Designation: D 2222 – 94 (Reapproved 1999)

Standard Test Method for Methanol Extract of Vinyl Chloride Resins[1]

This standard is issued under the fixed designation D 2222; 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 covers the determination of the methanol extract, or nonvolatile methanol-soluble portion, of vinyl chloride resins.

1.2 The values stated in SI units are to be regarded as the 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.*

NOTE 1—There are no ISO standard covering the primary subject matter of this ASTM standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 883 Terminology Relating to Plastics[2]

D 1600 Terminology of Abbreviated Terms Relating to Plastics[2]

3. Terminology

3.1 *Definitions:* Definitions are in accordance with Terminologies D 883 and D 1600 unless otherwise indicated.

4. Summary of Test Method

4.1 The methanol-soluble materials are extracted from the resin in a Soxhlet extractor, the methanol evaporated to dryness, and the residue weighed as the weight percent methanol extract.

5. Significance and Use

5.1 The methanol extract test is most commonly employed with paste- or dispersion-type vinyl resins intended for organosol or plastisol applications. The test result is a quantitative measure of the methanol-soluble, nonvolatile, essentially nonpolymeric content of the virgin, unmodified resin. The major ingredient removed is the soap system employed in the polymerization reaction; methanol extract provides a measure of lot-to-lot uniformity of the resin in this respect.

6. Apparatus

6.1 *Soxhlet Extractor*. [3]

6.2 *Extraction Thimbles*, 33 by 94-mm.[4]

6.3 *Oven, Constant-Temperature*, maintained at 105 ± 3°C.

6.4 *Electric Heating Mantle* for a 250-mL extractor flask, or electric strip heaters, equipped with a suitable variable transformer to control the rate of heating.

7. Reagents

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.[5] 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.

8. Procedure

8.1 Weigh to the nearest 0.001 g approximately 12 g of the resin sample into an empty extraction thimble.

8.2 Cover the resin in the thimble with a small pad of glass wool to prevent spattering of resin during the extraction and place the thimble in the extractor.

8.3 Weigh to the nearest 0.001 g and record the tare weight of a clean, dry, flat-bottom, extractor flask containing a Raschig ring.

8.4 Add 200 mL of anhydrous methanol to the flask and connect the flask to the extractor.

---

[1] This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D 20.15 on Thermoplastic Materials (Section D20.15.08).

Current edition approved March 15, 1994. Published May 1994. Originally published as D 2222 – 63 T. Last previous edition D 2222 – 66 (1988).

[2] *Annual Book of ASTM Standards*, Vol 08.01.

[3] Corning Glass Co. No. 3840, Fisher Scientific Co. No. 9-556 (Size B), or equivalent have been found satisfactory for this purpose.

[4] Fisher Scientific Co. No. 9-655, or equivalent, has been found satisfactory for this purpose.

[5] *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.

8.5 Run a blank determination on the methanol simultaneously with each set of extractions.

8.6 Start the extraction and regulate the reflux so that the methanol collecting in the thimble drains at least six times per hour.

8.7 Timing the extraction from the complement of the first cycle, extract the resin for 6 h ± 10 min.

NOTE 2—Continuous or bulk extractions that do not employ an extractor assembly require approximately 12 h to complete.

8.8 Drain all the methanol from the extractor into the flask and evaporate most of the methanol over a steam bath.

8.9 Complete the methanol evaporation by heating the flask in an oven at 105 ± 3°C for 30 min.

8.10 Allow the flask to cool in a desiccator for 1 h.

8.11 Obtain two weights within 30-min intervals that agree to ± 0.001 g.

8.12 Run a blank using the same procedure but omitting the resin sample.

9. Calculation

9.1 Calculate the percentage of methanol extract as follows:

$$\text{Methanol extract, \%} = (A - B - C) \times (100/S)$$

where:

A = weight of flask, Raschig ring, and extract,
B = tare weight of flask and Raschig ring,
C = gain in weight of flask and Raschig ring during blank test, and
S = weight of sample.

10. Report

10.1 The report shall incude the following:

10.1.1 Complete sampling identification, and

10.1.2 Percentage methanol extract, average and range.

11. Precision

11.1 The average of duplicate determinations should agree to within 0.1 %.

NOTE 3—Supporting round-robin data have not been found as yet. A task group has been formed to generate new data in the event that no previous data can be found.

12. Keywords

12.1 methanol extract; PVC resin