BS 6057 : Part 3 : Section 3.15 : 1984
ISO 2008-1980

UDC 678.031 : 543.871 : 543.813

British Standard

Rubber latices

Part 3. Methods of test

Section 3.15 Determination of volatile unsaturates content of styrene-butadiene rubber latices

[ISO title: Rubber latex, styrene-butadiene — Determination of volatile unsaturates]

Latex de caoutchouc
Partie 3. Méthodes d'essai
Section 3.15 Dosage des composés non saturés volatils du latex de butadiène-styrène

Kautschuklatex
Teil 3. Prüfverfahren
Abschnitt 3.15 Bestimmung der flüchtigen, ungesättigten Bestandteile von Styrol-Butadien-Latex

NOTE. Attention is drawn to BS 6057 : Part 0 'General introduction', issued separately.

National foreword

This Section of BS 6057 is identical with ISO 2008-1980 'Rubber latex, styrene-butadiene — Determination of volatile unsaturates' published by the International Organization for Standardization (ISO). It supersedes method 8.4 of BS 3397 : 1976 'Methods of test for synthetic rubber latices'.

The main change incorporated in this standard is the inclusion of distillation apparatus with ground glass joints as an alternative to the Dean and Stark distillation apparatus (see **4.1**).

Terminology and conventions. The text of the international standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

Wherever the words 'International Standard' appear, referring to this standard, they should be read as 'British Standard'.

Additional information. Water complying with BS 3978 'Water for laboratory use' is suitable for use in this determination (see clause **3**).

A volumetric flask complying with BS 1792 'Specification for one-mark volumetric flasks' is recommended for use in the preparation of the potassium bromate/potassium bromide solution (see **3.2**). It is also recommended that the Dean and Stark distillation apparatus (see **4.1**), if used, should comply with BS 756 'Dean and Stark apparatus' and the iodine flask (see **4.2**) should comply with BS 2735 'Iodine flasks'.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

British Standards Institution

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, $c(KBr, 1/6\ KBrO_3) = 0{,}1$ 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, $c(Na_2S_2O_3) = 0{,}1$ 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 ± 0,2 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 thiosuphate 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;

V_1 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.

Publications referred to

See national foreword.

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This British Standard, having been prepared under the direction of the Rubber Standards Committee, was published under the authority of the Board of BSI and comes into effect on

31 January 1984.

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The Committees responsible for this British Standard are shown in Part 0.

The following BSI references relate to the work on this standard:
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Amendments issued since publication

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