International Standard 4645

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Rubber and rubber products — Guide to the identification of antidegradants — Thin layer chromatographic methods

Caoutchouc et produits en caoutchouc — Guide d'identification des agents de protection — Méthodes par chromatographie en couche mince

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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. Every 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.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4645 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

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Rubber and rubber products — Guide to the identification of antidegradants — Thin layer chromatographic methods

1 Scope and field of application

1.1 This International Standard describes two methods for the detection and identification, by thin layer chromatography, of antidegradants (antioxidants, antiozonants and stabilizers), which may be present in raw rubber, unvulcanized compounded rubber, or rubber products.

Method A is a simplified method, based on a single solvent system, which provides for the identification of known materials and may be used to check the presence or absence of a particular antidegradant which should be present.

Method B is a more detailed method, using additional solvents and sprays, which enables a greater degree of separation of the spots to be obtained and therefore may be used to detect and identify an unknown antidegradant.

1.2 Antidegradants to which these methods are applicable include phosphited polyalkyl phenols, substituted bisphenols, secondary amines, substituted cresols and substituted *p*-phenylenediamines. Examination for other types of antidegradants is possible, provided that the requirement of 11.1 is met.

2 Reference

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

3 Principle

Extraction of antidegradants from the rubber by means of a solvent. Evaporation of the original solvent, application of a solution of the dried extract as a spot on a thin layer chromatographic plate, evaporation of the second solvent and development of the plate in an appropriate solvent. If extender oil is present, removal of the oil either by column chromatography of the extract prior to the completion of the evaporation of the original solvent or by development of the plate in light petroleum prior to the normal development in an appropriate solvent. Identification of the unknown antidegradant by comparison of its chromatogram with standard chromatograms.

4 Reagents

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

WARNING — Use of fume hoods when handling volatile and toxic solvents is mandatory. Approved health and safety precautions shall be observed when using any solvent or chemical mentioned in this International Standard.

4.1 Plate adsorbent: silica gel, particle size 2 to 50 µm, with or without calcium sulphate binder.[1)]

Silica gel containing a fluorescent indicator is useful in many cases to allow observation of spots, under ultra-violet radiation, before spraying.

4.2 Column adsorbent: silica gel, to pass a sieve of aperture 200 to 600 µm[1)] activated by drying, either

— for at least 2 h at 110 °C, if the product is dry after that period, or

— overnight (approximately 16 h at 110 °C) for convenience.

4.3 Solvents:

4.3.1 Methanol.

4.3.2 Acetone.

4.3.3 Ethanol, anhydrous.

4.3.4 2-Propanol.

4.3.5 Light petroleum, boiling range 35 to 60 °C.

4.3.6 Dichloromethane.

4.3.7 Toluene.

1) Suitable material is available commercially. Details may be obtained from the Secretariat of ISO/TC 45 (BSI) or ISO Central Secretariat.