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Test Method for Volatile Content of Non-Heatset Web Offset Printing Inks¹

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1. Scope

1.1 This test method describes a procedure for determination of the weight percent volatile content of non-heatset web offset printing inks. Test specimens are heated at $110^{\circ} \pm 1^{\circ}\text{C}$ for 60 min.

1.2 This test method is also applicable to non-heatset web offset printing ink vehicles. It is not applicable to ultra-violet (UV) or electron beam cured materials, which must be cured by exposure to UV light or an electron beam as part of the test for volatile content.

NOTE 1—Vehicle is the liquid portion of the printing ink. Any substance that is dissolved in the liquid portion of the ink is a part of the vehicle.

1.3 This test method is based on Test Method D 2369, in which the allowable ranges are $\pm 0.1\text{g}$ for specimen weight and $\pm 5^{\circ}\text{C}$ for oven temperature. Interlaboratory studies have shown that specimen weight and oven temperature must both be more tightly controlled in order to improve the precision of test results for non-heatset web-offset inks. Such inks contain a wide range of high boiling hydrocarbons and often have a volatile content below 20 %.

1.4 *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. For a specific hazard statement see Note 5.*

2. Referenced Documents

2.1 ASTM Standards:

D 2369 Test Method for Volatile Content of Coatings²

D 362 Specification for Industrial Grade Toluene³

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens⁴

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁴

2.2 Other Standards:

EPA Reference Method 24 - Determination of Volatile Matter Content, Density, Volume Solids, and Weight Solids of Surface Coatings⁵

3. Summary of Test Method

3.1 A specimen size of $0.300 \pm 0.001\text{ g}$ is weighed into an aluminum foil dish, dispersed in 3 mL of toluene, and heated in an oven at $110 \pm 1^{\circ}\text{C}$ for 60 min. The percent volatile is calculated from the loss in weight.

4. Significance and Use

4.1 This test method is the procedure of choice for determining volatile content of non-heatset web offset inks. This information is useful to the ink manufacturer and user and to environmental interests as part of the determination of the mass of volatile organic compounds emitted from the ink.

NOTE 2—Since these inks do not contain water or any materials currently classified by US EPA as negligibly photochemically reactive (exempt solvents), volatile organic compound content is the same as volatile content. The volatile organic compounds in these inks are high boiling hydrocarbon oils which are, according to US EPA guidelines, 95 % retained in the printed substrate or oxidized into the ink film. Therefore, the mass of volatile organic compound emitted from the ink would be calculated as only 5 % of the volatile organic compound content of the ink as derived from the results of this test method.

5. Apparatus

5.1 *Aluminum Foil Dishes*, 58 mm in diameter by 18 mm high with a smooth (planar) bottom surface. Precondition the dishes for 30 min. in an oven at $110 \pm 1^{\circ}\text{C}$ and store in a desiccator prior to use. Use tongs or rubber gloves or both, to handle the dishes.

5.2 *Desiccator*, equipped with a general purpose desiccant such as anhydrous calcium sulfate.

5.3 *Forced Draft Oven*, Type IIA or Type IIB as specified in Specification E 145.

NOTE 3—Be sure the shelves are level.

5.4 *Ink Knives or Taper Knives*, suitable for transferring the small specimen from the original sample container to the aluminum dish.

5.5 *Paper Clips*, bent for use as stirrers. Precondition the bent paper clips along with the aluminum foil dishes, as described in 5.1.

5.6 *Analytical Balance*, capable of weighing to 0.0001 g.

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 *Annual Book of ASTM Standards*, Vol 06.01.

³ *Annual Book of ASTM Standards*, Vol 06.03.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

5.7 *Stopwatch*, capable of reading to 1s.

6. Reagents

6.1 *Toluene*, technical grade, in accordance with Specification D 362.

7. Procedure

7.1 Weigh a preconditioned foil dish that has been equipped with a preconditioned bent paper clip to 0.0001g and record as W_1 . Similarly weigh a duplicate.

7.2 Mix the sample by hand using an ink knife (taper knife) prior to extracting the exact specimens to be used in the test.

7.3 Weigh a $0.300\text{ g} \pm 0.001\text{ g}$ specimen directly into each of the weighed dishes. Record the specimen weight to 0.0001g as S .

NOTE 4—Due to the relatively high viscosity and low volatility of non-heatset web offset inks, this may best be achieved using ink knives (taper knives). The quickest way to achieve this specimen size and precision is to slightly overweigh and then remove tiny amounts using the tip of a clean ink knife.

7.4 To each dish add 1 mL of toluene and stir, using the bent paper clip, into the ink specimen. Then add a further 2 mL of toluene (for a total of 3 mL) and repeat the stirring to achieve a homogeneous dispersion.

7.5 Heat the aluminum foil dishes containing the dispersed specimens in the forced draft oven (5.2) for exactly 60 min. at $110 \pm 1^\circ\text{C}$. The bent paper clips should remain in the dishes throughout the test. Position the dishes as near to the center of the oven as possible. Use the stopwatch to time the 60-min oven exposure.

NOTE 5—**Precaution:** In addition to other precautions, provide adequate ventilation, consistent with accepted laboratory practice, to prevent solvent vapors from accumulating to a dangerous level.

7.6 Remove the dishes from the oven, place immediately in a desiccator, cool to ambient temperature, and weigh to 0.0001g. Record weight as W_2 .

8. Calculation

8.1 For each specimen, calculate the weight percent volatile matter V , in the non-heatset web offset ink as follows:

$$V = \frac{(W_1 + S) - W_2}{S} \times 100 \quad (1)$$

where:

W_1 = weight of dish plus paper clip,

W_2 = weight of dish plus paper clip, plus specimen after heating, and

S = specimen weight prior to heating.

Average the duplicate determinations and report the average as the weight percent volatile matter.

9. Precision and Bias

9.1 *Precision*—An interlaboratory study of this test method was conducted in which operators in 10 laboratories tested in triplicate 5 non-heatset web offset black inks whose volatile content ranged from 3 to 39 %. The test results were analyzed in accordance with Practice E 691. In the same interlaboratory study, the full range of temperature and sample sizes permitted by Test Method D 2369 was also studied. The precision, characterized by repeatability (S_r , r) and reproducibility (S_R , R) was much better for this specific test method. Repeatability was improved by a factor of 4.2 and reproducibility was improved by a factor of 1.3. Based on the statistical analysis of the results, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

9.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator should be considered suspect if they differ by more than 13 % relative.

9.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 54 % relative.

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

10. Keywords

10.1 non-heatset web offset inks; non-heatset web offset vehicles; printing inks; printing ink vehicles; VOC in inks; volatile determination; volatiles

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