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AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
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Standard Test Method for Volatile Organic Compound (VOC40) Content of Non-Heatset Paste Printing Ink Systems at 40°C[1]

This standard is issued under the fixed designation D 5328; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the volatile organic compound (VOC40) content of non-heatset printing ink systems of the paste type using a baking cycle of 1 h at 40°C.

1.2 This test method is applicable to paste printing inks and vehicles that dry primarily by absorption, polymerization, or related means without the application of heat. Such systems do not contain appreciable quantities of low-boiling solvents that evaporate readily at ordinary room temperatures.

NOTE 1—The 40°C baking temperature in this test method is the same as that specified in Bay Area Method 30. The temperature of heating is 110°C in several related test methods, for example, D 2369, D 4713 and EPA Reference Method 24. VOC determinations are reviewed in ASTM Manual Series MNL 4.[2]

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1475 Test Methods for Density of Paint, Varnish, Lacquer and Related Products[3]

D 2369 Test Methods for Volatile Content of Coatings[3]

D 3792 Test Method for Water Content of Water-Reducible Paints by Direct Injection into a Gas Chromatograph[3]

D 3960 Practice for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings[3]

D 4017 Test Method for Water in Paints and Paint Materials by Karl Fischer Method[3]

D 4713 Test Methods for Nonvolatile Content of Heatset and Liquid Printing Ink Systems[4]

E 1 Specification for ASTM Thermometers[5]

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens[6]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of Test Methods[6]

2.2 *Other Standards:*

EPA Federal Reference Method 24—Determination of Volatile Matter Content, Water Content, Density, Volume Solids, and Weight Solids of Surface Coatings[7]

Method 30—Determination of Volatile Organic Compounds (VOC) in Solvent-Based Non-Heatset Inks[8]

3. Terminology

3.1 *Definitions*—Definitions pertaining to this test method are covered in Practice D 3960.

4. Summary of Test Method

4.1 The test material is spread in a weighing dish to a nominal thickness corresponding to about 115 g/m^2. The weight percent of total volatile compounds is determined after heating at 40°C for one h.

4.2 The typical paste ink does not contain water or exempt solvents; in such cases, the volatile organic content is the same as the total volatile content. If the test sample is based on an emulsion system or is suspected of containing more than 5 % water, the water content is determined by the Karl Fischer method or by gas chromatography.

4.3 The density of the test sample is measured, and the volatile organic content in weight percent at 40°C is converted to units of grams per litre or pounds per gallon. Alternatively, if the water content was found to be greater than 5 %, the volatile organic content is calculated in accordance with Note 3.

5. Significance and Use

5.1 VOC content data are required by various regulatory agencies. This test method determines the volatile organic content of non-heatset paste printing ink systems at 40°C in accordance with Bay Area Method 30. This temperature is considered more realistic in terms of end use than the 110°C

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[1] This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.56 on Printing Inks.

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[2] Brezinski, J. J., *Manual on Determination of Volatile Organic Compounds in Paints, Inks, and Related Coatings, ASTM Manual Series MNL4*, ASTM, 1989.

[3] *Annual Book of ASTM Standards*, Vol 06.01.

[4] *Annual Book of ASTM Standards*, Vol 06.02.

[5] *Annual Book of ASTM Standards*, Vol 14.03.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

[7] Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

[8] Available from Bay Area Quality Measurement District (BAAQMD), 939 Ellis Street, San Francisco, CA 94109.

used in several similar test methods. Due to the complex chemical composition and boiling characteristics of the oils used in these inks, temperature of 110°C may cause volatile loss that would not occur at ambient temperatures, even over extended periods of time.

5.2 The inverse value, nonvolatile, is used to determine the weight percent solids content.

5.3 The specimen film thickness must be controlled in order to obtain reproducible test results within the specified 1-h heating time.

6. Apparatus

6.1 *Balance*, accurate to 1 mg.

6.2 *Oven*, forced-ventilation type conforming to Type IIB in Specification E 145, properly leveled and maintained at 40° ± 1°C (104 ± 2°F). If air flow is adjustable, set control dampers, at 50 %.

6.3 *Thermometer*, bulb-type, capable of reading to ±1°C, such as Thermometer 88C prescribed in Specification E 1.

