Designation: D 4713 – 92 (Reapproved 2002)

Standard Test Methods for Nonvolatile Content of Heatset and Liquid Printing Ink Systems[1]

This standard is issued under the fixed designation D 4713; 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 These test methods cover the determination of weight content of nonvolatile matter in two types of printing inks.

1.2 Test Method A is applicable to heatset-type printing inks and resin solutions; solvents in such systems typically have initial boiling points in the range from 240 to 275°C (470 to 535°F) and vapor pressures less than 0.2 mm Hg.

1.3 Test Method B is applicable to liquid-type printing inks and vehicles based on aqueous or organic solvents that evaporate readily at ordinary room temperatures.

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.*

NOTE 1—Test Method A (for heatset systems) specifies a specimen film thickness that is much thinner than those produced by related test methods; one exception is Test Method B in Test Methods D 1259, which is recommended as a referee test.

NOTE 2—Test Method B (for liquid ink systems) is similar to Test Method D 2369 except that a solvent is not required for spreading the test specimen.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1259 Test Methods for Nonvolatile Content of Resin Solutions[2]

D 2369 Test Method for Volatile Content of Coatings[2]

E 1 Specification for ASTM Thermometers[3]

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens[4]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[4]

3. Summary of Test Methods

3.1 *Test Method A—Heatset Systems*. A 0.15-g specimen is mechanically spread in a 57-mm weighing dish to a nominal thickness of 80 ± 10 g/m^2 and heated in a forced ventilation oven at 110°C for 3 h.

3.2 *Test Method B—Liquid Ink Systems*. A 0.5-g specimen is dispensed into a 57-mm weighing dish by means of a disposable syringe, mechanically spread out, and heated in an oven at 110°C for 1 h.

4. Significance and Use

4.1 Nonvolatile content of printing inks is useful for specification acceptance between the producer and the user.

4.2 In order to obtain accurate results for heatset systems within the specified 3-h heating time, the specimen film thickness must be less than 100 g/m^2, and the oven must have forced ventilation. Thickness of the specimen film is less critical for liquid ink systems.

5. Apparatus

5.1 *Balance*, accurate to 1 mg.

5.2 *Oven*, forced-ventilation type conforming to Type IIB in Specification E 145 and maintained at 110 ± 2°C.

5.3 *Thermometer*, bulb-type, capable of reading 110 ± 2°C, such as Thermometer 88C prescribed in Specification E 1.

5.4 *Weighing Dish*, such as an aluminum foil dish 57 mm wide, the lid of a 1-lb ink can 94 mm wide, or other flat-bottomed container. The bottom of the container must not have a trough or depression into which the test material might collect.

5.5 *Spatula*, or small ink knife.

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

5.7 *Forceps*,

5.8 *Desiccator*,

5.9 *Syringe* [5] (*for liquid ink systems only*), single-use 2 to 5-mL capacity without needle, or other weighing device listed in the Apparatus section of Test Methods D 1259.

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[1] These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are 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 14.03.

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

[5] Available from any scientific supply house.

6. Reagents

6.1 *Toluene*, technical grade.

7. Preparations of Equipment and Sample

7.1 Check the levelness of shelving in the oven; adjust, if necessary. Lay the thermometer on shelf with the bulb at the place where the samples will be placed. Adjust oven controls until thermometer reads 110 ± 2°C. If air flow is adjustable, set control dampers at 50 %.

7.2 Wear disposable gloves prior to handling weighing dish, spreading device, or syringe in order to minimize contamination by moisture from hands.

7.3 Measure diameter of bottom of weighing dish in millimetres. For example, a catalogue listing of 57 mm refers to the top diameter, whereas the bottom diameter may be only 50 mm. The bottom diameter must be used in the calculations of weight per unit area in 9.1.

7.4 Mark weighing dishes with a suitable notation. Rinse with toluene and heat in oven at 110°C for ½ h.

7.5 Thoroughly mix ink in container to ensure that the sample is uniform. Close can after removing specimen. Reseal when finished.

