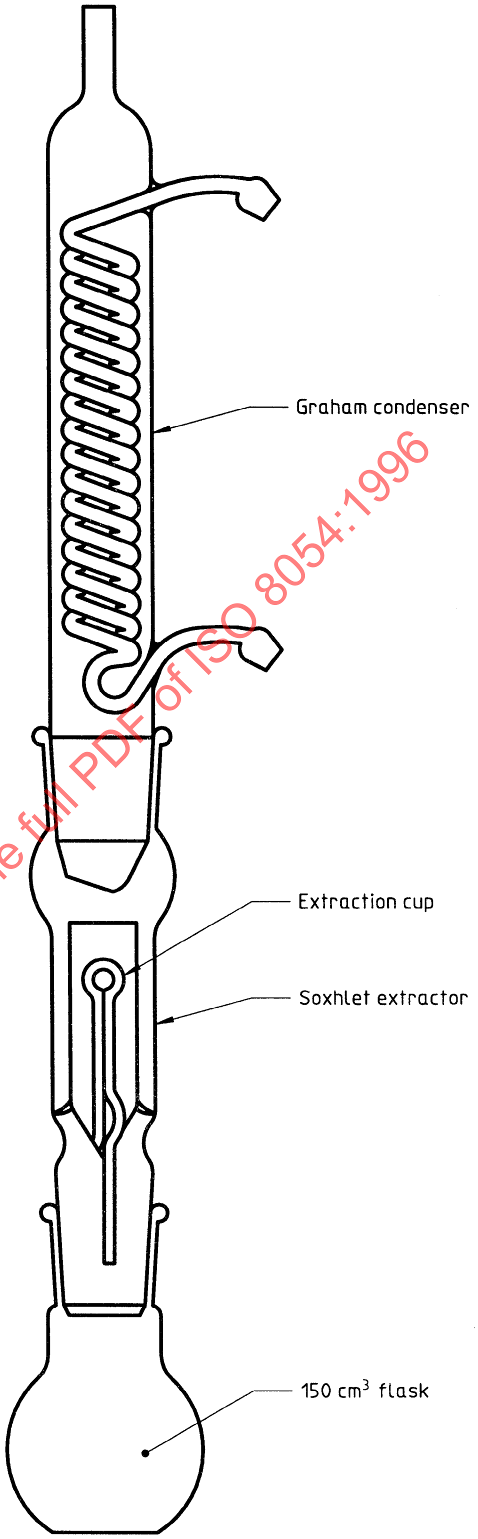

Figure 1 — Extraction apparatus

---

[1] No. 1 Whatman is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

5.2 All-glass apparatus for the evolution and absorption of hydrogen sulfide (see figure 2), comprising the elements described in 5.2.1 to 5.2.8.

5.2.1 Extraction flask (A), capacity 150 cm^3, with a 34/35 socket conforming with the requirements of ISO 383 (see also 5.1.1).

5.2.2 Adapter (D), with 14/23 socket and 34/35 cone, and with a gas inlet tube to reach to within 3 mm of the bottom of the extraction flask (5.2.1) when assembled.

5.2.3 Conical flask (J), of capacity 250 cm^3, with a 24/29 socket.

5.2.4 Gas-washing bottles (K and L).

5.2.5 Receiver adapter (E), with 10/19 socket and 24/29 cone, and with an internal sealed dip-tube.

5.2.6 Condenser, Liebig type (F), effective coolant-tube length approximately 250 mm, with 14/23 angle drip cone, 14/23 socket and side arm with 10/19 cone.

5.2.7 Dropping funnel (G), capacity 100 cm^3, with 14/23 cone, modified stem and 14/23 stopper.

5.2.8 Micro-immersion filter (Emich pattern) (H), stem approximately 75 mm long, diameter of disc 10 mm, porosity grade P 40 (see ISO 4793).

6 Sampling, sample preparation and selection of test portion

6.1 Cut from several areas of the sample a test portion of between 0,5 g and 2,0 g, depending on the expected sulfide sulfur level.

6.2 Pass the test portion six times between the cold rolls of a laboratory mill set to a nip not exceeding 0,5 mm. If it is not possible to pass the test portion through the mill, the test portion may be cut into pieces less than 1 mm per side. Weigh the test portion to the nearest 1 mg.

6.3 Extract the test portion with acetone (4.1) in the extraction apparatus (5.1) as described in ISO 1407.

6.4 Transfer the extracted test portion from the Soxhlet extractor (5.1.2) to a clock glass and evaporate the acetone by heating in an oven for 15 min at 70 °C. Transfer the dried sample to the 150 cm^3 flask (A).

7 Procedure

7.1 Determination

7.1.1 Assemble the flask (A) into the apparatus of figure 2, ensuring that all ground glass joints are lubricated with glycerol (4.9). Introduce 100 cm^3 of cadmium acetate solution (4.3) and 1 cm^3 of surfactant solution (4.4) into the conical flask (J) and half fill the gas-washing bottles (K and L) with the cadmium acetate solution. Pass a stream of nitrogen (4.8) through the apparatus to displace the air and then adjust the flow to about one bubble per second in the conical flask (J).

7.1.2 Introduce 50 cm^3 of the hydrochloric acid/acetic acid mixture (4.2) slowly from the dropping funnel (G) into the flask (A), heat slowly to boiling and continue to boil gently for 30 min to 40 min. Towards the end of this period, increase the flow of nitrogen to sweep over all traces of hydrogen sulfide. The solution in bottle K should be clear and colourless; if this is not so, it is an indication that the nitrogen flow has been too rapid and the determination shall be repeated using a smaller test portion or slower rate of nitrogen flow. Disconnect the receiver adapter (E) from the delivery tube and, holding it at an angle, add by means of a pipette a measured volume V_0 of the iodine solution (4.5) (usually 25 cm^3 is a suitable quantity) to flask J, so that excess iodine is present.

