Designation: D7232 – 06 (Reapproved 2022)

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

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

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]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

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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.

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.

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.