BS 6057 : Part 3 : Section 3.15 : 1989
ISO 2008 : 1987

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

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 : 1987 'Rubber latex, styrene-butadiene–Determination of volatile unsaturates' published by the International Organization for Standardization (ISO). It supersedes BS 6057 : Part 3 : Section 3.15 : 1984, which is withdrawn.

Additional information. Water complying with grade 3 of 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 'Specification for Dean and Stark apparatus' and the iodine flask (see **4.2**) should comply with BS 2735 'Specification for 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method for volatile unsaturates was confirmed but the ultra-violet spectrophotometric method for residual styrene was withdrawn because it was not sufficiently specific to styrene and was little used. 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.

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, $c(KBr, 1/6\ KBrO_3) = 0{,}1$ 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, $c(Na_2S_2O_3) = 0{,}1$ 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 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.