



Designation: D 7245 – 07

Standard Test Method for Measuring Total Water and Volatiles in Liquid Coatings Which Produce Cure Water Upon Heating¹

This standard is issued under the fixed designation D 7245; 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 is designed to measure total water which includes cure water resulting from the heat induced condensation reaction of coatings. Cure water cannot be measured directly by Test Method D 4017. This task is accomplished by measuring water content in the vapors evolved during heating. This test method will yield total water content. This test method also permits for the simultaneous determination of total volatile content. The results of this test method may be used to calculate VOC content. Although this test method was designed for phenolic coatings, it can be used with other types of coatings.

1.2 Materials used for method development and evaluation had total water values from 20 to 37 %. Use of this test method on coatings outside these values will need to be validated by the user.

1.3 Sample heating is accomplished with a Brinkmann Instruments Model 832 drying oven,² or other mutually agreed upon alternative, passing all of the evolved vapors into a Karl Fischer titration vessel.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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 to determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:³

D 1193 Specification for Reagent Water

¹ This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

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² Round-robin collaborators used the Model 832 drying oven which were loaned to them by Brinkmann Instruments Westbury, New York 11590. It is not known whether this method is applicable to other similar instruments.

³ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings

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

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

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

3. Terminology

3.1 Definitions:

3.1.1 *cure water, n*—water produced as a product of condensation reaction during cure.

3.1.2 *total water, n*—water in the liquid coating plus cure water produced by the condensation reaction.

4. Summary of Test Method

4.1 A measured quantity of coating is added to a tared glass vial which is sealed and then placed into a preheated oven chamber for the required test duration. Sample is heated at 110°C for one hour. The volatiles are passed into a Karl Fischer titration vessel and total water determined. By subtracting the percent water found in regular Karl Fischer titration, Test Method D 4017, from total water, the percent of cure water can be determined. With the weights being known and vial sealed, total volatile content is obtained with this method.

5. Significance and Use

5.1 In the determination of VOC, cure water is treated as a VOC in other test methods, as these methods are unable to account for cure water. This test method allows taking credit for cure water as total water is measured, a value which includes cure water.

5.2 Total water content and volatile content results obtained with this method may be used in Practice D 3960 to calculate VOC of the coating.

6. Apparatus

6.1 *Glass Vial*—A glass vial measuring 22 mm in diameter, 38 mm in height having a capacity of 6 ml capable of being sealed with a TFE-fluorocarbon septum.

6.2 *Analytical Balance*—Capable of weighing to ± 0.0001 g.

6.3 *Drying Oven*—This instrument is essentially a closed system in which the sample is heated within the heating chamber and the vapors passed to the titration vessel through a connecting tube. See Fig. 1.

6.4 *Karl Fischer Apparatus*—See Test Method D 4017.

6.5 *Syringe*—Minimum of 1 ml but no more than 5 ml capacity equipped without a needle, but with a cap, capable of properly dispensing the coating.



FIG. 1 Drying Oven

7. Reagents⁴

7.1 *Purity of Reagent*—Reagent grade chemicals shall be used in all tests unless otherwise indicated; it is intended that all reagents conform to the specifications of the committee on Analytical Reagents of the American Chemical Society where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit use.

7.2 *Purity of Water*—Unless otherwise indicated references to water shall be understood to mean reagent grade conforming to type II of Specification D 1193.

7.3 *Karl Fischer Reagent*—For ketones.

7.4 *Methyl Propyl Ketone (MPK), or other appropriate solvent*—Technical Grade.

8. Preparation of Apparatus

8.1 Connect transfer line from the oven into the Karl Fischer unit so the end of the tubing is beneath the level of the liquid in the Karl Fischer titration vessel.

NOTE 1—Equipment tested came equipped with a tapered plug designed for the tubing to fit through and which was tapered to fit into the Karl Fischer unit.

NOTE 2—Transfer line should be insulated to avoid condensation of vapors in the line. The use of a heated transfer line is preferred.

8.2 The air-inlet port shall be attached to a source of desiccant-dried air or nitrogen.

NOTE 3—Testing found no appreciable difference between the two.

8.3 Check equipment for leaks.

8.4 Precondition the glass vials and septum by heating in an oven at 110°C for 30 minutes and storing in a desiccator until needed.

9. Calibration and Standardization

9.1 Use the procedure specified in Test Method D 4017 for calibration and standardization of the Karl Fischer apparatus.

9.2 Run a blank on the Methyl Propyl Ketone (MPK) to determine if it contains water. If there is water present in the solvent, proceed to 9.2.1.

9.2.1 Weigh a sample of MPK, record as W_{solvent} , to the nearest 0.1 mg.

9.2.2 Perform Test Method D 4017, record the weight percent water results as W_{water} .

10. Procedure

10.1 Take a representative sample of the liquid coating in accordance with Practice D 3925.

10.2 Thoroughly mix the sample to be analyzed.

NOTE 4—Mixing time of 5 minutes has proven adequate for most samples.

10.3 Should amount of cure water need to be known, determine percent water content on the coating in accordance with Test Method D 4017 (W_{ws}).

10.4 Determine Total Water (W_{wt}) and Volatile content (W_{v}).

10.4.1 Preheat the Drying oven to $110 \pm 2^\circ\text{C}$.

10.4.2 Set the Airflow to 80 ml/min.

10.4.3 Purge transfer line for a period of 5 minutes at an airflow rate of 80 ml/min.

NOTE 5—Testing found use of empty sealed vial served this purpose.

10.4.4 Pretitrate contents of the Karl Fischer titration vessel to endpoint.

10.4.5 Weigh preconditioned empty vial and septum to the nearest 0.0001 g and record weight (W_1).

10.4.6 Using a syringe, draw a sample of coating and cap syringe.

10.4.7 Weigh to the nearest 0.0001 g and record (W_1).

10.4.8 Transfer approximately 0.2 ml (0.3 g) of the sample to the glass vial.

10.4.9 Cap syringe and re-weigh, record the weight (W_2).

10.4.10 Add approximately 0.8 ml of Methyl Propyl Ketone (MPK) or other mutually agreed upon acceptable solvent.

NOTE 6—If water was found in the MPK, the weight of the MPK is to be determined and recorded as W_{solvent2} .

10.4.11 Seal vial.

10.4.12 Shake the sealed vial well to mix.

10.4.13 Place vial in heating port and start the oven to run for a period of 1 hour with air flow at 80 ml/min.

10.4.14 Start the Karl Fischer Apparatus to determine total percent water (W_{wt}).

NOTE 7—If water was found in the MPK, then operator must include weight of MPK (W_{solvent2}) in the denominator of total water calculation.

⁴ Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.