8. Procedure

8.1 *Test Method A—Heatset Systems*:

8.1.1 For each specimen, tare to the nearest milligram two weighing dishes each with a spreading device. Retain spreading device throughout the test.

8.1.2 Transfer a representative portion of the sample to the tip of a spatula and dab about 0.15 ± 0.02 g around the bottom of each 57-mm dish, or 0.43 ± 0.02 g if a 94-mm can lid is used. Quickly reweigh and calculate the weight per unit area in accordance with 9.1. If in excess of 100 g/m^2, discard and weigh out a new specimen.

8.1.3 With spreader, smooth out the specimen into a reasonably uniform film covering the entire bottom of the dish. High viscosity inks may require a few drops of a suitable solvent to aid in spreading out the film.

8.1.4 Place the dishes in the forced draft oven at 110°C for 3 h. Remove dishes from oven, cool in desiccator, and reweigh.

8.2 *Test Method B—Liquid Ink Systems*:

8.2.1 Tare weighing dishes as in 8.1.1. Transfer 2 to 4 mL of representative sample to syringe and weigh. Dispense a 0.5 ± 0.1 g specimen from the syringe to a 57-mm dish, or 1.5 ± 0.1 g to a 94-mm can lid. Immediately spread out as in 8.1.2. Reweigh syringe.

8.2.2 Repeat 8.2.1 with second dish.

8.2.3 Place dishes in forced draft oven at 110°C for 1 h. Remove dishes from oven, cool in desiccator, and reweigh.

9. Calculation

9.1 Calculate initial weight/area of each specimen:

$$S/A = S \times 10^6/3.14\,R^2,\ \text{g/m}^2 \quad (1)$$

where:

S = initial specimen weight, g,

A = area, and

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

NOTE 3—For a dish with a 50-mm bottom diameter, weight/area = 510 *S*. For a can lid with a 94-mm bottom diameter, weight/area = 145 *S*.

9.2 Calculate content of nonvolatile matter as follows:

$$NVM, \% = (W/S) \times 100 \quad (2)$$

where:

W = specimen weight after heating, g.

9.3 *Optional*:

The percent of volatile matter may be calculated by difference as follows:

$$VM, \% = 100 - NVM\ \%. \quad (3)$$

10. Report

10.1 Report *NVM* to the nearest 0.1 % as the mean of replicate determinations.

10.2 *Optional:* Report *VM* to the nearest 0.1 %.

10.3 Report the mean weight per unit area of the initial specimens to the nearest gram per square metre.

11. Precision and Bias

11.1 *Precision*:

11.1.1 *Test Method A—Heatset Systems*. An interlaboratory[6] study was conducted in which one operator in each of five laboratories tested in duplicate on each of two days four heatset printing inks, of which two were low *NVM* (about 50 %) and two were high *NVM* (about 60 %). The round-robin data were analyzed according to Practice E 691. There were no outliers. The within-laboratory pooled standard deviation was found to be 0.44 % absolute at 12 degrees of freedom, and the between-laboratories pooled standard deviation was 2.0 % absolute at 16 degrees of freedom. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

11.1.1.1 *Repeatability*—Two results, each the mean of two runs obtained by one operator, should be considered suspect if they differ by more than 1.2 % absolute.

11.1.1.2 *Reproducibility*—Two results, each the mean of two runs obtained by operators in different laboratories, should be considered suspect if they differ by more than 5.7 % absolute.

11.1.2 *Test Method B—Liquid Ink Systems*. See Precision section of Test Method D 2369.

11.2 *Bias*—In the interlaboratory study of heatset inks described in 11.1, the mean values for *NVM* agreed with the calculated values within 1 % absolute.

12. Keywords

12.1 heatset-type printing inks; liquid printing ink; nonvolatile matter content; ovens; printing inks; resin solutions; solvents; vehicles

[6] Supporting data are available from ASTM International Headquarters. Request RR: D01 – 1053.