Designation: D7358 – 07 (Reapproved 2013)

Standard Test Method for Water Content of Paints by Quantitative Calcium Hydride Reaction Test Kit[1]

This standard is issued under the fixed designation D7358; 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 describes the determination of the total water content of paints using a calcium hydride reaction test kit, or water content between 2 and 85 % water.

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

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:*[2]

D1193 Specification for Reagent Water

D3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings

D4017 Test Method for Water in Paints and Paint Materials by Karl Fischer Method

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

3. Summary of Test Method

3.1 A 0.25 mL sample is reacted with a specially formulated calcium hydride reagent to convert water in the sample to hydrogen gas. The reaction is carried out in a sealed pressure vessel and the resulting pressure is then measured using a specially designed meter programmed to convert gas pressure into water content. The results are displayed in milligrams of water in the reacted sample extract. The actual weight percent water in the whole paint sample is manually calculated using the exact weights of the paint sample and the diluted extract sample, if used.

4. Significance and Use

4.1 Although Test Method D4017 is widely used for the determination of water in paints and related materials, this method may overcome some of the variability found in the Karl Fischer method.

4.2 Control of water content is often important in controlling the performance of paints, and it is critical in determining volatile organic compound (VOC) content when VOC content is measured by difference from total volatile matter and water content as required in certain federal and state regulations.

5. Interferences

5.1 The following compounds, tested at twice the sample size, have been found to produce no measurable response to the calcium hydride reaction test kit and, therefore, cause no interference at levels up to 40 %:[3]

Ethanol
Methanol
Acetone[A]
Methyl Ethyl Ketone
Tetrahydrofuran
Diethylene Glycol Dimethyl Ether
Ethylene Glycol[A]
Diethylene Glycol
Dipropylene Glycol
Stearic Acid
2–Ethyl Hexanoic Acid
Lead Oxide (II and III)
Aluminum Oxide (Brockman I)

[A] 0.25 mL sample size program A and B.

5.2 When using the 5 mL sample size programs, that is, programs C, D, or E, ethylene glycol and acids have been found to produce a positive result in the presence of a small amount of water. Ethylene glycol responds at approximately 7 % of the volumetric equivalent.

[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] 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.

[3] HydroSCOUT User's Manual.

6. Apparatus

6.1 *HydroSCOUT* [4]System—A test kit system consisting of the HydroSCOUT meter, reaction tubes, sampling syringes and dilution vials.[5] The entire analytical sequence, including sampling, sample dilution, chemical reactions and quantification, is available in kit form using pre-dispensed and encapsulated reagents. An analytical balance is required in order to obtain the most precise results. In addition, a mechanical shaker may be necessary to obtain a representative sample.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical reagents of the American Chemical Society, where such specifications are available.[6]

7.2 *Purity of Water*—Unless otherwise indicated, reference to water shall mean reagent water conforming to Specification D1193, Type II.

7.3 *Calcium Hydride (20 %) in HI SOL 10*—contained in a crushable glass ampule.

7.4 *Water*—contained in a crushable glass ampule.

7.5 *Diethylene Glycol Dimethyl Ether (Diglyme)*—2.25 mL contained in a glass vial.

8. Hazards

8.1 Store ampules in a cool, dry place. Keep away from heat, sparks, water and open flames.

8.2 See manufacturer's instructions and Material Safety Data Sheet (MSDS) before use.

8.3 The gray ampule in the tube contains calcium hydride, which is a flammable solid and water reactive.

8.4 Perform test only in a well ventilated area.

8.5 Always wear rubber gloves and safety glasses.

8.6 Take care to ensure that fingers are not cut by glass in the kits. Each ampule should be crushed only once to reduce the risk of glass pieces piercing the sides of the tube.

8.7 When venting reaction tube after completion of test, point tube upright and away from user and bystander.

8.8 When breaking the green water ampule after completion of test, the tube must be shaken vigorously for the full 15 seconds to ensure that the water contacting the remaining calcium hydride does not overheat the side of the tube, otherwise tube failure can result.

9. Sampling, Test Specimens, and Test Units

9.1 *Sample Collection*—Take a representative sample of the liquid coatings in accordance with Practice D3925. Mix thoroughly before taking specimens for individual tests.

10. Preparation

10.1 *Preparation*—Remove a sampling syringe with cap and a test tube from the kit box. Check the contents to ensure that all items are present and intact. If a dilution is to be used, remove a dilution vial and an additional sampling syringe and cap. Remove the screw cap and rubber septum from the tube and the cap from the dilution vial, if used. Insert one green water ampule into the top of the polypropylene ampule sleeve and replace the holder in the tube.

