Designation: D7232 − 06 (Reapproved 2016)

Standard Test Method for
Rapid Determination of the Nonvolatile Content of Coatings by Loss in Weight[1]

This standard is issued under the fixed designation D7232; 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 used to obtain rapid determination of the weight percent nonvolatile (solids) content via instrumental loss in weight technology. It is not meant as a replacement for Test Method D2369.

1.2 This test method is principally intended for quality control labs and manufacturing environments where previously characterized materials will be tested repeatedly for different batches or lots.

1.3 This test method can be used for waterborne and solventborne resins, intermediates and finished paint products. This test method may not be applicable to all types of coatings.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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.*

NOTE 1—There is no similar or equivalent ISO standard.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D16 Terminology for Paint, Related Coatings, Materials, and Applications
D2369 Test Method for Volatile Content of Coatings
E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]

3. Terminology

3.1 *Definitions:*

3.1.1 The definitions used in this test method are in accordance with Terminology D16.

3.1.2 *nonvolatile content, n*—the coating material that remains in the pan at the conclusion of the test.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *flip and squish, n*—a testing technique that may be used when the expected nonvolatile content is greater than 40 %, or when the sample is highly viscous and does not absorb well into the filter paper.

3.2.1.1 *Discussion*—The specimen is applied to the filter paper on the sample pan, the filter paper is "flipped" over and the specimen is then "squished" between the filter paper and the sample pan in order to more uniformly distribute the specimen. In addition, use of this technique forces the glass fibers of the filter paper into the specimen, helping to create pathways for volatiles release from the specimen and avoiding incomplete volatiles removal due to "skinning over" of the sample material.

3.2.2 *lift, n*—the result of convection currents created during the heating of the specimen that raises the sample pan off of its support and falsely indicates a weight loss.

3.2.2.1 *Discussion*—This effect is compensated for by the use of an algorithm that is applied to the digital data.

3.2.3 *syringe tare, n*—a testing technique that may be used when the expected nonvolatile content is less than 40 %, or when the sample is highly volatile and tends to evaporate rapidly.

3.2.3.1 *Discussion*—The specimen weight is determined using an external balance by calculating the difference between the syringe weight before (initial weight) and after (final weight) the specimen is applied to the pan. This difference between initial and final weight is the actual weight of specimen (see 10.2), and is used to minimize error due to rapid change of the specimen weight after addition to a heated sample pan.

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[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] The last approved version of this historical standard is referenced on www.astm.org.

4. Summary of Test Method

4.1 The specimen is spread onto a sample pan that is supported on a balance in a heating chamber that has been preheated and equilibrated to the specified idle temperature. It is then heated to the specified test temperature to vaporize the volatiles. The analysis is completed when the indicated rate of weight loss falls below a rate specified in the test conditions. The total weight loss is calculated and reported as weight percent nonvolatiles. Both the analyzer's balance and heater are calibrated with NIST-traceable standards to achieve precise and accurate results.

4.2 Through adjustment of the analyzer's parameter settings, a set of optimal conditions is developed for each material type to measure the percent nonvolatiles. These optimal conditions are recorded and may be used for repeat testing of that material.

5. Significance and Use

5.1 This test method is intended for use as a rapid quality control, acceptance, and assessment test. Results are obtained in five to fifteen minutes on most materials. Since the instrument parameters are adjusted to produce the same results as Test Method D2369, which takes over one hour to run, the time and effort expended on determining the optimal conditions for testing a coating with this instrumental method is valuable when numerous measurements are going to be made on different lots or batches of the same material. Also, the automation of the measurement and the calculations should lead to fewer mistakes being made by less-trained operators.

6. Apparatus

6.1 *Analyzer,* containing:

6.1.1 An oven capable of heating the sample to at least 225°C.

6.1.2 A balance capable of measuring to the nearest 0.0001 g.

6.1.3 An electronic means of compensating for lift caused by convection currents created during testing.

6.1.4 A processor that is capable of converting the loss of weight to digital data.

6.1.5 Digital display for presenting the digital data as weight percent nonvolatiles.

6.2 Flat disposable pan, of aluminum alloy 3003, with smooth, uncoated, oil-free surface.

6.3 Round glass-fiber filter paper, Grade 111.

6.4 Syringe, 3 cc plastic slip-tip without needle but with cap, capable of dispensing specimen onto pan.

6.5 Nitrogen compressed gas (N_2) – dry and oil-free.

6.6 Compressed gas regulator(s), as needed to supply N_2 from high-pressure sources to controlled delivery pressures that are appropriate for the apparatus.

