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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 bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each 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 2008 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This third edition cancels and replaces the second edition (ISO 2008, 1980), of which it constitutes a minor revision.

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Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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method for volatile unsaturates was confirmed but the ultra-violet spectrophotometric method for residual styrene was withdrawn because it was not sufficiently specific to styrene and was little used. The second edition referred, therefore, only to volatile unsaturates.

The second edition was reviewed in 1985, and it was agreed that a new edition was required to incorporate several minor, and essentially editorial, changes. These are included in this present, third, edition.

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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

A test portion is distilled with methanol and the distillate is collected. Potassium bromate/bromide solution is added to the distillate and, after addition of potassium iodide, the liberated iodine is titrated with sodium thiosulfate.

3 Reagents

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

3.1 Methanol reagent: methanol containing 0,01 g/kg (10 ppm) of *p*-tert-butyl catechol or an equivalent polymerization inhibitor.

3.2 Potassium bromate/bromide, standard volumetric solution, $c(\text{KBr}, 1/6 \text{ KBrO}_3) = 0,1 \text{ mol/dm}^3$.

Dissolve 2,784 g of potassium bromate (KBrO_3) and 10,0 g of potassium bromide (KBr) in water and dilute to 1 000 cm^3 in a one-mark volumetric flask.

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

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

3.5 Sodium thiosulfate, standard volumetric solution, $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,1 \text{ mol/dm}^3$.

3.6 Indicator, starch solution or equivalent.

4 Apparatus

4.1 Dean and Stark distillation apparatus, including a distillation flask of capacity 500 cm^3 and a receiver suitable to hold 25 cm^3 of distillate, or equivalent distillation apparatus with ground glass joints.

4.2 Iodine flask, of capacity 250 cm^3 .

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 cm^3 of water and 25 cm^3 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 cm^3 of distillate in the receiver.

Transfer the distillate to the iodine flask (4.2) and rinse the condenser and receiver into the iodine flask with 20 cm^3 of the methanol reagent. If desired, the distillate may be collected in the iodine flask.

From a burette add 20 cm^3 of the potassium bromate/bromide solution (3.2), and cool the solution to 30 °C.

Rapidly add 15 cm³ of the sulfuric 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 cm³ portions of the potassium bromate/bromide solution from the burette until a slight yellow colour persists for 60 s after the addition. Make the additions by running the solution 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. Note the total volume of potassium bromate/bromide solution that has been used.

After the final addition of the potassium bromate/bromide solution, add 10 cm³ 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. Wash the funnel lip with water as described above.

Shake the flask and contents and titrate the liberated iodine with the sodium thiosulfate solution (3.5) to a faint yellow colour. Add 1 cm³ of the indicator solution (3.6) and continue the titration with the sodium thiosulfate solution until the solution is colourless.

5.3 Blank test

Carry out a blank test by repeating the procedure, but using 25 cm³ of water in place of the test portion. Accurately add the same volume of potassium bromate/bromide solution as recorded for 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 thiosulfate solution;

V_1 is the volume, in cubic centimetres, of sodium thiosulfate solution used in the blank test;

V is the volume, in cubic centimetres, of sodium thiosulfate solution used in the determination.

7 Test report

The test report shall include the following particulars :

- a reference to this International Standard;
- all details necessary for the identification of the sample;
- the results and method of expression used;
- any unusual features noted during the determination;
- any operation not included in this International Standard or regarded as optional.

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