6.4 *Weighing Dishes*, such as aluminum foil dishes 57 or 70-mm wide, the lid of 1-lb ink can 94-mm wide, or other flat-bottomed containers. The bottom of the containers must not have a trough or depression into which the test material might collect. Dishes should be rinsed with toluene or other appropriate solvent, preconditioned for 30 min in an oven at 110 ± 5°C, and stored in a desiccator prior to use.

6.5 *Spatula*, or small ink knife.

6.6 *Spreading Device*, one per weighing dish, of heat-stable material, such as a glass stirring rod or thick L-shaped wire.

6.7 *Forceps*.

6.8 *Desiccator*.

6.9 *Water Content Measuring Apparatus*, such as a Karl Fischer apparatus conforming to Test Method D 4017 or a gas chromatograph conforming to Test Method D 3792.

6.10 *Weight-Per-Gallon Cup*, 20 to 100-mL capacity calibrated in accordance with Test Methods D 1475.

7. Reagents

7.1 *Toluene*, technical grade, or other appropriate solvent.

8. Preparations and Sample

8.1 Wear disposable gloves prior to handling weighing dish, spreading device, or weight-per-gallon cup in order to minimize contamination by moisture from hands.

8.2 Measure diameter of bottom of weighing dish in millimetres. The bottom diameter must be used in the calculations of weight per unit area in 13.1.

8.3 Prior to removing a specimen, carefully remove surface skin, if any, and thoroughly mix the sample in its container to ensure uniformity. Close can and reseal when finished.

9. Procedure for Volatile Content

9.1 Set the oven for 40° ± 1°C.

9.2 For each sample, tare to the nearest milligram two preconditioned weighing dishes each with a spreading device. Retain spreading device in the dish throughout the test.

9.3 Transfer a representative portion (see 8.3) of the sample to the tip of a spatula and dab an appropriate quantity around the bottom of the dish as follows:

57-mm dish	0.20–0.25 g
70-mm dish	0.40–0.50 g
94-mm dish	0.70–0.90 g

Quickly reweigh and calculate the weight per area following 12.1. *If not between 100 and 130* g/m^2, adjust the quantity of the specimen.

9.4 With the spreader, smooth out the specimen into a reasonably uniform film covering the entire bottom of the dish. If necessary, add a few drops of toluene or other appropriate solvent to aid in spreading out the film.

9.5 Place the dishes in the forced-draft oven at 40°C for exactly 1 h. Remove dishes from oven, cool in desiccator, and reweigh.

10. Procedure for Water Content and Exempt Solvents

10.1 Paste inks are not water-reducible. If the sample is an emulsion or is believed to contain more than 5 % water, measure the water content in one of the following ways:

10.1.1 *Test Method D 4017*—Dilute the specimen with pyridine, add a catalyst (1-ethylpiperidine), titrate to the end point in the Karl Fischer apparatus, and compute the water content.

10.1.2 *Test Method D 3792*—Dilute the specimen with dimethyl formamide, add an internal standard (2-propanol), inject an aliquot directly into the gas chromatograph, and compute the water content from appropriate area calculations on the chromatogram.

10.2 Exempt solvents are generally too volatile to be used in paste ink systems. Therefore, there is no need to test for their presence. (A list of exempt solvents is covered in Practice D 3960.)

11. Procedure for Density

11.1 Determine the density of the sample at 25°C using a calibrated weight-per-gallon cup in accordance with Test Method D 1475.

11.2 Transfer the thoroughly mixed sample to the weighed cup in a manner so as to avoid occluding air bubbles. Fill to the top and cap, leaving the overflow orifice open. Immediately remove excess sample using an absorbent material. Quickly reweigh the filled cup.

11.3 Clean the cup and repeat 11.2.

12. Calculation

12.1 Calculate initial weight/area *S/A* in grams per square metre of each specimen as follows:

$$S/A = (S \times 10^6)/3.14\,R^2 \quad (1)$$

where:

S = initial specimen weight, g,

R = radius of the dish bottom, = diameter/2, mm.

Record to the nearest whole number.

NOTE 2—Weight per area equals 510 *S* for a 57-mm dish with a 50-mm diameter bottom, 260 *S* for a dish with a 70-mm diameter bottom, and 145 *S* for a dish with a 94-mm diameter bottom.

12.2 Calculate the weight percent nonvolatile content at 40°C *NVC*40 as follows:

$$NVC40 = (W_1/S) \times 100 \quad (2)$$

where:
W_1 = weight of the specimen after heating at 40°C, g and
S = initial specimen weight, g.