7. Reagents

7.1 *Sodium Tartrate Dihydrate*—ACS certified reagent grade.

8. Calibration and Standardization

8.1 To maintain the integrity of the test results, the balance shall be calibrated using NIST-traceable weights and the heater shall be calibrated using an NIST-traceable temperature calibration interface per the analyzer manufacturer's guidelines.

8.2 The calibration may be verified using *sodium tartrate dihydrate*, which has a theoretical water content of 15.66 %, with an acceptable result range of 15.61 to 15.71 %. Other procedures for materials with known theoretical water content are acceptable for verification as specified by the analyzer manufacturer.

8.3 Prepare the analyzer for use, select the preprogrammed instrument parameters for *sodium tartrate dihydrate* (or other standard material if applicable) and prepare analyzer for analysis as described in 9.1 using a flat pan without filter paper.

8.4 Initiate the test on the analyzer and follow the prompts for placing the specimen on the sample pan.

8.5 Spread a thin, even layer of *sodium tartrate dihydrate* of appropriate specimen size onto the pan, then close lid to begin test. Specimen size shall be determined by analyzer manufacturer.

8.6 If results are not within the acceptable range, first perform a temperature calibration, temperature calibration verification, and then a balance calibration to ensure proper analyzer performance. Retest with *sodium tartrate dihydrate* (or other standard material as specified by the instrument manufacturer). If results still are not within the acceptable range, contact analyzer manufacturer.

9. Procedure

9.1 *Preparing Analyzer for Sample Analysis:*

9.1.1 Place the analyzer on a flat, level surface.

9.1.2 Establish N_2 purge to the heating chamber per the instrument manufacturer's instructions.

9.1.3 Turn the analyzer on and allow equilibration at the recommended idle temperature for balance calibration for 30 min.

9.1.4 Perform balance calibration per the analyzer manufacturer's instructions.

9.2 *Performing Sample Analysis:*

9.2.1 Program the analyzer with the desired test parameters, or select the suggested test conditions from Annex A1. See 9.3.1 for determining the optimal conditions for testing a coating. See 9.3.5 for repeat testing of a coating using previously determined optimal conditions.

9.2.2 Place a clean, flat sample pan with glass filter paper, rough side up, on the pan support and close the lid. Allow the analyzer to equilibrate at the desired idle temperature.

9.2.3 Ensure sample material is thoroughly mixed before drawing specimen into syringe.[4] If using syringe tare technique (see 3.2.3), proceed to step 9.2.4. If using flip and squish (see 3.2.1) technique, proceed to step 9.2.5.

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[4] The specimen size will depend on the test conditions specified for a particular analyzer. See Table A1.1 in Annex A1 for suggested sample size for specified test conditions.

9.2.4 If syringe tare is used:

9.2.4.1 Initiate the test on the analyzer and follow the prompts for placing the specimen on the sample pan.

9.2.4.2 Draw sample material into the syringe, then wipe the syringe to ensure that no sample material remains on the exterior, then cap the syringe tip.

9.2.4.3 Weigh the loaded syringe with cap on a balance and record the result. This is the initial weight.

9.2.4.4 Quickly apply specimen to the filter paper on the pan by dispensing material from syringe onto the filter paper in a spiral pattern, then recap the syringe and close lid to begin test.

9.2.4.5 Immediately weigh the syringe and cap and record the result. This is the final weight. The difference between the initial and final weight of the syringe and cap is the actual specimen weight for the test, which is calculated as follows:

$$W_A = W_I - W_F \tag{1}$$

where:

W_A = actual weight of specimen to be entered into analyzer,
W_I = initial weight of loaded syringe and cap, and
W_F = final weight of syringe and cap after dispensing specimen.

9.2.5 If the flip and squish is used:

9.2.5.1 Draw sample material into the syringe.

9.2.5.2 Initiate the test on the analyzer and follow the prompts for placing the specimen on the sample pan.

9.2.5.3 Quickly apply specimen to the filter paper on the pan by dispensing material from syringe onto the filter paper in a spiral pattern.

