Designation: E1673 – 21

Standard Test Method for Percent Suspensibility[1]

This standard is issued under the fixed designation E1673; 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 determine the percent suspensibility of dry pesticide formulations.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements see Section 7.

1.4 *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]

D1126 Test Method for Hardness in Water

D1193 Specification for Reagent Water

3. Summary of Test Method

3.1 A known quantity of dry pesticide is slurried into 50 mL of test water in a 100 mL beaker. The slurry is quantitatively transferred to a 250 mL mixing cylinder using additional test water to rinse the beaker. The 250 mL mixing cylinder is then filled to volume with test water. The mixing cylinder is stoppered and inverted 15 complete cycles. The mixing cylinder is allowed to stand for 30 min. After 30 min the top 225 mL is drawn off and the remaining suspension is dried. The residue weight will determine the percent suspensibility.

4. Significance and Use

4.1 This test method is designed specifically for dry formulations, but need not be restricted to these materials.

4.2 Either option of this test method (see Section 8) may be used to determine the percent suspensibility.

4.3 This test method may not be applicable to all dry formulations such as those containing liquid or soluble technical/pesticides or ingredients that rise to the top upon separation.

4.4 This test method may not be applicable to formulations containing ingredients that decompose under the test conditions.

4.5 This test method may not give reproducible results if flocculation occurs.

4.6 This test method should be run in duplicate.

4.7 Products containing water soluble or volatile components may result in errors.

5. Apparatus

5.1 *Balance,* top loading, with an accuracy of ±0.01 g or better.

5.2 *Beaker,* 100 mL.

5.3 *Mixing Cylinder,* stoppered, 250 mL, flat bottom, KIMAX series 20039 or equivalent.

5.4 *Timer,* adjustable, with an accuracy of ± min.

5.5 *Magnetic Stirrer,* 120 rpm to 1200 rpm, or equivalent.

5.6 *Stir Bar,* magnetic 1 in. in length and ⅜ in. in diameter (2.5 cm × 1 cm).

5.7 *Weighing Dish,* aluminum (57 mm × 18 mm) or petri dish, or equivalent.

5.8 *Vacuum Apparatus,* see Fig. 1, equipped with a vented stopper to prevent the formation of a vacuum.

5.9 *Filtering Flask,* heavy wall, 500 mL, KIMAX Series 27060 or equivalent.

5.10 *Gravity Oven.*

[1] This test method is under the jurisdiction of ASTM Committee E35 on Pesticides, Antimicrobials, and Alternative Control Agents and is the direct responsibility of Subcommittee E35.22 on Pesticide Formulations and Delivery Systems.

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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.

