Designation: E 1116 – 98

Standard Test Method for Emulsification Characteristics of Pesticide Emulsifiable Concentrates[1]

This standard is issued under the fixed designation E 1116; 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 describes a general procedure for the determination of emulsification spontaneity and the emulsion stability characteristics of pesticide emulsifiable concentrates when diluted with water.

1.2 Proper safety and hygiene precautions must be taken when working with pesticide formulations to prevent skin or eye contact, vapor inhalation, and environmental contamination. Read and follow all handling instructions for the specific formulation and conduct the test in accordance with good laboratory practice.

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:*

D1126 Test Methods for Hardness in Water[2]

D1193 Specification for Reagent Water[2]

3. Terminology

3.1 *Definitions*:

3.1.1 *emulsification spontaneity*—the rapid formation of an emulsion in the test water from agitation provided only by the gravity addition of the product. For products of density greater than the water used, an excellent spontaneity rating is assigned when the emulsion bloom (billowing) extends downward to near the bottom of the water, with no visible oil or cream droplets reaching the bottom of the test cylinder. For products of density less than the water used, a rating of excellent is given if bloom occurs near the top of the water and no free oil is present. Spontaneity descriptions between excellent and nil (no emulsion formed, only free oil) are assigned very-good, fair, and poor on a subjective basis.

3.1.2 *emulsion quality*—a subjective evaluation of the emulsion appearance. A rating of excellent (homogeneous), very good, good, fair, and poor (nonhomogeneous) is assigned.

3.1.3 *Discussion*—Cream and oil separation may coexist. Normally, oil is located at either the extreme top or bottom of the liquid with cream between it and the rest of the emulsion. On rare occasions, separation occurs at both top and bottom of the liquid (because of partition and solubility properties) and care must be taken to so note and record.

3.1.4 *Separation*:

3.1.4.1 *separation, cream*—a discrete, opaque layer of concentrated emulsion occurring at either the top or the bottom of the liquid.

3.1.4.2 *separation, oil*—a discrete layer of nonemulsified liquid occurring at either the top or the bottom of the liquid.

4. Summary of Test Method

4.1 In this test method, emulsifiable pesticide concentrates are added to water of a given hardness and at a specified temperature to form an oil-in-water emulsion. Performance of the formulation is measured in terms of emulsion spontaneity, emulsion stability under static conditions, and re-emulsification of the coalesced phase.

5. Significance and Use

5.1 This test method provides a guide for evaluating emulsification characteristics of pesticide emulsifiable concentrates. It defines the stability of emulsified particles in water. Although not absolute, the test method is a measure of expected emulsion stability in agricultural application equipment.

6. Apparatus

6.1 *Analytical Balance*, accurate to 0.01 g.

6.2 *Burets*, 50 and 100-mL capacity.

6.3 *Graduated Cylinders*, flat bottom, 100-mL capacity, glass stoppered, volume divisions from 1 to 100 mL in 1-mL increments, having an overall length of 300 ± 15 mm and a head space between 50 and 75 mm.[3]

6.4 *Pipets*, graduated serological, various delivery volumes.

6.5 *Pipet Filler*, bulb-type or equivalent.

6.6 *Thermometer*, graduated in 1°C increments and having a minimum range from 0 to 50°C.

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[1] This test method is under the jurisdiction of ASTM Committee E-35 on Pesticides and is the direct responsibility of Subcommittee E35.22 on Pesticide Formulation and Application Systems.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] The glass cylinder available from Kontes Glass Co., Spruce St., Vineland, NJ 08360, Model K-482500 has been found suitable. Equivalent cylinder from other suppliers may be used.

6.7 *Volumetric Flasks*, 1000-mL capacity calibrated at 20°C.

6.8 *Constant Temperature (±1°C) Device*, of minimum vibration for housing graduated cylinders. This may be a controlled room, incubator, or a water bath of sufficient depth to allow immersion of graduated cylinders to above the 100-mL mark.

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.[4]

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water, Type IV, as defined by Specification D 1193.

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

7.3 *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.

7.3.1 *Soft Water*, equivalent to a total hardness of 35 ppm as calcium carbonate ($CaCO_3$). Transfer 2.6 mL of synthetic hard water stock by pipet to a 1000-mL volumetric flask and dilute to volume with water at 20°C. Mix this solution thoroughly.

NOTE 2—It is recommended that total hardness as $CaCO_3$ be checked in accordance with Test Method MT-73, CIPAC F, EDTA titration.[5] An alternate method is provided in Test Method D 1126 where the value is represented as $CaCO_3$. A value within ±5 % of the nominal hardness value is acceptable.

7.3.2 *Hard Water*, equivalent to a total hardness of 342 ppm as calcium carbonate ($CaCO_3$). Transfer 26.0 mL of synthetic hard water stock by buret to a 1000-mL volumetric flask and dilute to volume with water at 20°C. Mix this solution thoroughly (Note 2).

7.3.3 *Extra-hard Water*, equivalent to a total hardness of 1000 ppm as calcium carbonate ($CaCO_3$). Transfer 73.0 mL of synthetic hard water stock by buret to a 1000-mL volumetric flask and dilute to volume with water at 20°C. Mix this solution thoroughly (Note 2).

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

Desired Water Hardness + 13.680 =

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

NOTE 3—Soft water, hard water, and extra-hard water may be prepared according to the World Health Organization (WHO) method WHO-M-13.R2. (approved Nov. 14, 1983).

8. Procedure

8.1 Transfer into three separate 100-mL graduated cylinders, 100 mL of 25°C soft water, hard water, and extra-hard water less the amount specified in 8.2.

NOTE 4—Other temperatures may be examined as defined by actual field use applications.

NOTE 5—Other water hardnesses as described in 7.3.4 may be examined as defined by actual field use applications.

8.2 Add a specified amount, in accordance with product label directions, of the test emulsifiable concentrate at 25°C (Note 4) to each cylinder. Deliver the emulsifiable concentrate using a pipet from a height of 50 ± 5 mm above the water surface to the center of the water surface. Delivery rate should be approximately 1 mL/s. Observe and record emulsification spontaneity.

8.3 Stopper the graduated cylinders (do not grease) then invert and right each cylinder ten times at the rate of about one complete cycle every 2 s. Record the initial emulsion quality.

8.4 Maintain each cylinder at 25°C or at a constant temperature (Note 4) and allow the cylinders to remain undisturbed. At 15 min, 30 min, 1 h, 2 h, and 24 h, observe and record the emulsion quality and the millilitres of cream or oil separation, or both.

8.5 After the 24-h observation, invert and right each cylinder ten times as outlined in 8.3. Return the cylinders to the 25°C constant temperature device. At 30 min observe and record the emulsion quality and the millilitres of cream or oil separation, or both.

8.6 Discard the emulsions in a safe manner.

9. Precision and Bias

9.1 Due to the variability of cream separation, oil separation, and subjectiveness of emulsion quality for the particular pesticide, no measurement of precision and bias can be made for this test method.

10. Keywords

10.1 bloom; cream; emulsifiable concentrate; emulsification; pesticide; spontaneity

[4] "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 U.K. Chemicals," BDH Ltd., Poole, Dorset, and the "United States Pharmacopoeia."

[5] "Hardness of Water," *Physico-Chemical Methods for Technical and Formulation Pesticides*, Vol F, editors, Dobret W., Martin A., Collaborative International Pesticide Analytical Council Ltd., 1995.

E 1116