

SOUTH COAST AIR QUALITY MANAGEMENT DISTRICT

APPLIED SCIENCE & TECHNOLOGY DIVISION

LABORATORY SERVICES BRANCH

SCAQMD METHOD 312-91

DETERMINATION OF PERCENT MONOMER IN POLYESTER RESINS

Approved June 1, 1991
Revised June 1993
Revised April 1996

SCAQMD METHOD 312-91**DETERMINATION OF PERCENT MONOMER IN POLYESTER RESINS**

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

CONTENTS

	<u>Page</u>
1. Principle	1
2. Equipment	1
3. Reagents	1
4. Procedure	2
5. Calculations	3
6. Appendix I: Typical Analysis Conditions	5

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 preceded 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.

$$5.1.1 \quad \text{Weight percent standard (Wpst)} = \frac{W_{stm}}{W_{st}} \times 100$$

Where:

$$\begin{aligned} W_{stm} \text{ (Weight of standard monomer)} &= W_2 - W_1 \\ W_{st} \text{ (Weight of standard preparation)} &= W_3 - W_1 \end{aligned}$$

- 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:

$$\text{Monomer, \% (w/w)} = \frac{W_s \times W_p}{S_p}$$

Where W_s = Percent monomer analyzed in the preparation (value obtained by linear regression)

W_p = Total weight, in grams of the preparation

S_p = 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°C

Detector Temp: 190°C

Injection volume: 1 uL

GC Oven Program

70°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.