11. Calibration and Standardization

11.1 The commercially available meter is calibrated at the factory over the pressure range of 0 to 60 psig. Each time the meter is turned on, the pressure transducer recalibrates the 0 psig point. If this changes significantly from the factory set point, an error message will be displayed. This error will also appear if a tube is accidentally inserted into the meter before the meter is turned on.

12. Procedure

12.1 *Sample Introduction for Expected Water Content Less Than 15 % (No Dilution)*—Work the plunger on an empty sampling syringe a few times to ensure that it slides easily. Place the tip of the syringe into the paint sample to be tested and slowly pull back on the plunger until it reaches the stop and cannot be pulled further. Remove the syringe from the paint sample and wipe any excess paint from the outside of the syringe with a tissue wipe provided with the kit. Cap the syringe with the cap provided and weigh; record the weight (gross sample weight) to the nearest 0.1 mg. Dispense the sample into the reaction tube. Recap the syringe without wiping, reweigh it, and record this weight (sample tare weight). The difference in the two weights becomes the sample weight (S_1) to be used in Eq 1. Replace the rubber septum (hole on top) by gripping the tube firmly and pressing the septum down with the thumb. (NOTE: Do not squeeze the sides of the tube while inserting the septum.) Replace the screw cap tightly, turning it until it cannot be turned further. Proceed to step 12.3.

12.2 *Sample Introduction for Expected Water Content Greater Than 15 % (With Dilution)*—Work the plunger on an empty sampling syringe a few times to ensure that it slides easily. Place the tip of the syringe into the paint sample to be tested and slowly pull back on the plunger until it reaches the stop and cannot be pulled further. Remove the syringe from the paint sample and wipe any excess paint from the outside of the syringe with a tissue wipe provided with the kit. Cap the syringe with the cap provided and weigh; record the weight (gross sample weight) to the nearest 0.1 mg. Dispense the sample into the dilution vial containing a premeasured amount of solvent (2.15 g). (Do not allow the syringe to contact the dilution solvent when dispensing the sample.) Recap the syringe without wiping, reweigh it, and record this weight (sample tare weight). The difference in the two weights

[4] HydroSCOUT is a trademark of the Dexsil Corporation.

[5] The sole source of supply of this kit known to the committee at this time is Dexsil Corporation, One Hamden Park Drive, Hamden, CT 06517. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[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.

TABLE 1 Calcium Hydride (Percent Water)

Fluid	Average	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit	Bias Versus D4017 Karl Fischer		
	$\bar{X}$	sr	sR	r	R	%	r%	R%
A	68.38	1.045	3.179	2.93	8.90	–4.40	4.28	13.02
B	47.95	1.128	3.656	3.16	10.24	–1.55	6.59	21.35
C	83.93	0.779	4.317	2.18	12.09	–2.41	2.60	14.40
D	17.96	0.659	1.428	1.85	4.00	–8.13	10.28	22.27
E	2.20	0.040	0.118	0.11	0.33	–14.13	5.12	15.02
F	12.89	0.239	0.320	0.67	0.89	2.72	5.20	6.94
			Average	1.81	6.07	–4.65	5.67	15.5

TABLE 2 Test Method D4017, Test Method for Water in Paints and Paint Materials by Karl Fischer Method (Percent Water)

Fluid	Average	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit		
	$\bar{X}$	sr	sR	r	R	r%	R%
A	71.53	0.931	5.656	2.61	15.84	3.64	22.14
B	48.71	0.699	2.623	1.96	7.34	4.02	15.08
C	86.01	0.943	2.198	2.64	6.15	3.07	7.16
D	19.54	0.367	1.623	1.03	4.55	5.27	23.26
E	2.56	0.248	0.286	0.69	0.08	27.08	31.24
F	12.55	0.338	2.345	0.95	6.56	7.55	52.33

becomes the sample weight S_1 to be used in Eq 2. Replace the cap on the dilution vial and shake the vial vigorously for at least 10 seconds. Remove the cap and quickly, using a new syringe, draw up a sample from the solution. Wipe any excess solution from the outside of the syringe, cap and weigh the syringe. Record this weight (diluted sample gross weight). Dispense this sample into the reaction tube. Recap the syringe, without wiping it, and reweigh the syringe and record this weight (diluted sample tare weight). The difference in the two weights becomes the diluted sample weight (S_2) to be used in Eq 2. Replace the rubber septum (hole on top) by gripping the tube firmly and pressing the septum down with the thumb. (NOTE: Do not squeeze the sides of the tube while inserting the septum.) Replace the screw cap tightly, turning it until it cannot be turned further.

