#### SCAQMD METHOD 309-91

#### DETERMINATION OF STATIC VOLATILE EMISSIONS

#### 1. Principle

1.1 A sample of resin is weighed onto a metal gallon can lid and allowed to stand until the sample has gelled. The weight may be determined at intervals to obtain the rate of weight loss. Volatile emissions are calculated as grams per square meter.

#### 2. Apparatus

- 2.1 Balance, top loading, accurate to 0.01 gram
- 2.2 Gallon can lid, metal, 145 mm diameter with a side rim of 8 mm high
- 2.3 Ruler, graduated in 1 mm
- 2.4 Thermometer, accurate to 1<sup>o</sup>F
- 2.5 Paper Clip, bent at 90-degree angle (See Figure 1.)
- 2.6 Timer, accurate to 0.1 minute
- 2.7 Beaker, glass, 400 mL
- 2.8 Stirrer, glass
- 2.9 Hood, "draft-free", lab bench top or equivalent

#### 3. Procedure

- 3.1 Analysis of Catalyzed Resins
  - 3.1.1 Measure the inside diameter of a gallon-can lid to the nearest millimeter (nominally 145 mm).
  - 3.1.2 Wash the inside surface with acetone and dry thoroughly.
  - 3.1.3 Place a balance in a draft-free hood.

- 3.1.4 Measure the room temperature. It must be 65-75°F.
- 3.1.5 Record the tare weight of a 400 mL beaker.
  - 3.1.5.1 Calculate the amount of catalyst, promoter, and resin to be used by following the given mix ratio to make 200 g of sample.
- 3.1.6 Place paper clip on the gallon can lid and tare to 0.01 gram. Record tare weight, (WT).
- 3.1.7 Immediately add to the gallon can lid 100 grams of catalyzed resin and record the initial weight, (WI), to 0.01 gram. Sample weight must be 100  $\pm 0.5$ g net weight.
- 3.1.8 Start the timer.
- 3.1.9 For percent volatile emissions.
  - 3.1.9.1 Check catalyzed resin for gel end point by lifting paper clip.
  - 3.1.9.2 Gel time occurs when, by lifting the paper clip, the resin tears, or the gallon-can lid lifts from the balance.
  - 3.1.9.3 Gel time is recorded to the nearest minute.
  - 3.1.9.4 Record the weight at one hour after gel time and at 15 minute intervals thereafter until constant weight is obtained. The final weight for use in calculations is designated WF.
- 3.2 Analysis of Uncatalyzed Resins
  - 3.2.1 Follow Sections 3.1.1, 3.1.2, 3.1.3, and 3.1.4.
  - 3.2.2 Tare gallon-can lid to 0.01 gram after it has been washed in acetone and thoroughly dried. Record tare weight, (WT).
  - 3.2.3 Add 100 grams of resin to the can lid and record the initial weight, (WI). Resin weight must be  $100 \pm 0.5$ g.

3.2.4 Record the weight at 5 minute intervals for a total of 30 minutes for rate of weight loss. Final weight, (WF), at 30 minutes is recorded.

#### 4. Calculations

4.1 Percent volatile emission by weight is calculated by the following:

% Volatile emission 
$$=\frac{WI - WF}{WI - WT} X$$
 100

4.2 Lid factor (LF) is calculated by the following:

LF =  $\frac{10000}{\pi r^2}$ (10,000 is the number of square cm in 1 square meter)

4.3 Volatile emissions, (VE), in grams per sq. meter is calculated by the following:

VE,  $g/m^2 = (WI - WF) X LF$ 

- 4.4 Definition of terms used in calculation
  - $\begin{array}{rcl} WI &=& \text{Initial weight, g (Sec. 3.1.7 or 3.2.3)} \\ WF &=& \text{Final weight, g (Sec. 3.1.9.4 or 3.2.4)} \\ WT &=& \text{Tare weight, g (Sec. 3.1.6 or 3.2.2)} \\ r &=& \text{The diameter of gallon-can lid divided by 2, cm (Sec. 3.1.1)} \\ LF &=& \text{Lid factor (Sec. 4.2)} \end{array}$

### 5. **Report Parameters**

- 5.1 Information to be included in the report
  - 5.1.1 Resin and promoter/catalyst system
  - 5.1.2 Gel time (in minutes), and temperature (in <sup>o</sup>F)
  - 5.1.3 Volatile Emission (in percent by weight)
  - 5.1.4 Weight loss from VOC emission (in grams per square meter)

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This method is applicable to samples regulated under Rule 1162.

This test procedure determines the volatile emission of a sample during polymerization. It is susceptible to potential errors resulting from changes in film thickness, temperature of conditions used, and amount of air circulation.

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