

**SOUTH COAST AIR QUALITY MANAGEMENT DISTRICT
APPLIED SCIENCE AND TECHNOLOGY DIVISION**

LABORATORY SERVICES BRANCH

SCAQMD METHOD 316B

**DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOC)
IN ADHESIVES CONTAINING CYANOACRYLATES**

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This test method, developed by Loctite Corporation, is a modification of SCAQMD Method 316A. The modifications extend the applicability of SCAQMD Method 316A to adhesives containing cyanoacrylates. These products have been shown to satisfy the requirements of the SCAQMD definition of reactive diluent. This method applies to adhesives that do not contain water or exempt compounds.

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DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOC) IN ADHESIVES CONTAINING CYANOACRYLATES

1. Principle

A portion of the material is applied to an aluminum or other non-porous substrate. The specimen is cured at room temperature for 48 hours. After curing, the specimen is heated for one hour at 110°C. The amount of VOC emitted is determined by the weight of VOC lost during application, curing and heating. This value is then used to calculate the VOC in g/L.

This method is not applicable to cyanoacrylate adhesives containing water or exempt compounds.

2. Equipment

- 2.1 Laboratory exhaust hood
- 2.2 Balance, analytical, capable of weighing accurately to 0.1 mg
- 2.3 Oven, forced draft, capable of maintaining the temperature at 110°C for one hour; refer to ASTM D2369 for specifications
- 2.4 Desiccator

3. Reagents and Materials

- 3.1 Aluminum foil, heavy gauge (0.05 mm minimum), or other non-porous substrate, free of pinholes, cut into 6 inch squares, conditioned in an oven at 110°C for one hour and cooled in a desiccator prior to weighing
- 3.2 Aluminum plates, minimum 1/8" thickness, 6" x 6", deburred and polished;
cleaned and dried. Other flat, rigid, material may be used in place of aluminum.

- 3.3 Spring clamps, Hargrave No. 1, Pony No. 3201 or similar clamps capable of opening at least 3/4 inches.

4. Procedure

The adhesive to be tested must be in a container that will allow the application of the adhesive directly from the container to the substrate without the use of any application device.

- 4.1 Condition the unopened adhesive and container at room temperature for at least 2 hours. The weight of the adhesive and container must not exceed 50 grams.
- 4.2 Weigh a pair of substrates from Section 3.1 to the nearest 0.1 mg. Record the weight as W_i .
- 4.3 Using gloves or tongs, weigh the adhesive and container from Section 4.1 to the nearest 0.1 mg. Record the weight as A.
- 4.4 Place one of the weighed aluminum substrates on an aluminum plate. Apply approximately 0.5 g of adhesive directly from the weighed adhesive and container (Section 4.1) to the center of the substrate.
- 4.5 Affix the mate of the substrate on top of the adhesive and place another aluminum plate on top. Attach the two substrates together with 4 spring clamps in order to evenly distribute the adhesive on the surfaces of the substrates.
 - 4.5.1 Take precautions (when performing Section 4.5) to prevent the adhesive material from getting squeezed beyond the edges of the substrates.
- 4.6 Weigh the adhesive and container to the nearest 0.1 mg after dispensing the adhesive. Record this weight as B.
- 4.7 Calculate the weight of adhesive applied to the substrate (C) by the difference in adhesive and container weight before and after dispensing the adhesive. $C = (A-B)$.
- 4.8 Calculate the test specimen weight (W_1) by adding the weight of the substrate pair (W_i) and the weight of applied adhesive (C). $W_1 = (W_i + C)$.

- 4.9 Repeat steps 4.2 through 4.8 to prepare another test specimen.

- 4.10 Allow the assembled test specimens to cure at room temperature for 48 hours.
- 4.11 Carefully remove the spring clamps and aluminum plates from the assemblies.
- 4.12 Heat the cured test specimens in a forced draft oven at $110^{\circ} \pm 5^{\circ}\text{C}$ for one hour.
- 4.13 Cool the cured test specimens in a desiccator.
- 4.14 Reweigh each test specimen to 0.1 mg. Record the weight as D.
- 4.15 Determine the density (dm) of the sample using SCAQMD Method 304.

5. Calculations

- 5.1 Weight percent of VOC emitted (VOCe) for each run:

$$\text{VOCe} = (\text{W1} - \text{D}) \times 100 / \text{C}$$

- 5.2 Total VOC emitted, g/L:

$$\text{VOC} = \text{VOCe} \times \text{dm} \times 10$$

Where:

- A = weight of adhesive and container before dispensing adhesive, g
- B = weight of adhesive and container after dispensing adhesive, g
- C = (A-B) = weight of adhesive applied to the substrate, g
- D = weight of the test specimen after it has been oven treated and cooled in a desiccator, g
- dm = density of material, g/mL
- W1 = (Wi + C) = weight of the test specimen before heating, g
- Wi = weight of the substrate pair, g

6. Precision

- 6.1 The precision estimates are based on an interlaboratory study in which 1 operator in each of 6 laboratories analyzed in duplicate on 2 different days 3 samples of cyanoacrylate adhesives. The average VOC emission of adhesives tested in trials was 0.4 weight percent. The results were analyzed statistically in accordance with practice ASTM E 180.

- 6.2 Repeatability (Single Analyst) - The coefficient of variation of results (each the average of duplicate determinations), obtained by the same analyst on different days, was estimated to be 0.4 % absolute at 12 DF. The 95 % limit for the difference between two such averages is 1.1 % absolute.
- 6.3 Reproducibility (Multilaboratory) - The coefficient of variation of results (each the average of duplicate determinations), obtained by analysts in different laboratories, has been estimated to be 0.8 % absolute at 3 DF. The 95 % limit for the difference between two such averages is 2.2 % absolute.