

METHOD 23

REF: Reg 8-50

DETERMINATION OF VOLATILE EMISSIONS FROM POLYESTER RESINS

1) PRINCIPLE

- 1.1 A pour pan method is used, where a weighed amount of polyester resin is catalyzed with peroxide initiators under controlled conditions of $77^{\circ} \pm 3^{\circ}\text{F}$ and 50% relative humidity (**% RH**). If a controlled environment is not available, conditions under which the measurements are made must be reported.
- 1.2 The weight loss of a one gallon can lid filled with 100 g of catalyzed resin is accurately measured over a period of time. The weight losses attributed to the monomer and other volatile emissions are to be reported in grams per square meter (**g/m²**).
- 1.3 This method is applicable only to polyester resins.

2) APPARATUS

- 2.1 **Analytical Balance, Top Loading.** Capable of weighing to ± 0.01 g.
- 2.2 **Gallon Paint Can Lid.** The lid should have an inside diameter of **approximately 14.5 cm** (Area = 165.13 square cm).
- 2.3 **Clock or Timer.** Capable of reporting time to a precision of 0.01 minute.
- 2.4 **Constant Temperature and Humidity Room,** as described in **Section 1.1**. The room must also be free of drafts. If a constant temperature room is not available, measure the ambient temperature and humidity prior to each weight measurement or test procedure.
- 2.5 **8 oz wide mouth bottles, with screw caps.**
- 2.6 **Stirring rod, glass or metal**
- 2.7 **Thermometer.** 20 to 120°F in 1° division.
- 2.8 **Hygrometer.** Capable of determining 1% RH.

2.9 Paper clip. Bent to approximately 90° angle.

2.10 Pipette. Various sizes as needed.

2.11 Syringe. Disposable, 3 ml capacity.

2.12 Constant Temperature Water Bath.

3) ANALYTICAL PROCEDURE

3.1 Measure the inside diameter (**d**) of the bottom part of the lid (**2.2**) to ± 0.1 centimeter (**normally, d = 14.5 cm**).

3.2 Clean the gallon can lid with acetone and wipe dry before using.

3.3 Place the balance in a constant temperature room free from drafts. If a constant temperature room is not available, place the balance on the lab bench in an area free of draft. Determine the temperature and humidity of the room before starting the test.

3.4 Place the bent paper clip (**2.9**) on the gallon can lid and weigh to ± 0.01 g. Record the weight.

3.5 Weigh accurately approximately 150 ± 0.05 g of the prepromoted resin into a dry and clean 8 oz wide mouth bottle with screw cap.

3.6 Cap the bottle containing the resin and place in a constant temperature water bath. Adjust the bath temperature to $77 \pm 3^\circ\text{F}$.

3.7 Place the tared gallon can lid with the bent paper clip on the balance.

3.8 Check the resin temperature in the bottle using a thermometer. Be sure it is $77^\circ \pm 3^\circ\text{F}$. Take the glass bottle containing the resin out of the water bath and using a pipette or syringe, add the appropriate amount and type of catalyst.

3.9 Mix the catalyst and the resin for one minute, using a spatula. Start the timer.

3.10 Pour 100 ± 0.5 g of the catalyzed resin into the tared can lid. Record this as the initial weight. The sample weight must be 100 ± 0.5 g net.

- 3.11 Use the bent paper clip to determine if the resin has hardened sufficiently to allow the resin or, resin and lid, to be lifted. Stop the timer. Record this as the gel time.
- 3.12 Allow the resin to cure completely in the can lid and continue weighing every 15 minutes until concurrent weighings agree to within ± 0.05 g. Record the weights. The last weight is the final weight.
- 3.13 For duplicate analysis, repeat **Section 3.1** through **Section 3.12**.
- 3.14 Duplicate sample analysis must agree to the nearest 5 g/m².

4) CALCULATION FOR COMPLIANCE

4.1 Volatile Emissions Per Square Meter (m²)

$$4.1.1 \quad \text{Area of Gal Can Lid in m}^2 = \frac{(D/2)^2}{10^4} \times 3.1416$$

Where: D = Diameter of the gal can lid in centimeters (cm)
 3.1416 = Value of Pi
 10⁴ = Sq cm in m².

$$4.1.2 \quad \text{Volatile Emissions in g/m}^2 = \frac{(3.10) - (3.12)}{(4.1.1)}$$

$$4.1.3 \quad \% \text{ Volatile Emission} = \frac{(3.10 - 3.12) \times 100}{(3.10) - (3.4)}$$

5) REPORTING REQUIREMENTS.

The following analytical data must be included in the report :

- 5.1 **Temperature (°F) and % Relative Humidity.**
- 5.2 **Resin Identification and Batch Number.**

- 5.3 Catalyst used.
- 5.4 Resin/Catalyst Ratio.
- 5.5 Volatile Emissions in g/m².
- 5.6 Percent Volatile Emission
- 5.7 Gel time under test conditions.

6) REFERENCES

- 6.1 "Static Method for Determination of Volatile Emissions from Polyester and Vinyl Ester Resins". Society of Plastic Industry Composites Institute Method.
- 6.2 "Standard Method for Static Volatile Emissions". South Coast Air Quality Management District.