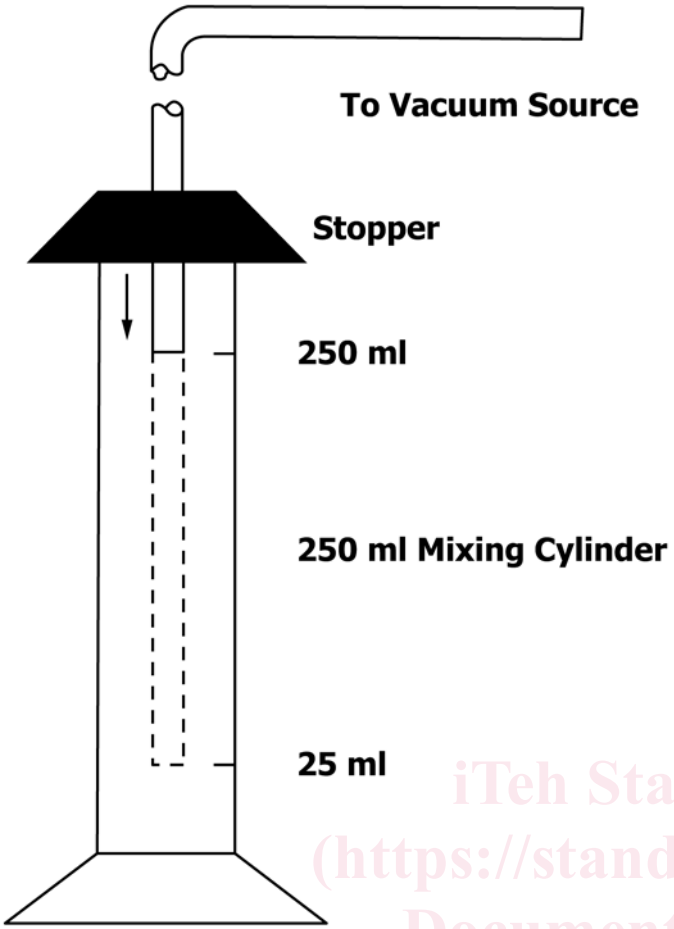

FIG. 1 Vacuum Apparatus

5.11 *Centrifuge,* any centrifuge capable of holding a 50 mL or larger tube and maintaining a minimum speed of 1500 rpm.

5.12 *Centrifuge Tube,* plastic or glass, 50 mL or larger.

6. Reagents (Test Water)

6.1 *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.[3]

6.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water, Type IV, as defined by Specification D1193.

NOTE 1—Type IV grade reagent water may be prepared by distillation, ion exchange, reverse osmosis, electrodialysis, or a combination thereof.

6.3 *Synthetic Hard Water Stock*—Transfer 12.14 g of anhydrous calcium chloride ($CaCl_2$) and 5.55 g of magnesium chloride hexahydrate ($MgCl_2 \cdot 6H_2O$) to a 1000 mL volumetric flask. Dissolve the reagents with approximately 750 mL of water and equilibrate to 20 °C. Dilute the solution to 1000 mL total volume with water at 20 °C, stopper the flask and mix the solution thoroughly. This mixture is equivalent to 13 680 ppm as calcium carbonate ($CaCO_3$) and is based on a compositional ratio of 4:1 calcium carbonate to magnesium carbonate.

6.3.1 *Soft Water*—Equivalent to a total hardness of 34.2 ppm as calcium carbonate ($CaCO_3$). Transfer 2.50 mL of synthetic hard water stock by pipet to a 1000 mL volumetric flask. Dilute to the 1000 mL mark with water at 20 °C. Mix solution thoroughly.

NOTE 2—It is recommended that total hardness be checked in accordance with Test Method MT-73, CIPAC 1, EDTA titration.[4] An alternate method is provided in Test Method D1126 where the value is represented as $CaCO_3$. A value within ±% of the nominal hardness value is acceptable.

6.3.2 *Hard Water*—Equivalent to a total hardness of 342 ppm as calcium carbonate ($CaCO_3$). Transfer 25 mL of synthetic hard water stock by buret to a 1000 mL volumetric flask. Dilute to the 1000 mL mark with water at 20 °C. Mix this solution thoroughly (see Note 2).

6.3.3 *Extra-Hard Water*—Equivalent to a total hardness of 1000 ppm as calcium carbonate ($CaCO_3$). Transfer 73.1 mL of synthetic hard water stock by buret to a 1000 mL volumetric flask. Dilute to the 1000 mL mark with water at 20 °C. Mix this solution thoroughly (see Note 2).

6.3.4 *Other Test Waters*—Other synthetic waters can be prepared by using the following calculation:

$$\text{Desired Water Hardness} \div 13.680 = \quad (1)$$

[millilitres of synthetic hard water stock at 20 °C to be diluted volumetrically to 1000 mL with water at 20 °C]

6.4 *Other Carriers*—Carriers other than water may be used when appropriate.

7. Safety Precautions

7.1 Before testing, read the precautionary statements on the product label or the Material Safety Data Sheet (MSDS), or both. Take proper precautions to prevent skin contact and inhalation of the fines or vapors, or both. Take care to prevent contamination of the surrounding area. Always wear the appropriate safety equipment and, where indicated, wear respiratory devices approved by the National Institute of Occupational Safety and Health (NIOSH) for the product being tested.

8. Procedure

8.1 *Option A (Evaporation):*

8.1.1 Each sample should be run in duplicate.

8.1.2 Weigh a 4 g sample into a weighing dish. Record the sample weight (W_1) to an accuracy of ±0.01g.

8.1.3 Transfer the sample into a 100 mL beaker containing 50 mL of test water and stir with a magnetic stirrer at high speed for two min.

[3] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.

[4] "Analysis of Technical and Formulated Pesticides," *CIPAC Handbook*, Vol 1, compiled by Ashworth, R. de B., Henriet, J., Lovett, J. F., Collaborative International Pesticide Analytical Council Ltd., Great Britain, 1970.