12.3 *Reaction*—Break the bottom (gray) ampule in the tube by compressing the sides of the tube. Mix thoroughly by shaking the tube vigorously for 30 seconds. The mixture will be seen to bubble and perhaps foam. Allow the reaction to proceed for 2 minutes. Shake the reaction tube again for 10 seconds and let stand for an additional 1 minute.

12.4 *Analysis*—Ensure the meter is set for the correct program for determining water in paint and that the meter is ready for measurement. Insert the reaction tube firmly into the opening at the bottom of the meter. The hole in the white cap must be lined up with the metal pin extending from the inside of the hole in the meter. Press the "READ" button on the face of the meter. The display will flash the word "CALC," then the results will be displayed in milligrams of water in the sample. Record the results. Keep the meter and tube upright at all times.

12.5 *Disposal*—Carefully remove the reaction tube from the meter. Vent tube by inserting completely into venting cap and removing. Tube and cap must be upright and pointed away from user and bystanders. Tap the tube on the bench top to shake any loose glass to the bottom of the tube, then break the top (green) ampule in the reaction tube by compressing the sides of the tube. Shake the tube for 15 seconds. After any foam has settled, vent the tube again. Discard the tube and vial as laboratory waste. Do not remove cap.

13. Calculations

13.1 *Calculations for Direct Sample (No Dilution)* —The meter is programmed to display the weight, in milligrams, of the water contained in the sample introduced into the reaction tube (in this case the paint sample). Calculate the water content in the original paint sample in weight percent using Eq 1:

$$\textit{Water in paint, wt. percent} = R/(10{*}S_1) \quad (1)$$

where:

R = meter reading in milligrams of water
S_1 = actual weight of whole paint sample, grams

13.2 *Calculations for Diluted Sample (With Dilution)*—The meter is programmed to display the weight, in milligrams, of the water contained in the sample introduced into the reaction tube (in this case the diluted sample). Calculate the water content in the original paint sample in weight percent using Eq 2:

$$\textit{Water in paint, wt. percent} = (R/S_2)(1+(2.15/S_1))/10 \quad (2)$$

where:

R = meter reading in milligrams of water
S_1 = actual weight of whole paint sample, grams
S_2 = actual weight of diluted paint sample, grams
2.15 = weight of the dilution solvent, grams

14. Report

14.1 Report calculated water content in weight percent to three significant figures (see Practice E29 for guidance). The

report should also include the method used to determine the water content: Test Method D7358.

15. Precision and Bias[7]

15.1 The precision of this test method is based on an interlaboratory study of Test Method D7358, conducted in 2006. Each of six laboratories tested six different materials using two analyses for the determination of water content; Karl Fischer and a calcium hydride test. Every "test result" represents an individual determination. Each laboratory obtained two replicate test results (from one operator) for each material/analysis combination.

15.1.1 *Repeatability*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than 5.7 % of the measured value; Repeatability "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory. For more detailed information on the repeatability data for the individual samples tested, see Table 1.

15.1.2 *Reproducibility*—Two test results shall be judged not equivalent if they differ by more than 15.5 % of the measured value; Reproducibility "*R*" is the interval representing the difference between two test results for the same material, obtained by different operators using different equipment in different laboratories. For more detailed information on the reproducibility data for the individual samples tested, see Table 2.

15.1.3 Any judgment in accordance with these two statements would have an approximate 95 % probability of being correct.

15.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

15.3 *Relative Bias*—The interlaboratory study (ILS) was not specifically designed to determine the relative bias between this test method and Test Method D4017. A statistically significant statement of relative bias is, therefore, not possible. The relative bias information (Table 1) is presented here for informational purposes only.

15.4 The precision statement was determined through statistical examination of 144 results, from six laboratories, on six materials. Following are descriptions of the six fluids tested:

Fluid A:	Clear Acrylic sealer
Fluid B:	Acrylic white tint base
Fluid C:	Acrylic stain
Fluid D:	Phenolic resin containing approximately 10 % unreacted Phenol
Fluid E:	Pigmented polyester polyol resin system containing MEK, Toluene, Ethyl Acetate, PM Acetate, and Ethyl 3-Ethoxy Propionate
Fluid F:	Phenolic coating containing approximately 5 % unreacted Phenol as well as Ethyl Alcohol, PM Acetate, and Dipropylene Glycol Monomethyl Ether

15.4.1 To judge the equivalency of two test results, it is recommended to choose the fluid closest in characteristics to the test fluid.

16. Keywords

16.1 moisture content; test kits; water content; water content of paints by test kits

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1139.