9.2.5.4 In rapid succession, remove the sample pan from the support, flip over the filter paper with tweezers and gently squish the specimen between the filter paper and the sample pan. Place sample pan back onto pan support and close lid to begin test. Do not allow tweezers to come in contact with the specimen on the filter paper.

9.2.6 At the end of the test, allow the analyzer to cool and remove the sample pan. If syringe tare was used, input the actual specimen weight at the completion of the test to obtain the final result.

9.2.7 Record the result as displayed in percent nonvolatiles.

9.3 *Determination of Optimal Test Conditions:*

NOTE 2—When determining the optimal test conditions for a material, it is useful to have a calibrated forced-draft oven available and test the material in accordance with Test Method D2369.

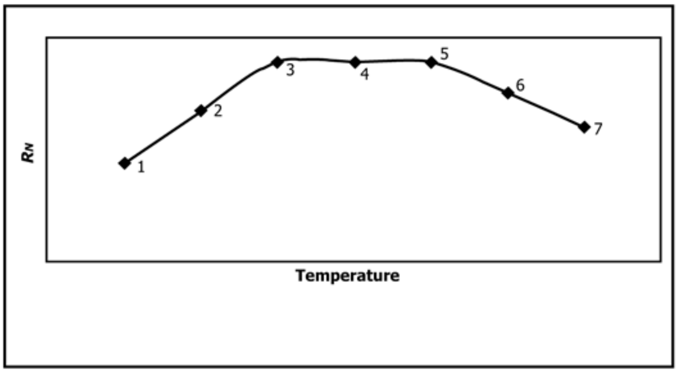

FIG. 1 Optimum Test Temperature Selection

9.3.1 Program the analyzer according to the conditions listed in Annex A1.3.

9.3.2 To determine the optimum test temperature for a coating, run one series of tests on a single coating specimen that consists of several consecutive programs that have been linked together. Each program is identical in its parameters except for the temperature, which is progressively increased 5°C on each successive program.

NOTE 3—For each test in the series, ensure that the ending weight of one test is used for the beginning weight of the subsequent test.

NOTE 4—Ensure that the program selected to run first corresponds to the lowest temperature in the linked series.

9.3.3 Record the result for each test in the linked series as the ratio of the ending weight to the beginning weight in percent by calculating as follows:

$$R_N = \left[\left(E_N / B_N \right) \times 100 \right] \tag{2}$$

where:

R_N = ratio of ending weight to beginning weight in percent for a given linked test N (to be plotted against temperature),
E_N = ending weight for linked test N, and
B_N = beginning weight for linked test N.

9.3.4 After the tests are completed, plot each linked test result, R_N, versus temperature to make a curve as in Fig. 1.

9.3.4.1 Most of the volatiles are vaporized in the temperature range from points 1 to 3.

NOTE 5—The ratio of the ending weight to beginning weight increases with temperature for the specimen during the first few tests in linked series as the higher temperatures evaporate more and more of the volatile content.

9.3.4.2 Between points 3 and 5, the results approach 100 % (the ratio of ending weight to beginning approaches 1) and become constant. Choose a temperature in this range as the optimum test temperature for that specimen material. A temperature in this range, where the R_N value first becomes constant, ensures that there will be a total loss of volatiles from the specimen material during routine analysis, and that the temperature is not excessively high.

9.3.4.3 Beyond point 5, the results may begin to decrease. This trend is likely caused by decomposition of the sample.

NOTE 6—The region in the graph beyond point 5 may not necessarily be observed. The optimum temperature may be determined as described in step 9.3.4.2 before a temperature sufficient to degrade the specimen is reached.

9.3.5 After the optimal test temperature has been determined, adjust other appropriate parameters as needed to optimize correlation with results from an analysis by D2369 on the same material.

9.3.6 Once correlation has been optimized, record parameters for use on repeat tests of the same material.

9.4 *Sample Analysis for Repeat Tests:*

9.4.1 Following steps 9.2 through 9.2.7, and using optimized test conditions as determined in 9.3, determine nonvolatile content for each coating by performing the analysis in duplicate.