Record to the nearest 0.1 %.

12.3 Calculate the weight percent volatile organic content *VOC*40 as follows:

$$VOC40 = 100 - NVC40 \quad (3)$$

12.4 Compute the density of the sample, D_m in grams per millilitre as follows:

$$D_m = W_2/V \quad (4)$$

where:
W_2 = specimen weight in the weight-per-gallon cup, g, and
V = volume of the cup, mL.

Record to three decimal places.

12.5 Compute the *VOC*40 content in grams per litre or pounds per gallon as follows:

12.5.1 Grams per litre:

$$VOC40 = D_m\, VOC40 \times 10 \quad (5)$$

12.5.2 Pounds per gallon:

$$VOC40 = 8.3454\, D_m\, VOC40 \times 10^{-2} \quad (6)$$

where:
8.3454 = factor converting g/mL to lb/gal (see Test Method D 1475).

Record to two decimal places.

NOTE 3—If the sample contains more than 5 % water, compute the $VOC40_{nw}$ in grams per litre as follows:

$$VOC40_{nw} = \frac{D_m\,(VOC40 - W_w)}{100 - (W_w)(D_m/D_w)} \times 1000 \quad (7)$$

where:
$VOC40_{nw}$ = VOC40, ink minus water, g/L,
$VOC40$ = (100 − NVC), weight %,
W_w = water content, weight %, and
D_w = density of water, at 25°C (0.997), g/L.

Multiply $VOC40_{nw}$ in grams per litre by 8.3454 to obtain $VOC40_{nw}$ in pounds per gallon.

13. Report

13.1 Report the following:

13.1.1 The weight/area of the sample,

13.1.2 The temperature and time of heating,

13.1.3 The total nonvolatile content at 40°C (*NVC*40) in weight percent as the mean of two determinations,

13.1.4 The volatile organic content at 40°C (*VOC*40) in weight percent,

13.1.5 The density of the sample in g/mL as the mean of two determinations, and

13.1.6 The *VOC*40 content in g/L or lb/gal.

NOTE 4—For those samples that contain more than 5 % water, report the water content in weight percent as the mean of two determinations and the volatile organic content at 40°C in accordance with Eq 5 of Practice D 3960.

14. Precision and Bias

14.1 *Precision of VOC40 in Weight Percent*[9]—An interlaboratory study of this test method was conducted in which operators in 12 laboratories tested the weight percent volatile content in duplicate on each of two days of four non-heatset wet-offset black inks whose *VOC*40 ranged from 0.5 to 8 % by weight (92 to 99.5 % expressed as nonvolatile content). Since the inks did not contain water, the volatile organic content was the same as the total volatile content. The test results were analyzed in accordance with Practice E 691. Two laboratories did not conform to the prescribed film thickness range and were deleted from the analysis, as was another laboratory because of a faulty oven. The ink with the 0.5 % by weight *VOC*40 showed more variability (less when expressed as *NVC*40) than the other three inks and was also deleted from the analysis. For the inks with a *VOC*40 content between 1 and 8 % by weight, the within-laboratory pooled standard deviation was 0.53, and the between-laboratories pooled standard deviation was 0.90. Based on statistical analysis of the results, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

14.1.1 *Repeatability*—Two *VOC*40 weight percent results, each the mean of two determinations obtained by the same operator on different days, should be considered suspect if they differ by more than 36 % relative (1.4 % relative if expressed as *NVC*40).

14.1.2 *Reproducibility*—Two *VOC*40 weight percent results, each the mean of determinations obtained on different days by operators in different laboratories, should be considered suspect if they differ by more than 60 % relative (2.3 % relative if expressed as *NVC*40).

14.2 *Precision of VOC40 in g/L or lb/gal*—A study has been initiated to determine the precision of weight-per-gallon density measurements on paste inks. If less than that given in Test Method D 1475, a new round robin will be conducted to determine the precision of *VOC*40 in g/L or lb/gal.

14.3 *Bias*—Bias cannot be determined as there are no standard materials.

15. Keywords

15.1 non-heatset paste printing inks; non-heatset paste printing ink vehicles; nonvolatile content; printing inks; vehicles; volatile organic compound (VOC40) content at 40°C

[9] Supporting data are available from ASTM Headquarters. Request RR:D01-1078.