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Rubber latex, styrene-butadiene — Determination of volatile unsaturates

Latex de butadiène-styrène — Dosage des composés non saturés volatils

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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.

International Standard ISO 2008 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This second edition was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the first edition (i.e. ISO 2008-1972), which had been approved by the member bodies of the following countries :

| | | |
|---------------------|-----------------------|----------------|
| Australia | Hungary | Switzerland |
| Austria | India | Thailand |
| Canada | Israel | Turkey |
| Ceylon | Italy | United Kingdom |
| Egypt, Arab Rep. of | New Zealand | USA |
| France | South Africa, Rep. of | USSR |
| Germany, F.R. | Spain | |
| Greece | Sweden | |

No member body had expressed disapproval of the document.

Rubber latex, styrene-butadiene — Determination of volatile unsaturates

0 Introduction

The first edition of this International Standard specified methods for the determination of both volatile unsaturates and residual styrene in styrene-butadiene rubber latices. On review, the method for volatile unsaturates was confirmed but the ultra-violet spectrophotometric method for residual styrene was withdrawn because it was not sufficiently specific to styrene and was little used.

This second edition refers, therefore, only to volatile unsaturates. A gas chromatographic method specific to residual styrene will form the subject of a future International Standard.

1 Scope and field of application

This International Standard specifies a method for the determination of volatile unsaturates in styrene-butadiene rubber latices.

The method measures, in addition to residual styrene, other unsaturates such as butadiene dimer.

2 Principle

Distillation of a test portion with methanol and collection of the distillate.

Addition of potassium bromate/bromide solution to the distillate and, after addition of potassium iodide, titration of the liberated iodine with sodium thiosulphate.

3 Reagents

Use only reagents of recognized analytical grade and distilled water or water of equivalent purity.

3.1 Methanol reagent: methanol containing 100 mg/kg (100 ppm) of *p*-tert-butyl catechol or an equivalent polymerization inhibitor.

3.2 Potassium bromate/potassium bromide, standard volumetric solution, $c(\text{KBr}, 1/6 \text{ KBrO}_3) = 0,1 \text{ mol/l.}^{1)}$

Dissolve 2,784 g of potassium bromate (KBrO_3) and 10,0 g of potassium bromide (KBr) in water and dilute to 1 000 ml in a one-mark volumetric flask.

3.3 Sulphuric acid, 18 % (*m/m*) solution.

3.4 Potassium iodide, 10 % (*m/m*) solution.

3.5 Sodium thiosulphate, standard volumetric solution, $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,1 \text{ mol/l.}^{1)}$

3.6 Indicator, starch solution or equivalent.

4 Apparatus

4.1 Dean and Stark distillation apparatus, including a distillation flask of capacity 500 ml and a 25 ml receiver, or equivalent distillation apparatus with ground glass joints.

4.2 Iodine flask, of capacity 250 ml.

5 Procedure

5.1 Test portion

Weigh $25,0 \pm 0,2 \text{ g}$ of latex into the distillation flask (see 4.1).

5.2 Determination

Add 25 ml of water and 25 ml of the methanol reagent (3.1) to the test portion (5.1). Distil the mixture, adjusting the rate of boiling to control frothing, and collect the first 25 ml of distillate in the receiver.

1) Hitherto expressed as "0,1 N standard volumetric solution".

Transfer the distillate to the iodine flask (4.2) and rinse the condenser and receiver into the iodine flask with 20 ml of the methanol reagent. If desired, the distillate may be collected in the iodine flask.

From a burette add 20 ml of the potassium bromate/bromide solution (3.2), and cool the solution to 30 °C.

Rapidly add 15 ml of the sulphuric acid solution (3.3), stopper the flask, shake, and add water to the funnel lips as a vapour seal. If no yellow colour remains after allowing the flask to stand for a least 60 s, add successive 10 ml portions of the potassium bromate/bromide solution until a slight yellow colour persists for 60 s after the addition. Make the additions by running the solution from the burette into the funnel lip and lifting the stopper so that the solution enters the flask around the stopper. Wash the funnel lip with water in the same manner and seal with water.

After the final addition of the potassium bromate/bromide solution, add 10 ml of the potassium iodide solution (3.4) to the funnel lip and lift the stopper to allow the solution to enter the flask around the stopper.

Shake the flask and contents and titrate the liberated iodine with the sodium thiosulphate solution (3.5) to a faint yellow colour. Add 1 ml of the indicator solution (3.6) and continue the titration with the sodium thiosulphate solution until the solution is colourless.

5.3 Blank test

Carry out a blank test by repeating the procedure, but using 25 ml of water in place of the test portion.

6 Expression of results

The volatile unsaturates content, expressed as a percentage by mass as styrene, is given by the formula.

$$0,208 \times c (V_1 - V)$$

where

c is the concentration, in moles per litre, of the sodium thiosulphate solution;

V₁ is the volume, in millilitres, of sodium thiosulphate solution used in the blank test;

V is the volume, in millilitres, of sodium thiosulphate solution used in the determination.

7 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 results and method of expression used;
- d) any unusual features noted during the determination;
- e) any operation not included in this International Standard or regarded as optional.