



Standard Test Method for Measuring Repellency, Retention, and Penetration of Liquid Pesticide Formulation Through Protective Clothing Materials¹

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INTRODUCTION

The health and safety of agricultural workers involved in the mixing, loading and application of liquid pesticides can be affected by dermal exposure to liquid pesticide formulations. Use of protective clothing can assist in minimizing the danger of contact with potentially harmful pesticides. Nonporous materials that provide excellent protection to the user are usually not suitable for many agricultural environments in which there is a potential for heat stress. Therefore, garments made of porous materials that can provide a balance between risk from pesticide exposure and user comfort can also be used as personal protective equipment (PPE) for agricultural workers. The movement of liquid pesticides through these materials is primarily a result of penetration through spaces between fibers and interstices between yarns. As these materials provide protection either by repelling or retaining liquid pesticide, the measurement of these properties is also important. This test method is used to measure repellency, retention, and penetration of liquid pesticides through protective clothing materials.

The degree of contamination depends on numerous factors such as type of exposure, application technique, and pesticide formulation. Worker exposure to liquid pesticides can range from low exposure caused by spray drift to high exposure as in the case of an accidental spill while mixing or handling of concentrates. As the level of exposure can vary considerably, this test method is designed to rate relative performance of PPE materials at two levels of contamination.

1. Scope

1.1 This test method measures repellency, retention and penetration of a known volume of liquid pesticide when applied to protective clothing material. No external hydrostatic or mechanical pressure is applied to the test specimen during or after the application of the liquid pesticide.

1.2 This test method is designed to measure performance of protective clothing materials at two levels of contamination. Low level of contamination is achieved by applying 0.1-mL liquid formulation and high level by applying 0.2 mL.

1.3 This test method does not measure resistance to permeation or degradation.

1.4 This test method is suitable for field strength pesticide formulations. This test method may not be suitable for testing protective clothing materials against volatile pesticides.

1.5 The values stated in SI units are to be regarded as the standard.

1.6 *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:*²
D123 Terminology Relating to Textiles

¹ This test method is under the jurisdiction of ASTM Committee F23 on Personal Protective Clothing and Equipment and is the direct responsibility of Subcommittee F23.30 on Chemicals.

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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.

E105 Practice for Probability Sampling of Materials
F1494 Terminology Relating to Protective Clothing

3. Terminology

3.1 Definitions:

3.1.1 *analytical technique, n*—a procedure whereby the concentration of the test chemical in a collection medium is quantitatively determined.

3.1.1.1 *Discussion*—These techniques are often specific to individual chemical and collection medium combinations. Applicable techniques include, but are not limited to, flame ionization, photo ionization, electro-chemical, ultraviolet and infrared spectrophotometry, gas and liquid chromatography, colorimetry, length-of-stain detector tubes, and radionuclide tagging/detection counting.

3.1.2 *coated fabric, n*—flexible material composed of a textile fabric and an adherent polymeric or other material applied to one or both surfaces.

3.1.3 *degradation, n*—deleterious change in one or more properties of a material.

3.1.4 *penetration, n*—for chemical protective clothing, the movement of substances through voids in protective clothing materials or items on a non-molecular level.

3.1.4.1 *Discussion*—Voids include gaps, pores, holes, and imperfections in closures, seams, interfaces and protective clothing materials. Penetration does not require a change of state; solid chemicals move through voids in the materials as solids, liquids as liquids and gases as gases. Penetration is a distinctly different mechanism from permeation.

3.1.5 *permeation, n*—for chemical protective clothing, the movement of chemicals, as molecules, through protective clothing materials by the processes of (1) absorption of the chemical into the contact surface of the material, (2) diffusion of the absorbed molecules throughout the material, and (3) desorption of the chemical from the opposite surface of the material.

3.1.5.1 *Discussion*—Permeation is a distinctly different mechanism from penetration.

3.1.6 *pesticide retention, n*—amount of pesticide active ingredient retained in the protective clothing material.

3.1.7 *protective clothing, n*—an item of clothing that is specifically designed and constructed for the intended purpose of isolating all or part of the body from a potential hazard; or, isolating the external environment from contamination by the wearer of the clothing.

3.1.8 *repellency, n*—characteristic to resist wetting and penetration by a liquid.

3.1.9 *test chemical, n*—solid, liquid, gas, or mixture thereof, used to evaluate the performance of a protective clothing material.

3.1.9.1 *Discussion*—For the purpose of this test method, test chemical is limited to liquid chemicals that are a mixture of raw materials, including, but not limited to, active ingredients, inert ingredients, and a base solvent used in pesticide formulation. Additional ingredients could include emulsifiers and surfactants. Solvents used in the formulation could be water,

isopropyl alcohol, or petroleum distillate. Solid materials (powders, granules, and so forth) may be dissolved or emulsified to form a liquid or suspension. These formulations may be ready to use or concentrates that require dilution to field strength. In some ultra-low volume applications, concentrated oil-based formulations are used without dilution; testing for this application is beyond the scope of this test method.

