SCAQMD METHOD 312-91

DETERMINATION OF PERCENT MONOMER IN POLYESTER RESINS

1. Principle

1.1 An aliquot of the sample is dissolved in an appropriate solvent, (e.g. o-xylene) and is analyzed by gas chromatography (GC) using a thermal conductivity detector.

1.2 The percentage monomer in the sample is determined by linear regression analysis of the areas of the standards followed by calculation using areas of diluted samples.

1.3 The monomer(s) present in the resin must be known in order that appropriate monomer standard(s) may be selected.

2. Equipment

2.1 Gas chromatograph of standard manufacture equipped with a thermal conductivity detector and liquid injection system

2.2 Integrator or computer capable of calculating and reporting peak area data

2.3 Calculator or computer capable of performing linear regression analysis

2.4 Column: DB^{R} Wax, 30 m X 0.53 mm, fused silica

2.5 Balance, analytical, accurate to 0.1 mg

2.6 Flasks, volumetric, Class A, 50 mL

2.7 Syringe, 5 mL, gas-tight, graduated in 0.1 mL

2.8 Pipettes, Pasteur, 9-inch, borosilicate glass

3. Reagents

3.1 Styrene, or appropriate monomer standard, 98+% purity

3.2 o-xylene, reagent grade

3.3 Other appropriate solvents, reagent grade

3.4 Helium, 99 mole % purity

4. Procedure

4.1 Preparation of Standards

4.1.1 Prepare standards of the appropriate monomer in o-xylene to nominal concentrations of 1.0% (w/w), 0.2% (w/w) and 0.1% (w/w). The concentrations of the standard should bracket the concentration of the sample.

4.1.1.1 Record the weight to nearest 0.1 mg of a 50 mL volumetric flask (W_1).

4.1.1.2 Using a 5 mL gas-tight syringe add monomer to 50 mL volumetric flask, stopper and record weight to nearest 0.1 mg (W_2).

4.1.1.3 Add o-xylene to the mark and record the weight to the nearest 0.1 mg (W_3).

4.2 Sample preparation

4.2.1 Prepare two concentrations of sample in o-xylene such that there is 0.25 g of sample in one preparation and 1.0 g of sample in the other.

4.2.1.1 Follow sections 4.1.1.1 through 4.1.1.3 except transfer of sample (in place of the monomer) is performed with a Pasteur pipette (instead of a gas-tight syringe).

4.3 Analysis

4.3.1 A blank (o-xylene), the standards and the samples are analyzed in duplicate. The standards are preceeded by a blank and are run in increasing concentrations before and after the samples.

4.3.1.1 The areas for each duplicate injection should be within 10% of the average area.

4.3.1.2 The average area at each concentration of standard run before and after the sample should be within 10% of each other.

4.3.2 Analyze no more than five samples per set of standards.

4.3.3 Refer to typical GC and integrator parameters in Appendix I.

4.3.4 Ideally, sample areas should fall within the range of the standard areas.

5. Calculations

5.1 Calculate the weight percent monomer for each of the standard preparations.

5.1.1 Weight percent standard (Wpst) $= \frac{Wstm}{Wst} \times 100$

Where:

Wstm (Weight of standard monomer) $= W_2 - W_1$
Wst (Weight of standard preparation) $= W_3 - W_1$

5.2 Generate the average area for each dilution of standard and sample.

5.3 Perform linear regression analysis for standard concentration versus average area.

5.3.1 Generate slope and y-intercept values using a calculator equipped with linear regression function capability.

5.3.2 Force the linear regression function through zero.

5.4 Calculate the prepared concentrations of monomer, (Ws), for each dilution of sample from area response.

5.5 Calculate weight percent monomer in the sample for each dilution by:

$$\text{Monomer, \% (w/w)} = \frac{Ws \ X \ Wp}{Sp}$$

Where Ws = Percent monomer analyzed in the preparation (value obtained by linear regression)
Wp = Total weight, in grams of the preparation
Sp = Weight in grams, of sample added to the preparation

5.6 Generate the average weight percent monomer from the weight percent monomer obtained from the two preparations. This value is reported as the weight percent monomer for the sample.

Appendix I
Typical Analysis Conditions*

Instrument Parameters

Column flow rate:	16 mL/min
Reference flow rate:	24 mL/min
Injection Temp:	200^{O}C
Detector Temp:	190^{O}C
Injection volume:	1 uL

GC Oven Program

70^{O}C isothermal

Integrator Parameters

Run time:	9 min
Attenuation:	3
Threshold:	2

*These are the optimum conditions for the analysis of styrene. Modifications to these parameters may be necessary to address the requirements of other monomers.

SOUTH COAST AIR QUALITY MANAGEMENT DISTRICT

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This method describes a gas chromatography method for the analysis of monomer content in polyester resins. It is applicable to Rule 1162.

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