10. Calculation

10.1 Result is reported in weight percent nonvolatiles to three decimal places so no further calculations are necessary.

10.2 Calculate mean, N, percent nonvolatile content as follows:

$$N = (N_A + N_B)/2 \tag{3}$$

where:

N = mean percent nonvolatile content,
N_A = first weight percent nonvolatile determination, and
N_B = duplicate weight percent nonvolatile determination.

11. Report

11.1 Report the following information:

11.1.1 Complete identification of the sample tested,

11.1.2 Analyzer settings, and

11.1.3 N, the weight percent nonvolatiles as the mean of two determinations if the absolute percent difference is 0.6 or less. If the absolute difference between N_A and N_B is greater than 0.6, repeat duplicate determinations.

12. Precision and Bias

12.1 The estimated precision is based on an interlaboratory study in which 1 operator in each of 7 laboratories analyzed in duplicate on 2 different days 4 samples of waterborne paints and 2 samples of solventborne paints containing between 43 and 72 % volatile material. The paints were commercially supplied. The tests were conducted with Computrac MAX2000XL moisture analyzers.[5] The results were analyzed statistically in accordance with Practice E180. The within-laboratory standard deviation for two results, each the mean of duplicate determinations, obtained by the same operator, was found to be 0.22 absolute at 42 DF and the between-laboratories standard deviation to be 0.62 absolute at 6DF. Based on these values, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

12.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 0.6 absolute.

12.1.2 *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 1.7 absolute.

12.2 *Bias*—No information can be presented on the bias of the procedure in Test Method D7232 for measuring nonvolatile content because there is no accepted standard for nonvolatile content in coatings.

13. Keywords

13.1 nonvolatile; VOC; volatile; solids

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[5] The interlaboratory and within-laboratory studies were conducted using this particular model of analyzer. The precision and bias statement may be not applicable to other analyzers.

ANNEX

(Mandatory Information)

A1. COMPUTRAC MAX 2000XL MOISTURE ANALYZER

A1.1 The test conditions for *sodium tartrate dihydrate* are pre-programmed into the MAX 2000XL as a method labeled TARTRATE.

A1.2 Suggested test conditions for selected coatings are given in Table A1.1:

A1.3 Use the following guidelines for determining optimal test conditions:

A1.3.1 For solventborne coatings program the instrument as follows and then perform 9.3 to determine the optimal test temperature:

Temperatures – Test – Set to 150°C
Hi Start - 25°C
Idle - 50°C
Ending Criteria – End on Rate – 0.100 %/min
Sample Size – 2 +/− 0.5 g sample window
Tare Options – Pan Tare – Standard
Sample Tare – 3 s
Lift Compensation – 100 %

A1.3.2 For waterborne coatings program the instrument as follows and then perform section 9.3 to determine the optimal test temperature:

Temperatures – Test – Set to 135°C
Hi Start - 25°C
Idle - 50°C
Ending Criteria – End on Rate – 0.100 %/min

TABLE A1.1 Suggested Test Conditions for Selected Coatings

Material	Test Temperature (°C)	Idle Temperature (°C)	Rate (%/min)	Sample Size (grams)	Pan Tare	Sample Tare (seconds)	Lift Compensation
Solventborne	150	50	0.100	1.5 – 2.5	Standard	3	100
Waterborne	135	50	0.100	1.5 – 2.5	Standard	3	100

Sample Size – 2.0 +/− 0.5 g sample window
Tare Options – Pan Tare – Standard
Sample Tare – 3 s
Lift Compensation – 100 %

A1.3.3 After the optimal test temperature has been determined, use the following guidelines to correlate with a result obtained using Test Method D2369:

A1.3.3.1 If the nonvolatile content obtained on the MAX 2000XL is greater than Test Method D2369 result, decrease the ending criteria rate in increments of 0.025 %/min until acceptable correlation is achieved. If unable to achieve correlation using ending criteria, decrease the lift compensation in increments of 25 until acceptable correlation is achieved.

A1.3.3.2 If the nonvolatile content obtained on the MAX 2000XL is less than Test Method D2369 result, increase the ending criteria rate in increments of 0.025 %/min until acceptable correlation is achieved. If unable to achieve correlation using ending criteria, increase the lift compensation in increments of 25 until acceptable correlation is achieved.

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