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Styrene butadiene rubber latices — Determination of volatile unsaturates and residual styrene

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FOREWORD

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Australia	Hungary	Switzerland
Austria	India	Thailand
Canada	Israel	Turkey
Ceylon	Italy	United Kingdom
Egypt, Arab Rep. of	New Zealand	U.S.A.
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Germany	Spain	
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Styrene butadiene rubber latices — Determination of volatile unsaturates and residual styrene

1 SCOPE AND FIELD OF APPLICATION

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

The method for volatile unsaturates measures, in addition to residual styrene, other unsaturates such as butadiene dimer. The second method measures only residual styrene, and may not be valid if the spectrum does not conform to that of a styrene solution of the same concentration over the wavelength range 220 to 300 nm.

2 PRINCIPLE

The latex is distilled with methanol and the distillate is collected.

For the determination of volatile unsaturates, potassium bromate/bromide solution is added to the distillate and after addition of potassium iodide the liberated iodine is titrated with sodium thiosulphate.

For the determination of residual styrene, the styrene content of the distillate is obtained by ultra-violet spectrophotometry.

3 METHOD FOR VOLATILE UNSATURATES

3.1 Reagents

All reagents shall be of recognized analytical reagent quality and distilled water or water of equivalent purity shall be used wherever water is specified.

3.1.1 *Methanol reagent.* Methanol containing 100 ppm of paratertiary butyl catechol or an equivalent polymerization inhibitor.

3.1.2 *Potassium bromate/potassium bromide* standard volumetric solution, 0.1 N.

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.

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

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

3.1.5 *Sodium thiosulphate* solution, 0.1 N.

3.1.6 *Indicator.* Starch solution or equivalent.

3.2 Apparatus

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

3.2.2 *Iodine flask,* 250 ml..

3.3 Procedure

Weigh 25.0 ± 0.2 g of latex into the distillation flask, and add 25 ml of water and then 25 ml of methanol reagent (3.1.1). Distil the mixture, adjusting the rate of boiling to control frothing, and collect the first 25 ml of distillate in the receiver.

Transfer the distillate to the iodine flask (3.2.2) and rinse the condenser and receiver into the iodine flask with 20 ml of 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 (3.1.2), and cool the solution to 30 °C.

Rapidly add 15 ml of the sulphuric acid (3.1.3), stopper the flask, shake it, and add water to the funnel lips as a vapour seal. If no yellow colour remains after standing the flask for at least 60 s, add successive 10 ml portions of the potassium bromate/bromide solution (3.1.2) 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 potassium bromate/bromide solution (3.1.2), add 10 ml of the potassium iodide solution (3.1.4) 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 (3.1.5) to a faint yellow colour. Add 1 ml of starch indicator solution (3.1.6) and continue the titration with the sodium thiosuphate solution until the solution is colourless.

Carry out a blank determination by repeating the whole procedure with 25 ml of water in place of the latex.

3.4 Expression of results

Calculate the volatile unsaturates content as styrene as follows :

$$\text{Volatile unsaturates, \% (m/m) of latex} = 0.208 \times N\,(V_1 - V)$$

where

N is the normality of the sodium thiosulphate solution;

V_1 is the volume, in millilitres, of sodium thiosulphate solution used in the blank titration;

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

4 METHOD FOR RESIDUAL STYRENE

4.1 Reagents

All reagents shall be of recognized analytical reagent quality and distilled water or water of equivalent purity shall be used wherever water is specified.

4.1.1 *Methanol reagent.* Methanol containing 100 ppm of paratertiary butyl catechol or an equivalent polymerization inhibitor.

4.1.2 *Methanol.*

4.1.3 *Styrene.*

4.2 Apparatus

4.2.1 *Ultra-violet spectrophotometer.*

4.2.2 *Dean and Stark distillation apparatus,* with distillation flask of 500 ml capacity and 25 ml receiver.

4.3 Procedure

Weigh 10.0 ± 0.1 g of latex into the distillation flask, and add 100 ml of water and 30 ml of methanol reagent (4.1.1). Distil the mixture, adjusting the rate of boiling to control frothing, and collect the first 25 ml of distillate in the receiver.

Drain the distillate into a one-mark volumetric flask, rinse the receiver into the flask with methanol (4.1.2), make up to the mark with methanol, stopper and agitate thoroughly. The capacity of the flask shall be selected so that the optical density of the solution which it contains, determined as in the following paragraph, is within the limits for minimum photometric error. These limits depend on the particular spectrophotometer and are approximately 0.2 to 0.7.

Measure the optical density of the solution, using 10 mm silica cells in the ultra-violet spectrophotometer (4.2.1) with methanol in the reference cell, at the point of maximum absorption within the wavelength range 282 ± 3 nm.

4.4 Expression of results

Calculate the residual styrene content as follows :

$$\text{Residual styrene, \% (m/m) of latex} = \frac{A}{K} \times \frac{V_0}{100}$$

where

A is the optical density at the point of maximum absorption within the range 282 ± 3 nm;

K is the specific extinction coefficient, determined as in 4.5;

V_0 is the volume, in millilitres, of the volumetric flask.

4.5 Determination of K

Weigh, to the nearest 1 mg, about 500 mg of freshly distilled styrene and dilute to 500 ml with methanol in a one-mark volumetric flask. Pipette 3 ml of the solution and dilute to 100 ml with methanol in a one-mark volumetric flask (Solution A). Similarly, prepare Solutions B and C containing respectively 5 ml and 8 ml per 100 ml of the first solution.

Measure the optical densities of Solutions A, B and C, using 10 mm silica cells in the ultra-violet spectrophotometer with methanol in the reference cell, each at the point of maximum absorption within the wavelength range 282 ± 3 nm.

Calculate K for Solutions A, B and C as follows :

For Solution A $K = 16.7\,A/m$
For Solution B $K = 10\,A/m$
For Solution C $K = 6.25\,A/m$

where

A is the optical density at the point of maximum absorption within the range 282 ± 3 nm;

m is the mass, in grams, of styrene taken.

The three results for K shall lie within a range of 0.2, and the average of the three results shall be taken as the specific extinction coefficient.

5 TEST REPORT

The test report shall include the following particulars :

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.