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Rubber, raw synthetic — Determination of anti-degradants by high-performance liquid chromatography

Caoutchouc synthétique brut — Détermination des agents de protection par chromatographie en phase liquide à haute performance

Reference number
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Foreword

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Rubber, raw synthetic — Determination of anti-degradants by high-performance liquid chromatography

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 describes a procedure for the determination of the following anti-degradants in raw synthetic rubbers:

N-alkyl-*N*′-phenyl-*p*-phenylenediamine;
N-aryl-*N*′-aryl-*p*-phenylenediamine;
N-phenyl-β-naphthylamine;
poly-2,2,4-trimethyl-1,2-dihydroquinoline.

Extender oils, when present, may interfere.

The method, with modification if necessary, may be applied to the determination of other amine anti-degradants.

NOTE — The method has been tested successfully on CR, NBR and SBR.

2 Principle

The anti-degradant, following quantitative extraction from the rubber, is separated by high-performance liquid chromatography (HPLC) from other extracted components, and its component peaks detected and their areas measured. Its concentration in the rubber is calculated by comparison with the area of the anti-degradant component peaks of a known amount of the same anti-degradant analysed under the same analysis conditions.

NOTE — Some anti-degradants contain more than one component peak and the ratio of the areas of single component peaks may differ depending on the source of the anti-degradant. The procedure described will give accurate results providing the composition of the reference anti-degradant and the anti-degradant being analysed contain the same number of component peaks with the same area ratios. Inaccurate results are obtained when this condition is not satisfied, with the extent of the error being dependent on the difference in composition between reference and sample anti-degradants.

3 Materials

3.1 Eluent A: a 1:1 (*V*/*V*) mixture of methanol (HPLC grade) and a 0,01 M aqueous solution of ammonium acetate (AR grade).

3.2 Eluent B: methanol (HPLC grade).

3.3 Extraction solvent: a 2:1 (*V*/*V*) mixture of isopropanol (HPLC grade) and dichloromethane (HPLC grade).

4 Apparatus

4.1 HPLC apparatus, with gradient elution capability, a 10 mm^3 fixed-loop injector, a variable-wavelength ultraviolet-visible (UV-VIS) detector and a recording-integrator data system.

4.2 HPLC column, reverse-phase type.

NOTE — Different columns may be used provided a good separation of anti-degradant component peaks from other extracted components is obtained. The method has been tested using 5 μm particle size HYPERSIL ODS and SPHERI 5 ODS[1)] columns. However, the elution programme may need to be modified when columns different from the ones described in this International Standard are used.

4.3 Ultrasonic bath, typically of about 2 dm^3 capacity, operating at a frequency of 47,6 kHz ± 10 %.

NOTE — An ultrasonic bath of different capacity and operating frequency may be used provided that extraction of the anti-degradant is complete.

4.4 Analytical balance, capable of weighing to 0,01 mg.

5 Chromatographic conditions

5.1 Pump A: Elution solvent A (3.1).

5.2 Pump B: Elution solvent B (3.2).

5.3 Flow rate: 0,25 cm^3/min.

5.4 Column oven temperature: 40 °C.

5.5 Injection volume: 10 mm^3 (10 μl).

5.6 Detector wavelength:

poly-2,2,4-trimethyl-1,2-dihydroquinoline: 233 nm;

all other anti-degradants: 295 nm.

5.7 Reference wavelength: 550 nm.

5.8 Elution programme

Time min	Eluent A %	Eluent B %
0	100	0
20	0	100
40	0	100
50	100	0
55	End	

1) HYPERSIL ODS and SPHERI 5 ODS are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

6 Procedure

6.1 On a laboratory mill, thinly sheet, to about 0,25 mm to 0,5 mm thickness, a representative sample of the rubber to be analysed. Cut about 1 g of the thinly sheeted sample into small pieces about 5 mm square. Carry out the procedure in 6.2 to 6.10 twice, on duplicate test portions.

6.2 Weigh, to the nearest 0,1 mg, about 200 mg of the small pieces and place each test portion into a 20 cm^3 flask.

6.3 Add accurately, by pipette, 10 cm^3 of extraction solvent (3.3) and stopper the flask.

6.4 Extract in the ultrasonic bath (4.3) for 3 h at a temperature not exceeding 30 °C.

NOTE — Should the bath temperature exceed 30 °C, the flask may rupture. Therefore it may be necessary periodically to add cold water to the bath during the extraction in order to maintain the bath below 30 °C.

6.5 Weigh, to the nearest 0,01 mg, a quantity of the reference anti-degradant as close to the expected quantity of anti-degradant contained in the test portion and place in a 20 cm^3 flask.

6.6 Add accurately, by pipette, 10 cm^3 of extraction solvent (3.3), stopper the flask and dissolve the reference anti-degradant, using the ultrasonic bath below 30 °C, if necessary.

6.7 Inject into the HPLC column (3.2) a 10 mm^3 volume of the rubber sample extract prepared in 6.4 and elute in accordance with the programme given in 5.8.

6.8 Record the areas of the sample anti-degradant peaks.

6.9 Inject into the HPLC column a 10 mm^3 volume of the reference anti-degradant solution prepared in 6.6 and elute in accordance with the programme given in 5.8.

6.10 Record the areas of the reference anti-degradant peaks.

7 Expression of results

Calculate the concentration of anti-degradant in the sample for each of the two determinations, using the following equation:

$$\% \text{ anti-degradant} = \frac{m_s \times A_c}{m_c \times A_s} \times 100$$

where

m_s is the mass, in milligrams, of the reference anti-degradant test portion;

m_c is the mass, in milligrams, of the sample test portion;

A_s is the area of the reference anti-degradant peaks;

A_c is the area of the sample anti-degradant peaks.

Record the average of the two results.

8 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

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

c) the concentration, in percent, of the anti-degradant in the sample;

d) the date of the test.

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Descriptors: rubber, synthetic rubber, crude rubber, chemical analysis, determination of content, antioxidants, high performance liquid chromatography.

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