Designation: E2316 – 14

Standard Test Method for Determination of Particles Resulting from the Attrition of Granular Pesticides[1]

This standard is issued under the fixed designation E2316; 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 amount and particle size distribution curve of particles with diameter 106 micrometers or smaller resulting from the attrition of granular pesticides.

1.2 The values stated in SI units are to be regarded as 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.* For specific hazard statement, see Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E725 Test Method for Sampling Granular Carriers and Granular Pesticides

2.2 *CIPAC Standard:*

CIPAC Test Method MT 187[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *fines*—a synonym for particles with diameter of 106 micrometers or smaller.

3.1.2 *micron and μm*— synonyms for micrometer.

4. Summary of Test Method

4.1 The initial weight of a test sample of granular pesticide is determined. The sample is then air jet sieved using 106 micron openings to remove the inherent fines. The fines-free granules are then combined with glass beads in a glass jar, the lid is placed on the jar, and the jar is placed on a roller system with a drive bed capable of rotating the jar at a known rpm. After rolling for a specified time period, the jar is removed from the rollers and the contents of the jar are poured through a sieve sized to remove the glass beads. The sample minus the glass beads is again air jet sieved using 106 micron openings to remove the fines attrited during the rolling of the jar. The total of particles smaller than 106 microns for the test sample is the inherent fines plus the attrited fines. The particle size distribution curve of the combined inherent and attrited fines is determined by laser light diffraction using CIPAC Test Method MT 187.

5. Significance and Use

5.1 This test method is designed specifically for granular pesticide formulations.

5.2 This test method helps provide information on health hazards likely to arise from exposures by the inhalation route. It can be of use in selecting dose levels for chronic studies and for establishing safety criteria for human exposure.

5.3 The amount of fines determined by this method is a measure of potential inhalation and respiration toxicity because the hazards of inhaled solid substances are influenced by physical factors such as particle size.

6. Apparatus

6.1 *Roller System,* two or more rollers with a drive bed, capable of rotating the specified glass jar at 75 ± 15 rpm.

6.2 *Glass Jar,* with lid, capacity ~500 mL, outer diameter ~8 cm, height ~15 cm.

[1] This test method is under the jurisdiction of ASTM Committee E35 on Pesticides, Antimicrobials, and Alternative Control Agentsand 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.

[3] The size distribution of the particles with sieve diameter less than 106 μm is determined by laser light diffraction using CIPAC Test Method MT 187.

6.3 *Glass Beads,* diameter 4.0 ± 0.2 mm, bulk density ~1.5 g/cc.

6.4 *Micron Air Jet Sieve,* with GAZ 125 cyclone fines collector or *Alpine Air Jet Sieve*, with GAZ 125 cyclone fines collector,[4] or equivalent.

6.5 *Sieves,* U.S. standard series conforming to Specification E11, diameter 8 in. (203 mm), height 2 in. (51 mm): 10-mesh (2 mm openings), 140-mesh (106 µm openings).

NOTE 1—The amount of particles with sieve diameter 106 µm or smaller is determined by sieving.

6.6 *Balance,* sensitivity of 0.01 g.

6.7 *Hygrometer,* minimum range of 25 to 95 % relative humidity.

6.8 Apparatus specified in CIPAC Test Method MT 187.

7. Reagents and Materials

7.1 Granular pesticide to be tested.

7.2 Reagents and materials specified in CIPAC Test Method MT 187.

8. Hazards

8.1 Before testing, read the precautionary statements on the product label and the Material Safety Data Sheet (MSDS). Take proper precautions to prevent skin contact and inhalation of the fines. 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.

8.2 Storage, handling, and disposal of test pesticides should be done with consideration for health and environmental safety, and in accordance with federal, state, and local regulations.

9. Sampling, Test Specimens, and Test Units

9.1 See Test Method E725 for proper sampling practices and procedures to reduce a gross sample to a representative, suitable size for this test method.

10. Preparation of Apparatus

10.1 For all apparatus, see manufacturers' instructions for proper calibration, operation, and maintenance.

11. Procedure

11.1 The test sample should be 15 g, nominal. The exact amount of each sample should be recorded for use in necessary calculations.

11.2 Weigh the test sample to the nearest 0.01 g (15 g nominal) and record as W (granules plus inherent inhalable particles).

11.3 Weigh the cyclone collection jar to the nearest 0.01 g and record as J1. Attach the jar to the apparatus.

11.4 Measure and record the relative humidity of the air that will come in contact with the granules. In the absence of humidity controlled air, the air of concern will be the air of the room in which the tests are performed.

11.5 Place the 106 µm sieve in the air jet manifold of the Micron Air Jet Sieve or Alpine Air Jet Sieve. Gently transfer the entire test sample to the sieve and put the sieve cover in place. Set the timer for 3 min. Start the vacuum and adjust the vacuum gauge to read 7 to 9 in. of water (1.7 to 2.4 kPa). During the sieving period, material adhering to the sieve cover or sieve sides may be loosened by use of tapping or by use of trace amounts of anti-static compound.