8.1.4 Quantitatively transfer the slurry from 8.1.3 into a 250 mL mixing cylinder using up to 100 mL of additional test water to rinse the beaker.

8.1.5 Fill the mixing cylinder to the 250 mL mark with test water.

8.1.6 Stopper and invert the mixing cylinder 15 complete cycles, 2 s per cycle.

8.1.7 Let the mixing cylinder stand for 30 min at 25 °C. Record ambient temperature. (See Note 3.)

NOTE 3—Other temperatures may be examined as defined by actual field use applications.

8.1.8 Remove the top 225 mL from the mixing cylinder using the vacuum apparatus in accordance with 5.8. Ensure that the tip of the suction tube remains slightly below the liquid surface while removing the 225 mL. Take care not to disturb the bottom 25 mL layer.

8.1.9 Record the tare weight of the weighing dish to an accuracy of ±0.01 g.

8.1.10 Gently swirl the remaining suspension to loosen the hard-packed material. Transfer quantitatively to the weighing dish.

8.1.11 Dry the weighing dish containing residue from 8.1.10 in a 50 °C gravity oven to a constant weight.

8.1.12 Weigh the dish from 8.1.11 to an accuracy of ±0.01 g and subtract the tare weight to determine the dried residue weight (W_2).

8.2 *Option B (Centrifuge/Evaporation):*

8.2.1 Each sample should be run in duplicate.

8.2.2 Weigh a 4 g sample into a weighing dish. Record the sample weight (W_1) to an accuracy of ±0.01 g.

8.2.3 Transfer the sample into a 100 mL beaker containing 50 mL of test water and stir with a magnetic stirrer at high speed for 2 min.

8.2.4 Quantitatively transfer the slurry from 8.2.3 into a 250 mL mixing cylinder using up to 100 mL of additional test water to rinse the beaker.

8.2.5 Fill the mixing cylinder to the 250 mL mark with test water.

8.2.6 Stopper and invert the mixing cylinder 15 complete cycles, 2 s per cycle.

8.2.7 Let the mixing cylinder stand for 30 min at 25°C (see Note 3). Record ambient temperature.

8.2.8 Remove the top 225 mL from the mixing cylinder using the vacuum apparatus in accordance with 5.8. Ensure that the tip of the suction tube remains slightly below the liquid surface while removing the 225 mL. Take care not to disturb the bottom 25 mL layer.

8.2.9 Record the tare weight of the centrifuge tube to an accuracy of ±0.01 g.

8.2.10 Gently swirl the remaining suspension to loosen the hard-packed material. Transfer quantitatively to the centrifuge tube.

8.2.11 Centrifuge the sample at a minimum speed of 1500 rpm for 15 min or until the solution remaining above the packed sediment appears free of any suspended particles. Gently decant the liquid, leaving as little as possible above the sediment.

8.2.12 Dry the centrifuge tube with the sediment from 8.2.11 in a 50 °C gravity oven to a constant weight.

8.2.13 Weigh the centrifuge tube from 8.2.12 to an accuracy of ± 0.01 g and subtract the tare weight to determine the dried residue weight (W_2).

9. Disposal of Sample

9.1 After testing, store all materials in a safe manner and dispose of used material in accordance with product label directions or MSDS, or both.

10. Calculation

10.1 *Option A or B Calculations:*

10.1.1 Calculate % suspensibility of the insoluble material as follows:

$$\% \text{ suspensibility} = \frac{10}{9} \times \frac{(W_1 - W_2) \times 100}{(W_1)} \quad (2)$$

where:

W_1 = sample weight, and
W_2 = dried residue weight.

11. Report

11.1 Report percent suspensibility, ambient temperature, carrier, and option used.

12. Precision and Bias

12.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same analyst should be considered suspect if they differ by more than 3 % absolute.

12.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by analysts in different laboratories using the same option and sample weight should be considered suspect if they differ by more than 3 % absolute.

12.3 *Bias*—This test method has no bias because the value of suspensibility is defined only in terms of this test.

13. Keywords

13.1 dispersion; dry flowable; dry flowable test methods; percent suspensibility; suspension; water dispersible granules (WG) (WDG); water dispersible granules test methods; wettable granules test methods; wettable powders (WP); wettable powders test methods