

DETERMINATION OF ANTI-DEGRADANTS IN RUBBER BY THIN LAYER CHROMATOGRAPHY

Application

This procedure describes a method for "fingerprinting" the anti-degradants (e.g. anti-ozonants, anti-oxidants, anti-fatigue agents) in rubber compounds, and as such is used to check consistency of formulation for specific applications where close control is required. The method may also be used in certain cases for comparison work (e.g. to measure loss of anti-degradant from rubber due to environmental factors).

The method is not intended to be used, either for identification of anti-degradants or to predict the performance of a rubber compound, unless a degree of caution in interpretation is applied.

Equipment Required

Thin Layer Plates

Silica gel 60 F 254 precoated size 20 x 20 cm; layer thickness 0.25 mm.

Sources: Merck, Andermann, BDH

Developing Tank with Lid

22 x 6 x 21 mm or close approximation.

Syringe

5.0 µl capacity

Spotting Guide (optional)

Aerosol Spray Unit

A fine spray type as supplied for use in chromatography.

Spray Reagent

A freshly prepared 1 % solution of 2:6 dibromo-p-benzoquinone-4-chlorimine in industrial methylated spirit, unless otherwise stated on appropriate specification.

***Developing Solvent**

A mixture of the following:

Benzene	100 parts
Ethyl acetate	5 parts
Acetone	2 parts

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2001 01 31	Revised	Editorial – no technical change A. Cockman
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Reference solutions

Solutions of anti-degradant are used for reference purposes in order to ensure consistency of the technique and for comparisons of spot intensity. An 0.1 % solution in acetone of one or more of the following may be used unless otherwise required:

Suppliers

N, N-diphenylpPhenyleneDiamine	ICI, Monsanto
N-Phenyl-B-Naphthylamine	Bayer, ICI, Monsanto
Acetone/Diphenylamine Reaction Product	ICI, Monsanto
6-Ethoxy-2,2,4-Trimethyl-1,2-Dihydroquinoline	Monsanto

Standard Laboratory Equipment

Such as filter paper, flasks, soxhlet, etc.

Conditioning and Test Conditions

All test values indicated herein are based on material conditioned in a controlled atmosphere of 23 +/- 2 °C and 50 +/- 5 % relative humidity for not less than 24 h prior to testing and tested under the same conditions unless otherwise specified.

Procedure

1. Extract the rubber compound (1.0 g) for 24 h in acetone. See ASTM D 297 for details. Concentrate the extract to 3 mL.
2. Prepare the developing tank by lining with filter paper saturated with the developer solution, and maintain a depth of approx 10 mm at the bottom of the tank. (Tank must be cleaned and solution changed once per 2 days minimum). Tank should be covered and allowed to stand overnight for the atmosphere to equilibrate.
3. Mark out TLC plate with pencil as shown on the accompanying sketch.
4. Preheat plate for 1 h at 75 °C then cool.
5. Using syringe, spot 5 µl of extract solution onto plate as shown on line B. Do likewise with the reference solution(s) ensuring that there is adequate spacing (12 mm min) between spots. To keep spot size to a minimum it is helpful to dry during spotting with clean pressurized air.