Designation: D6050 – 09 (Reapproved 2016)

Standard Test Method for Determination of Insoluble Solids in Organic Liquid Hazardous Waste[1]

This standard is issued under the fixed designation D6050; 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 approximate amount of insoluble, suspended solid material in organic liquid hazardous waste (OLHW).

1.2 This test method is intended to be used in approximating the amount of insoluble, suspended solids in determining the material handling characteristics and fuel quality of OLHW. It is not intended to replace more sophisticated procedures for the determination of total solids.

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:*

D96 Test Method for Water and Sediment in Crude Oil by Centrifuge Method (Field Procedure) [2]

3. Summary of Test Method

3.1 A 10-mL aliquot of OLHW sample is decanted into a 15-mL graduated centrifuge tube and centrifuged for 3 min. The separated liquid phase of the OLHW is decanted into an appropriate waste vessel. The centrifuge tube with the separated solid material is brought back to its original 10-mL volume with a user-selected blend of clean solvents and agitated to mix the solid and liquid phases. The tube is centrifuged for 2 min, and the amount of remaining solid material is read.

4. Significance and Use

4.1 A high percentage of insoluble, suspended solid material can create pumping, filtering, or grinding difficulties in the off-loading of bulk shipments of OLHW and can contribute to excessive wear on processing equipment. High solids can also decrease the quality and consistency of commingled solutions by decreasing the effectiveness of agitation in storage tanks. These issues are of concern to the recycling industries (solvents, paints, and other materials handled in significant quantities) in addition to those activities that propose to use the waste as a fuel.

5. Apparatus

5.1 *Centrifuge*—Capable of spinning two or more centrifuge tubes at a speed controlled to give a relative centrifugal force of between 1200 to 1400. The speed to achieve this is generally between 3100 to 3600 rpm. The rotation speed necessary to achieve the relative centrifugal force can be determined from one of the following equations:

$$rpm = 1335\sqrt{\frac{rcf}{d}} \quad (1)$$

$$rpm = 265\sqrt{\frac{rcf}{d}} \quad (2)$$

where:

rpm = rotation speed, in revolutions per min,
rcf = relative centrifugal force,
d = diameter of swing, in mm (Eq 1) or in. (Eq 2), measured between the tips of opposite tubes when the tubes are in rotating position.

NOTE 1—Eq 1 and Eq 2 are described in Test Method D96.

5.2 *Centrifuge Tubes*— Centrifuge tubes shall be cone shaped, made of glass or a solvent resistant plastic or polymer, have a minimum capacity of 15 mL when filled to volume, and graduated with minimum subdivisions of 0.5 mL. Class A centrifuge tubes are recommended. If any grade other than Class A is used, refer to the section on Calibration and Standardization.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where

[1] This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.06 on Analytical Methods.

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

such specifications are available.[3] Other grades may be used, providing that it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Clean Solvent Blend*—The user should formulate the clean solvent blend based on the "average" normal chemical composition of the commingled OLHW. The chemical composition of OLHW is usually determined by gas chromatographic analysis. The clean solvent blend will vary in complexity depending on the specifications of the user.

NOTE 2—The clean solvent blend may be as simple as a single component (for example, toluene) or a complex mixture of aliphatic and aromatic hydrocarbons, alcohols, esters, ketones, ethers, terpenoids, and halogenated compounds. The user should formulate the clean solvent blend based on compositional knowledge of the OLHW that the sample material is to be commingled with. If the commingled OLHW contains water, it may be appropriate to include in the clean solvent blend.

7. Sample

7.1 Because settling of insoluble solid material in liquid samples is probable, the laboratory sample should be thoroughly mixed by shaking prior to withdrawing a portion for testing.

8. Calibration and Standardization

8.1 Centrifuge tubes other than Class A must be periodically checked for accuracy. The frequency of this check will be determined by the user, but should be minimally done for each new box or lot number of tubes.

9. Procedure

9.1 Fill a centrifuge tube to the 10-mL graduation mark with the OLHW sample.

NOTE 3—It will be necessary to use an additional tube with an equal mass of water or other suitable liquid to balance the centrifuge.

9.2 Place the tubes from 9.1 in the trunnion cups or centrifuge tube slots on opposite sides of the centrifuge. Close the centrifuge lid and engage safety lock, if so equipped.

9.3 Set the centrifuge speed (r/min) to provide a minimum relative centrifugal force between 1200 to 1400 (see 5.1).

9.4 Engage centrifuge, and allow to spin for 3 min.

9.5 Remove the tube containing OLHW, and decant the separated liquid phase to an appropriate waste vessel.

9.6 Return the tube with solids to its original 10-mL volume with the clean solvent blend. Cap or stopper the tube and shake vigorously in order to completely mix the centrifuged solid phase with the solvent phase. It may be necessary to use a Vortex mixer for thorough mixing.

9.7 Replace the OLHW tube in the centrifuge, and set centrifuge as described in 9.2 and 9.3.

9.8 Engage centrifuge, and allow to spin for 2 min.

9.9 Remove the OLHW tube, and decant the separated liquid phase of each to an appropriate waste vessel.

9.9.1 Visually estimate and record the amount of centrifuged solids in the tubes to the nearest 0.25 mL.

10. Quality Control

10.1 Each laboratory using this test method will operate a formal quality control program. This program shall include elements that address analyst proficiency through the evaluation of method blanks, duplicates, and reference materials if available.

11. Calculation

11.1 The estimated amount of solid material is calculated as follows:

$$(A/10\ \mathrm{m}L)\ 100 = \text{Insoluble solids, percent} \qquad (3)$$

where:

A = volume of solids from centrifuge tube.

12. Precision and Bias

12.1 *Precision*—The precision of this test method is being determined.

12.2 *Bias*—The bias of this test method has not been determined because there are no recognized reference materials.

13. Keywords

13.1 centrifuge; hazardous waste; insoluble solids; organic liquid hazardous waste; suspended solids

[3] *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.