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METHOD 312B—DETERMINATION OF RESIDUAL STYRENE IN STYRENE-BUTADIENE (SBR) RUBBER LATEX BY CAPILLARY GAS CHROMATOGRAPHY

1.0 Scope

1.1 This method is applicable to SBR latex solutions.

1.2 This method quantitatively determines residual styrene concentrations in SBR latex solutions at levels from 80 to 1200 ppm.

2.0 Principle of Method

2.1 A weighed sample of a latex solution is coagulated with an ethyl alcohol (EtOH) solution containing a specific amount of alpha-methyl styrene (AMS) as the internal standard. The extract of this coagulation is then injected into a gas chromatograph and separated into individual components. Quantification is achieved by the method of internal standardization.

3.0 Definitions

3.1 The definitions are included in the text as needed.

4.0 Interferences [Reserved]

5.0 Safety

5.1 This method may involve hazardous materials, operations, and equipment. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

6.0 Equipment and Supplies

6.1 Analytical balance, 160 g capacity, and 0.1 mg resolution

6.2 Bottles, 2-oz capacity, with poly-cap screw lids

6.3 Mechanical shaker

6.4 Syringe, 10-ul capacity

6.5 Gas chromatograph, Hewlett Packard model 5890A, or equivalent, configured with FID with a megabore jet, splitless injector packed with silanized glass wool.

6.5.1 Establish the following gas chromatographic conditions, and allow the system to thoroughly equilibrate before use.

Injection technique = Splitless

Injector temperature = 225 deg C

Oven temperature = 70 deg C (isothermal)

Detector: temperature = 300 deg C

range = 5

attenuation = 0

Carrier gas: helium = 47 ml/min

Detector gases: hydrogen = 30 ml/min

air = 270 ml/min

make-up = 0 ml/min

Analysis time: = 3.2 min at the specified carrier gas flow rate and column temperature.

6.6 Gas chromatographic column, DB-1, 30 M X 0.53 ID, or equivalent, with a 1.5 micron film thickness.

6.7 Data collection system, Perkin-Elmer/Nelson Series Turbochrom 4 Series 900 Interface, or equivalent.

6.8 Pipet, automatic dispensing, 50-ml capacity, and 2-liter reservoir.

6.9 Flasks, volumetric, class A, 100-ml and 1000-ml capacity.

6.10 Pipet, volumetric delivery, 10-ml capacity, class A.

7.0 Chemicals and Reagents

CHEMICALS:

7.1 Styrene, C_8H_8, 99 + %, CAS 100-42-5

7.2 Alpha methyl styrene, C_9H_{10}, 99%, CAS 98-83-9

7.3 Ethyl alcohol, C_2H_5OH, denatured formula 2B, CAS 64-17-5

REAGENTS:

7.4 Internal Standard Stock Solution: 5.0 mg/ml AMS in ethyl alcohol.

7.4.1 Into a 100-ml volumetric flask, weigh 0.50 g of AMS to the nearest 0.1 mg.

7.4.2 Dilute to the mark with ethyl alcohol. This solution will contain 5.0 mg/ml AMS in ethyl alcohol and will be labeled the AMS STOCK SOLUTION.

7.5 Internal Standard Working Solution: 2500 ug/50 ml of AMS in ethyl alcohol.

7.5.1 Using a 10 ml volumetric pipet, quantitatively transfer 10.0 ml of the AMS STOCK SOLUTION into a 1000-ml volumetric flask.

7.5.2 Dilute to the mark with ethyl alcohol. This solution will contain 2500 ug/50ml of AMS in ethyl alcohol and will be labeled the AMS WORKING SOLUTION.

7.5.3 Transfer the AMS WORKING SOLUTION to the automatic dispensing pipet reservoir.

7.6 Styrene Stock Solution: 5.0 mg/ml styrene in ethyl alcohol.

7.6.1 Into a 100-ml volumetric flask, weigh 0.50 g of styrene to the nearest 0.1 mg.

7.6.2 Dilute to the mark with ethyl alcohol. This solution will contain 5.0 mg/ml styrene in ethyl alcohol and will be labeled the STYRENE STOCK SOLUTION.

7.7 Styrene Working Solution: 5000 ug/10 ml of styrene in ethyl alcohol.

7.7.1 Using a 10-ml volumetric pipet, quantitatively transfer 10.0 ml of the STYRENE STOCK SOLUTION into a 100-ml volumetric flask.

7.7.2 Dilute to the mark with ethyl alcohol. This solution will contain 5000 ug/10 ml of styrene in ethyl alcohol and will be labeled the STYRENE WORKING SOLUTION.

8.0 Sample Collection, Preservation and Storage

8.1 Label a 2-oz sample poly-cap lid with the identity, date and time of the sample to be obtained.

8.2 At the sample location, open sample valve for at least 15 seconds to ensure that the sampling pipe has been properly flushed with fresh sample.

