METHOD 39

REF: Reg 8-50

DETERMINATION OF STYRENE MONOMER CONTENT OF POLYESTER RESIN MATERIAL

1) PRINCIPLE

1.1 This method is applicable to the determination of styrene or other monomers in polyester resin materials.

1.2 The polyester resin is dissolved in dimethylsulfoxide **(DMSO)** and the concentration of styrene in the solution is determined by gas chromatography.

1.3 The detection limit of this method is 0.50% **(W/W)** styrene in the sample. Other types of monomer would have detection limits similar to styrene.

1.4 In the event that other types of resin material are used, then replace the term **"styrene"** with the appropriate name of the monomer in this method.

2) APPARATUS

2.1 **Gas Chromatograph.** This unit is fitted with a flame ionization detector **(FID)**, a glass sleeve injection port, a temperature programmer and a compatible integrator or data station. Operating parameters are as follows:

		Initial	Final
Oven Temperature (°C)	**150**	**200**	
Iso Time (min)	**15.0**	**15.0**	
Program Rate (°C/min)	**30.0**		
Injector Temperature (°C)	**250**		
Detector Temperature (°C)	**250**		
Carrier Gas	**He or N**		
Carrier Gas Flow, (cc/min)	**20.0**		
Injection Sample Size, µl	**2.0**		

2.2 Analytical Column.

2.2.1 15' x 1/8" OD SS Column packed with 10% Carbowax 20 M in Chrom W HP, 100/120 Mesh.

2.3 10 µl Syringe.

2.4 Centrifuge Tube. 15 ml, graduated in 0.1 ml subdivision, with screwcap.

2.5 Metal quart can. Wide mouth with cover.

2.6 Pipets. Various sizes as needed.

2.7 Refrigerator.

2.8 Volumetric Flasks, 10 ml.

2.9 Spatula.

2.10 Analytical Balance. Capable of weighing to ± 0.0001 g.

3) REAGENTS

Reagents must be ACS analytical reagent quality.

3.1 Dimethylsulfoxide (DMSO), or any appropriate solvent. Reagent Grade.

3.2 Styrene or Monomer of Interest. Reagent grade or the best available grade. A minimum purity of 99% is acceptable

3.3 Cylinder Hydrogen.

3.4 Cylinder Helium or Nitrogen.

3.5 Compressed Air.

4) ANALYTICAL PROCEDURE

4.1 The sample is collected in a metal quart can, and must be refrigerated immediately after it is received in the laboratory.

4.1.1 Take the sample out of the refrigerator at least 30 minutes prior to analysis. This will allow the sample to reach equilibrium at room temperature.

4.1.2 Open the can and mix the sample thoroughly using a spatula. It is essential that the sample be well mixed to obtain valid results.

4.1.3 Preweigh a 15 ml graduated centrifuge tube. As rapidly as possible, weigh accurately approximately 0.8 to 1 g **(± 0.0001 g)** of the mixed sample in the tared 15 ml graduated centrifuge tube. Record the net weight of the sample.

4.1.4 Using a pipet, add 10.0 ml of DMSO **(3.1)** to the sample in the centrifuge tube. Record the total volume of solution in the centrifuge tube. Cap and invert the tube several times until the polyester resin is completely dissolved in the DMSO.

4.1.5 Allow the sample **(4.1.4)** to reach equilibrium by letting it stand for 10 to 15 minutes undisturbed.

4.1.6 Inject 2 µl of the DMSO solution into a gas chromatograph setup with the parameters described in Section **2.1**.

4.1.7 Record the retention time and the peak area of the styrene found in the chromatogram. Duplicate analysis should agree to within ± 5% of the mean.

4.1.8 Samples with concentrations more than three **(3)** times the standard must be diluted with DMSO. Record the dilution factor **(DF)** required to bring the sample into the working range of the standard.

5) STANDARDIZATION

5.1 Accurately weigh approximately 300 mg **(± 0.1 mg)** of styrene in a 10 ml volumetric flask. Add DMSO to the mark. Stopper the flask and mix the solution well by inverting the flask several times. This solution contains approximately 30 mg styrene per ml of DMSO. This standard must be prepared fresh prior to use.

5.2 The DMSO used for sample extraction and standard preparation must be checked for contamination. Inject 2 µl of the DMSO blank into the gas chromatograph as in **Section 4.1.6**. Record the retention times and peak areas of the compounds, if any are present. Retain the chromatogram.

5.3 Set the gas chromatograph as described in **Section 2.1**.

5.3.1 Inject 2 µl of the standard solution **(5.1)** into the gas chromatograph using a 10 µl syringe. Record the retention time and peak area of styrene. Retain the chromatogram.

6) CALCULATION FOR COMPLIANCE

6.1 Compare the chromatograms of **(5.3.1)** and **(4.1.7)** to confirm the identity of styrene. Quantitate the concentration of styrene in the sample using the following equations:

6.1.1 $$\text{\% Styrene (W/W)} = \frac{(\text{Conc})_{std} \times (PA)_p \times (4.1.4) \times DF \times 100}{(PA)_{std} \times W_s}$$

Where:

$(PA)_p$ = Peak area of the styrene found in the DMSO solution (4.1.7).

$(\text{Conc})_{std}$ = Concentration in mg/ml of styrene in the standard (5.1).

DF = Dilution factor, where no dilution is made, DF = 1

$(PA)_{std}$ = Peak area of styrene in the standard (5.3.1).

W_s = Weight of sample in mg

7) REFERENCES

7.1 **"Control Techniques for VOC Emissions from Stationary Sources"**, EPA 450/7-78-023, May 1978.

7.2 **"Boiling Range Distribution of Petroleum Fractions by Gas Chromatography"**, ASTM Designation D 2887-84, Annual Book of ASTM standards, Vol 05.02, 1986.

7.3 **"Standard Test Method for Determining Unreacted Monomer Content of Latexes Using Gas-Liquid Chromatography"**, ASTM Designation D 4747-87, Book of ASTM Standards, Vol 06.02, 1992.

7.4 **"Standard Test Method for Determining the Unreacted Monomer Content of Latexes Using Capillary Column Gas Chromatography"**, ASTM Designation D 4827-88, Book of ASTM Standards, Vol 06.02, 1990.