
INTERNATIONAL STANDARD**1408**

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Vulcanized rubber — Determination of carbon black content — Pyrolytic method

Caoutchouc vulcanisé — Détermination de la teneur en noir de carbone — Méthode pyrolytique

iTeh STANDARD PREVIEW

First edition — 1976-10-15

(standards.iteh.ai)

ISO 1408:1976

<https://standards.iteh.ai/catalog/standards/sist/ea7baf17-7f33-4b76-a8e1-54592e6f765d/iso-1408-1976>

UDC 678.4/7.063 : 543.824.4

Ref. No. ISO 1408-1976 (E)

Descriptors : vulcanized elastomers, natural rubber, synthetic elastomers, chemical analysis, determination of content, carbon black, pyrolytic analysis.

Price based on 2 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the technical committees were published as ISO Recommendations; these documents are in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 45, *Rubber and rubber products*, has reviewed ISO Recommendation R 1408-1971 and found it technically suitable for transformation. International Standard ISO 1408 therefore replaces ISO Recommendation R 1408-1971, to which it is technically identical. <https://www.iso.org/standards/catalog/standards/sist/ea7baf17-7f33-4b76-a8e1-54592e6f765d/iso-1408-1976>

ISO Recommendation R 1408 had been approved by the member bodies of the following countries :

Australia	India	Poland
Austria	Iran	South Africa, Rep. of
Brazil	Israel	Spain
Canada	Italy	Sweden
Egypt, Arab Rep. of	Japan	Switzerland
France	Korea, Dem. P. Rep. of	United Kingdom
Germany	Netherlands	U.S.A.
Hungary	New Zealand	

The member body of the following country had expressed disapproval of the Recommendation on technical grounds :

Czechoslovakia*

- * Subsequently, this member body approved the Recommendation.

No member body disapproved the transformation of the Recommendation into an International Standard.

Vulcanized rubber — Determination of carbon black content — Pyrolytic method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a pyrolytic method for the determination of the carbon black content of vulcanizates of the following polymers :

Natural rubber
Synthetic polyisoprene
Polybutadiene
Styrene butadiene rubber
Butyl rubber
Acrylate rubber
Ethylene-propylene copolymer
Ethylene-propylene terpolymer

This method is not suitable for vulcanizates containing halogens or nitrogen in the polymer or for those containing certain compounding materials, such as lead salts or phenolic resins, which cause the formation of a carbonaceous residue during pyrolysis.

The precision of the method may be affected if mineral fillers are present which dissociate at the pyrolysis temperature.

2 REFERENCE

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

3 PRINCIPLE

Extraction of a weighed test portion of the rubber with acetone and, if bitumen is present, with chloroform. Pyrolysis of the extracted rubber in a combustion boat at 800 to 900 °C in a stream of nitrogen. Cooling and weighing of the boat containing the non-volatile residue. Burning off of the carbon black in a muffle furnace. Cooling and reweighing of the boat and its contents. The loss in mass represents the carbon black.

4 REAGENTS

4.1 Nitrogen, dry and free from oxygen.

NOTE — Nitrogen sold commercially as "free from oxygen" may require further purification.

4.2 Xylene.

4.3 Acetone, analytical reagent grade.

4.4 Chloroform, analytical reagent grade.

5 APPARATUS

5.1 Combustion boat, made of silica, of length 50 to 60 mm, with handle.

5.2 Electric tube furnace, capable of being controlled at 800 to 900 °C, with means for advancing and withdrawing the combustion boat. One end of the tube is provided with a gas-inlet system for nitrogen, the other end with a suitable outlet system for the vapours produced during pyrolysis.

5.3 Vapour absorption system, consisting of :

5.3.1 Trap for readily condensable vapours.

5.3.2 Xylene trap for uncondensed vapours.

5.4 Muffle furnace, capable of being controlled at 800 to 900 °C.

5.5 Suitable extraction apparatus, as specified in ISO 1407.

5.6 Desiccator.

6 PROCEDURE

Weigh about 0,1 to 0,2 g of the sample to the nearest 0,000 2 g, wrap in filter paper and extract first with the acetone (4.3) and then, if bitumen is present, with the chloroform (4.4) in the extraction apparatus (5.5) for at least 4 h with each solvent.

NOTE — Extraction with chloroform is only necessary if materials not completely soluble in acetone, such as bitumen, are present.

Extraction can be facilitated by comminuting the sample before weighing. To do this, pass it through a mill with a minimum clearance between the rolls.

Remove the extracted rubber from the paper and dry in air at a temperature not higher than 100 °C. Heat the electric tube furnace (5.2) to a temperature of 800 to 900 °C, place the test portion in the combustion boat (5.1) and then place the boat in the entrance to the combustion tube and close the tube with the entry fitting.

Connect the supply of oxygen-free nitrogen through a flow meter to the inlet system, and connect the vapour absorption system (5.3) to the outlet of the combustion tube. Pass nitrogen through the furnace at about 200 cm³/min for 5 min; then reduce the rate of flow to 100 cm³/min and move the boat slowly into the centre of the furnace over a period of about 5 min. Leave the boat in the hot zone of the furnace for a further 5 min. At the end of the period, withdraw the boat to the cold part of the tube and allow to cool for 10 min, while still maintaining the flow of nitrogen. Then take out the boat, complete the cooling in the desiccator (5.6) and weigh at ambient temperature (mass m_1).

Place the boat in the muffle furnace (5.4) controlled at a temperature of 800 to 900 °C and ignite it until all traces of carbon have been removed. Cool the boat in the desiccator and weigh (mass m_2).

7 EXPRESSION OF RESULTS

The carbon black content is given, as a percentage by mass, by the formula

$$\frac{m_1 - m_2}{m_0} \times 100$$

where

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

m_1 is the mass, in grams, of the combustion boat and extract after removal from the combustion tube;

m_2 is the mass, in grams, of the combustion boat and ash after ignition.

8 TEST REPORT

The test report shall include the following particulars :

- a) identification of the sample;
- b) carbon black content.

ISO 1408:1976

<https://standards.iteh.ai/catalog/standards/sist/ea7baf17-7f33-4b76-a8e1-54592e6f765d/iso-1408-1976>