



Designation: D 3792 – 99

Standard Test Method for Water Content of Coatings by Direct Injection Into a Gas Chromatograph¹

This standard is issued under the fixed designation D 3792; 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 for the determination of the total water content of waterborne paints. It has been evaluated for latex systems (styrene-butadiene, poly(vinylacetate)-acrylic, acrylic), epoxy acrylic resin systems and acrylic systems. The established working range of this test method is from 15 to 90 %. There is no reason to believe that it will not work outside of this range.

1.2 The values given in inch/pound units are to be regarded as the preferred unit of measurement. The values given in parentheses are for information only.

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 1193 Specification for Reagent Water²

D 1364 Test Method for Water in Volatile Solvents (Karl Fischer Reagent Titration Method)³

E 180 Practice for Determining Precision Data of ASTM Methods for Analysis and Testing of Industrial Chemicals⁴

3. Summary of Test Method

3.1 A suitable aliquot of whole paint is internally standardized, diluted with dimethylformamide, and then injected into a gas chromatographic column containing a porous polymer packing that separates water from other volatile components.

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 D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved May 10, 1999. Published July 1999. Originally published as D 3792 – 79. Last previous edition D 3792 – 98.

² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.04.

⁴ *Annual Book of ASTM Standards*, Vol 15.05.

4. Significance and Use

4.1 In order to calculate volatile organic content (VOC) in waterborne paints, it is necessary to know the water content. This gas chromatographic test method provides a relatively simple and direct way to determine water content.

5. Apparatus

5.1 *Gas Chromatography*—Any gas-liquid chromatographic instrument equipped with a thermoconductivity detector may be used. Temperature programming capability is preferable, but isothermal operations may be adequate. See Table 1.

5.2 *Column*—The column should be at least 4 ft (1.22 m) of 1/8-in. (3.2-mm) outside diameter tubing of stainless steel, or other suitable material, lined with a TFE-fluorocarbon coating packed with 60/80 mesh (180 to 250 μm) porous polymer packing material.⁵ A longer 6-ft (1.83 m) column can be used to improve resolution.

5.3 *Integrator*—Any electronic integrator that can accurately quantify a gas chromatographic peak is acceptable. Alternatively, a recording potentiometer with a full-scale deflection of 1-10mV, full scale response time of 2 s or less and sufficient sensitivity and stability to meet the requirements of 5.1.

5.4 *Liquid Charging Devices*—Micro syringes of 5–10- μL capacity with a precision of $\pm 0.01 \mu\text{L}$. Automatic injection of samples improves the precision of this test method.

6. Column Conditioning

6.1 *Procedure*—Install the packed column in the gas chromatographic unit leaving the exit end disconnected from the detector. This will prevent any contamination of the detector with the column bleed.

⁵ HayeSep R (silanized), was used in the round robin. Any other porous polymer packing or other column giving equivalent or superior performance may be used. These products are available from most gas chromatography suppliers and distributors.

TABLE 1 Suggested Instrument Conditions

Detector	thermal conductivity
Column	1.22 m × 3.2 mm TFE-fluorocarbon coating coated stainless steel
Packing	60/80 mesh porous polymer
Temperatures, °C	
Sample inlet	240
Detector	250
Column	
Initial	140
Final	240
Program rate	40/min
Carrier gas	helium
Flow rate, ml/min	22.0-22.5
Detector current	150 mA
Specimen size	1-2 µl

6.1.1 Set the carrier gas flow rate at 20 to 30 mL/min if a 1/8-in. (3.2-mm) outside diameter column is used. Purge the column 5 or 10 min before heating.

6.1.2 Heat the column from room temperature to 200°C at 5°C/min and hold this temperature for at least 12 h (overnight). At the end of this time, heat the column at 5°C/min to 250°C (the maximum temperature for this packing) and hold for several hours. Cool the column to room temperature and connect to the detector.

6.1.3 Reheat the column to 250°C at 5°C/min to observe if there is column bleed. Optimum conditioning of this column may take several cycles of the heating program before a flat recorder baseline is achieved.

6.2 Before each calibration and series of determinations (or daily) condition the column at 200°C for 1 h with carrier gas flow and to eliminate any residual volatile compounds retained on the column.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁶ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

7.3 *Carrier Gas*—Helium of 99.995 % or higher purity. High-purity nitrogen may also be used.

NOTE 1—Care should be taken that any moisture that may be present in the carrier gas is eliminated through the use of a suitable carrier gas

⁶ *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.

purifier. Trace levels of water will accumulate on the column at low oven temperatures and may affect the reproducibility as well as the accuracy of the determination.

7.4 *Dimethylformamide (DMF) (Anhydrous)* gas chromatography, spectrophotometric quality (See Note 2).

7.5 *2-Propanol (Anhydrous) (Isopropanol)*—See Note 2.

7.6 *Methanol (Anhydrous)*—See Note 2.

7.7 *Septum Sample Vials*, 10-mL capacity with fluorocarbon-faced septa are preferred.

7.8 *Molecular Sieve*, 2A-3A, 8-12, mesh.

NOTE 2—Dry the DMF, 2-propanol and methanol with the molecular sieve. Verify the absence of water by analysis of the solvents by this GC method.

8. Hazards

8.1 *Dimethylformamide* is hazardous. Check the supplier's Material Safety Data Sheet (MSDS) before use.

9. Preparation of Apparatus

9.1 Install the column in the chromatograph and establish the operating conditions required to give the desired separation (see Table 1).

9.2 Allow sufficient time for the instrument to reach equilibrium as indicated by a stable base line.

9.3 Control the detector temperature so that it is constant to within 1°C without thermostat cycling, which causes an uneven baseline.

9.4 Adjust the carrier-gas flow to a constant value.

10. Calibration

10.1 Using the information in Table 1 (as a guide), select the conditions of temperature and carrier gas flow that give baseline resolution of the water and internal standard.

10.2 *Determination of Relative Response Factors:*

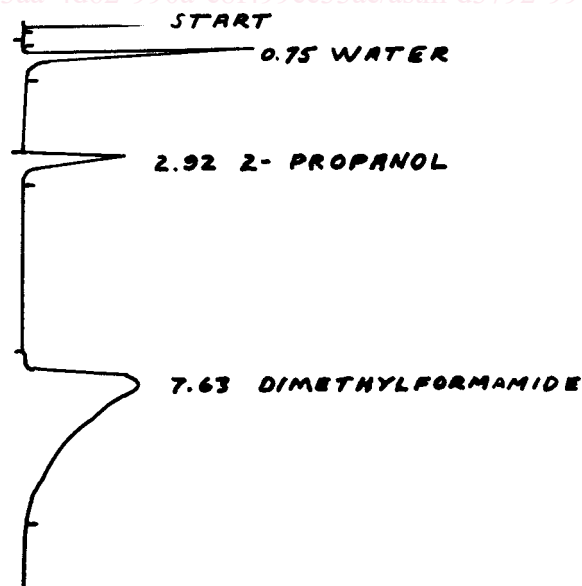


FIG. 1 Typical Chromatogram