11.6 After sieving, determine to the nearest 0.01 g the weight of granules retained on the sieve and record the weight as R1 (granules minus inherent fines).

11.7 Determine to the nearest 0.01 g the weight of the cyclone collection jar plus inherent fines and record the weight as J2. Leave the fines in the jar. Reattach the jar to the apparatus.

11.8 Calculate the weight per cent, to the nearest 0.1 %, of the inherent fines (IF) as determined by the weight of the fines collected in the jar. Use the following formula:

$$\% \; IF = [(J2 - J1) \div W] \times 100 \quad (1)$$

11.9 Transfer all the granules (R1) from the 106 µm sieve into the 500 mL jar. Add an equal weight (± 1 g) of glass beads. Place the lid on the jar.

11.10 Place the jar on the roller. Measure the rotational speed of the jar, which must be 75 ± 15 rpm. Calculate the time period needed for 4500 revolutions, using the following formula:

$$\text{Time period} = 4500 \div \text{measured rpm of the jar} \quad (2)$$

11.11 Rotate the jar for 4500 ± 10 revolutions. Record the time period and rpm with the data. With the rpm held constant, this time period should be used for all subsequent tests.

11.12 Remove the jar from the roller.

11.13 Again, place the 106 µm sieve in the air jet manifold of the Air Jet Sieve. Place the 2 mm sieve on top of the 106 µm sieve.

11.14 Gently pour the contents of the jar onto the 2 mm sieve (see Note 2). Be sure any material adhering to the jar wall is transferred from the jar. Be sure any material adhering to the glass beads retained on the 2 mm sieve is loosened and passes onto the 106 µm sieve below.

NOTE 2—A sieve with openings that will retain the glass beads but allow the granules to pass through must be used, and a reasonable size for most granular pesticides is 2 mm. If needed, a sieve with openings larger than 2 mm can be used.

11.15 Remove the 2 mm sieve and place the sieve cover on the 106 µm sieve. Set the timer for 3 min. Start the vacuum and adjust the vacuum gauge to read 7 to 9 in. of water (1.7 to 2.4 kPa). Material adhering to the sieve cover or sieve sides may be loosened by tapping or by use of trace amounts of anti-static compound.

[4] The Micron Air Jet Sieve and the Alpine Air Jet Sieve use the same principle of design and working components. Both are products of Hosokawa Micron International of New York. The GAZ 125 cyclone fines collector is an accessory.

11.16 Determine to the nearest 0.01 g the weight of the cyclone collection jar containing the combined inherent fines plus attrited fines and record the weight as J3.

11.17 Calculate the weight percent, to the nearest 0.1 %, of the total fines (TF) as determined by the weight of the fines in the jar. Use the following formula:

$$\% \; TF = \left[(J3 - J1) \div W \right] \times 100 \qquad (3)$$

11.18 Save the total fines (inherent plus attrited) from the cyclone collection jar for particle size distribution analysis by laser light diffraction using CIPAC Test Method MT 187.[3]

11.19 Clean and dry the sieves, glass beads, roller jar, and cyclone collection jar.

11.20 Repeat the test, 11.1 through 11.19, two more times using fresh samples.

11.21 Combine the collection of (3) total fines.

11.22 Analyze the combined total fines from 11.21 for particle size distribution by CIPAC Test Method MT 187.

12. Report

12.1 Report the average of three (3) test sample results. Table 1 is a guide for reporting.

12.1.1 Report the particle size results of the combined total fines as follows:

12.1.1.1 Particle size: d (10%), d (50%) and d (90%).

12.1.1.2 Particle size distribution printout.

13. Precision and Bias

13.1 When the set of three test results yields an average of 0.9 to 1.1 % fines, any two of the three must not differ more than 25 % from each other. Below this level of fines, the allowable variation increases significantly, while above this level it decreases significantly. Limits for these have not been assigned.

TABLE 1 Weight % Particles with Diameter 106 Microns or Smaller (Fines)

Sample	Sample Weight, g	Inherent Fines, Wt %	Attrited Fines, Wt %	Total Fines, Wt %
1				
2				
3				
Average				

13.2 This test method does not contain pass/fail limits. They must be set by the test method user in accordance with the purposes for using the test.

13.3 Increasing the size of the test sample to more than the nominal 15 g specified will increase the variability in the test results.

13.4 Particle size distribution analysis by light diffraction requires a minimum weight of fines which, in most cases, will be 0.3 g.

14. Keywords

14.1 attrition; exposure; granular pesticide; health hazard; inhalation; inhalation particles; inhalation toxicity; particle size distribution