7.1.3 Allow the iodine to react with any precipitate adhering to the entry tube and, when all the precipitate has dissolved, remove and rinse the tube, cool the solution to 15 °C, and titrate the excess iodine with the sodium thiosulfate solution (4.6), using starch solution (4.7) as indicator. Record the volume V_1 of sodium thiosulfate solution used.

7.2 Blank titration

Carry out, in parallel with the determination described in 7.1, a blank titration by titrating V_0 cm^3 of the iodine solution with the sodium thiosulfate solution in the presence of 100 cm^3 of the cadmium acetate solution (4.3), using starch solution as indicator. Record the volume V_2 of sodium thiosulfate solution used.

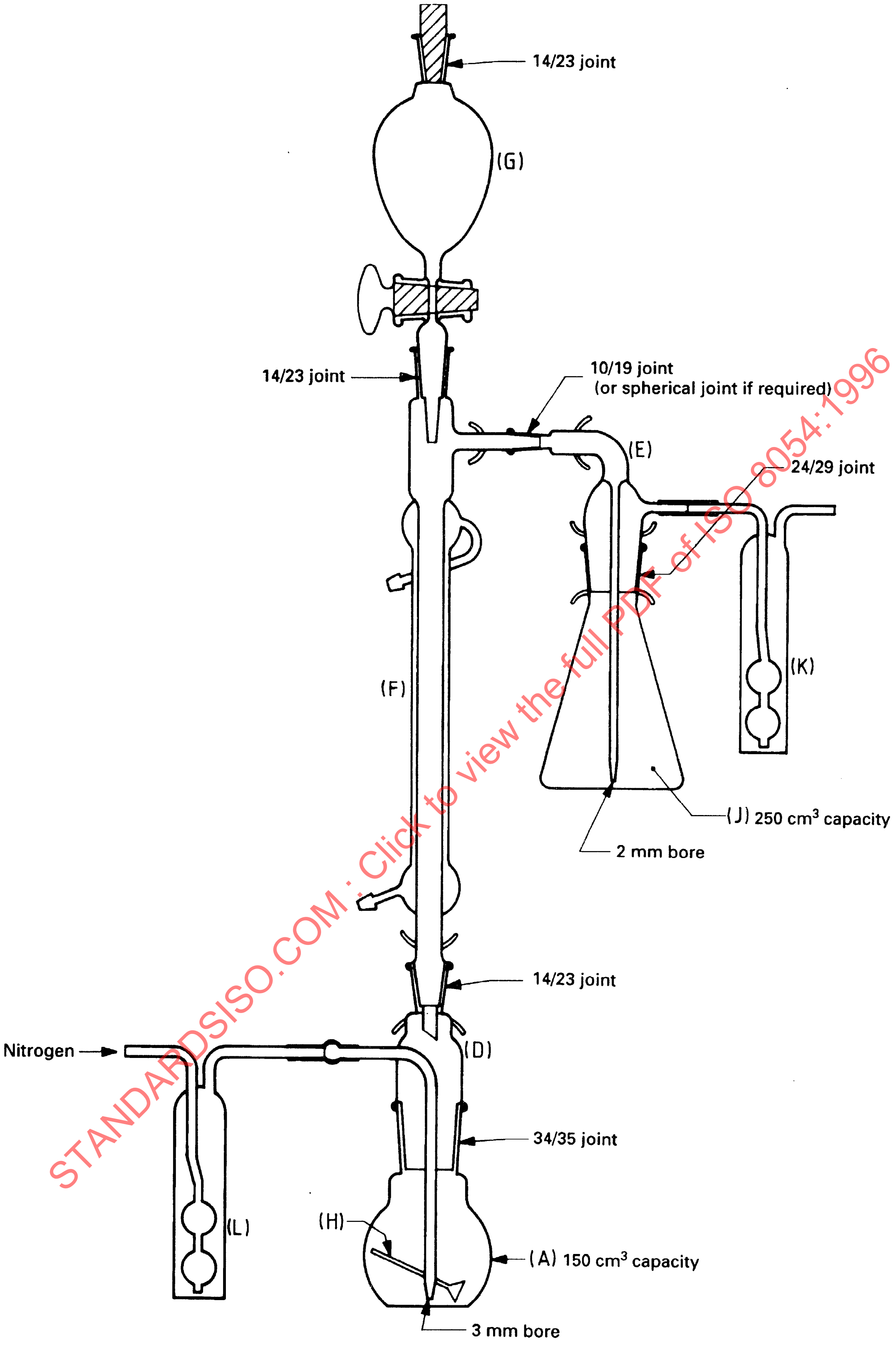

Figure 2 — Typical apparatus for the evolution and absorption of hydrogen sulfide

8 Expression of results

Calculate the sulfide sulfur content, expressed as the percentage of S by mass, from the formula

$$\frac{1{,}6(V_2 - V_1)c}{m}$$

where

- V_1 is the volume, in cubic centimetres, of the sodium thiosulfate solution (4.6) used in the titration described in 7.1.3;
- V_2 is the volume, in cubic centimetres, of the sodium thiosulfate solution (4.6) used in the blank titration described in 7.2;
- c is the actual concentration, in moles of $Na_2S_2O_3 \cdot 5H_2O$ per cubic decimetre, of the sodium thiosulfate solution (4.6);
- m is the mass, in grams, of the test portion (6.1).

The test result shall be the mean, rounded to one decimal place, of two determinations.

9 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for the identification of the sample;

c) the results and the units in which they are expressed;

d) any unusual features noted during the determination;

e) any operation not specified in this International Standard or in the International Standards to which reference is made, and any operation regarded as optional;

f) the date of the test.