8.3 Fill the sample jar to the top (no headspace) with sample, then cap it tightly.

8.4 Deliver sample to the Laboratory for testing within one hour of sampling.

8.5 Laboratory testing will be done within two hours of the sampling time.

8.6 No special storage conditions are required unless the storage time exceeds 2 hours in which case refrigeration of the sample is recommended.

9.0 Quality Control

9.1 For each sample type, 12 samples of SBR latex shall be obtained from the process for the recovery study. Half the vials and caps shall be tared, labeled "spiked", and numbered 1 through 6. The other vials are labeled "unspiked" and need not be tared, but are also numbered 1 through 6.

9.2 The six vials labeled "spiked" shall be spiked with an amount of styrene to approximate 50% of the solution's expected residual styrene level.

9.3 The spiked samples shall be shaken for several hours and allowed to cool to room temperature before analysis.

9.4 The six samples of unspiked solution shall be coagulated and a mean styrene value shall be determined, along with the standard deviation, and the percent relative standard deviation.

9.5 The six samples of the spiked solution shall be coagulated and the results of the analyses shall be determined using the following equations:

$M_r = M_s - M_u$

$R = M_r/S$

where:

M_u = Mean value of styrene in the unspiked sample

M_s = Measured amount of styrene in the spiked sample

M_r = Measured amount of the spiked compound

S = Amount of styrene added to the spiked sample

R = Fraction of spiked styrene recovered

9.6 A value of R between 0.70 and 1.30 is acceptable.

9.7 R is used to correct all reported results for each compound by dividing the measured results of each compound by the R for that compound for the same sample type.

10.0 Calibration

10.1 Using a 10-ml volumetric pipet, quantitatively transfer 10.0 ml of the STYRENE WORKING SOLUTION (section 7.7.2 of this method) into a 2-oz bottle.

10.2 Using the AMS WORKING SOLUTION equipped with the automatic dispensing pipet (section 7.5.3 of this method), transfer 50.0 ml of the internal standard solution into the 2-oz bottle.

10.3 Cap the 2-oz bottle and swirl. This is the calibration standard, which contains 5000 µg of styrene and 2500 µg of AMS.

10.4 Using the conditions prescribed (section 6.5 of this method), chromatograph 1 µl of the calibration standard.

10.5 Obtain the peak areas and calculate the relative response factor as described in the calculations section (section 12.1 of this method).

11.0 Procedure

11.1 Into a tared 2-oz bottle, weigh 10.0 g of latex to the nearest 0.1 g.

11.2 Using the AMS WORKING SOLUTION equipped with the automatic dispensing pipet (section 7.5.3 of this method), transfer 50.0 ml of the internal standard solution into the 2-oz bottle.

11.3 Cap the bottle. Using a mechanical shaker, shake the bottle for at least one minute or until coagulation of the latex is complete as indicated by a clear solvent.

11.4 Using the conditions prescribed (section 6.5 of this method), chromatograph 1 ul of the liquor.

11.5 Obtain the peak areas and calculate the concentration of styrene in the latex as described in the calculations section (Section 12.2 of this method).

12.0 Calculations

12.1 Calibration:

$$RF = (W_x \times A_{is}) / (W_{is} \times A_x)$$

where:

RF = the relative response factor for styrene

W_x = the weight (ug) of styrene

A_{is} = the area of AMS

W_{is} = the weight (ug) of AMS

A_x = the area of styrene

12.2 Procedure:

$$ppm_{styrene} = (A_x\, RF \times W_{is}) / (A_{is} \times W_s)$$

where:

$ppm_{styrene}$ = parts per million of styrene in the latex

A_x = the area of styrene

RF = the response factor for styrene

W_{is} = the weight (ug) of AMS

A_{is} = the area of AMS

W_s = the weight (g) of the latex sample

12.3 Correct for recovery (R) as determined by section 9.0 of this method.

13.0 Precision

13.1 Precision for the method was determined at the 80, 144, 590, and 1160 ppm levels. The standard deviations were 0.8, 1.5, 5 and 9 ppm respectively. The percent relative standard deviations (%RSD) were 1% or less at all levels. Five degrees of freedom were used for all precision data except at the 80 ppm level, where nine degrees of freedom were used. Note: These are example results and do not replace quality assurance procedures in this method.

14.0 Pollution Prevention

14.1 Waste generation should be minimized where possible. Sample size should be an amount necessary to adequately run the analysis.

15.0 Waste Management

15.1 Discard liquid chemical waste into the chemical waste drum.

15.2 Discard latex sample waste into the latex waste drum.

15.3 Discard polymer waste into the polymer waste container.

16.0 References

16.1 This method is based on Goodyear Chemical Division Test Method E-889.