3.1.10 For other textile terminology see Terminology **D123**.

3.1.11 For other protective clothing terminology see Terminology **F1494**.

4. Summary of Test Method

4.1 A pipettor is used to apply liquid pesticide to the surface of the test assembly. The test assembly consists of single- or multiple-layer protective clothing material (test specimen) and an absorbent paper backed by polyethylene film (collector layer).

4.1.1 Another absorbent paper backed by polyethylene film is placed on the surface test specimen after a specified time to remove the remaining liquid.

4.1.2 The contaminated test specimen, collector layer, and paper used to remove liquid from the surface of the material are separated and extracted.

4.1.3 The extracts are analyzed quantitatively.

4.1.4 Data are used to calculate percent repellency, pesticide retention, and penetration.

5. Significance and Use

5.1 This test method can be used for laboratory screening of protective clothing material used to manufacture garments and accessories worn by pesticide workers.

5.2 This test method can be used for the development and evaluation of new protective clothing materials.

5.3 This test method can be used for the evaluation of protective clothing materials against new pesticide formulations.

6. Apparatus and Materials

6.1 Apparatus and materials for contamination of test specimen:

6.1.1 *Test Chemical*, to contaminate the test specimen.

NOTE 1—Diluted and concentrated formulations can be used with this test method.

6.1.2 *Pipettor*, with disposable pipet tip, mounted on a support stand, for pipetting 0.1 ± 0.002 mL of liquid for low contamination level and 0.2 ± 0.004 mL for high contamination level.

6.1.3 *Specimen Holder*, that consists of a base plate (100 by 100 mm) and a cover plate (100 by 100 mm with a 60 by 60 mm opening in the center). The specimen holder is made of polymethyl methacrylate (PMMA) (4-mm thickness).

6.1.4 *Timer*, accurate to 1 s.

6.1.5 *Absorbent Paper*, two 80 by 80-mm squares of Whatman Benchkote Plus³ Paper (absorbent paper backed by polyethylene film) per test specimen. One square is used to measure penetration, and the second to measure repellency.

NOTE 2—Substitutions are not recommended, as due to differences in sorptive properties, use of absorbent papers other than Benchkote Plus may affect the test results.

- 6.1.6 *Container*, to discard contaminated materials.
- 6.1.7 *Fume Hood*, with airflow control and a glass door

6.2 *Apparatus for Analysis Using Method A:*

- 6.2.1 *Balance*, accurate to 0.001 g.
- 6.2.2 *Tweezers*.
- 6.2.3 *Transparency Film*, a 100 by 100-mm square cut from clear transparency film or similar material made of plastic film.

6.3 *Apparatus for Analysis Using Method B*

- 6.3.1 *Solvent*, appropriate for extraction of pesticide.

NOTE 3—Selection of the solvent is dependent on the pesticide and the analytical method used. A minimum extraction efficiency of 95 % is required. Procedure to calculate extraction efficiency is given in 11.2.1. Solvent with high volatility may not be appropriate, as there may be evaporation loss during handling operations.

6.3.2 *Airtight Chemically Resistant Flasks/Bottles*, suitable for extraction of pesticides.

- 6.3.3 *Tweezers*.
- 6.3.4 *Timer*, to measure time in minutes.
- 6.3.5 50 ± 0.2 mL *Graduated Cylinder*, bottle-top dispenser or other apparatus for accurate measurement of solvent.
- 6.3.6 *Orbital Shaker*.
- 6.3.7 *Airtight Chemically Resistant Bottles*, for storage.

7. Test Specimen

7.1 Protective clothing material specimen may consist of a single layer or a composite of multiple layers that is representative of an actual protective clothing garment. Specimens with seams, closures, or other unions shall be cut such that the stitching is centered on the specimen. In each test the outer surface should be contaminated with the pesticide formulation.

7.1.1 Each protective clothing material specimen should measure 80 by 80 mm.

7.1.2 A minimum of three specimens shall be tested for each test material. Random sampling procedures described in Practice E105 should be used for the selection of specimen.

8. Selection of Analytical Technique

8.1 The procedure used to quantify the mass of test chemical/liquid in the test specimen and absorbent papers shall be determined before conducting the tests. The selection of procedure for analysis is based on the liquid test chemical selected.

8.1.1 Gravimetric analysis can be used if the liquid test chemical has (1) a low evaporation rate and (2) no filtration or selective retention of ingredients. Typically, pesticide formulations that are categorized as emulsifiable concentrates (rela-

tively small particle size) and liquid concentrates (water-based solution concentrate with no particles) meet the criteria.

8.1.2 Analytical techniques such as gas chromatography or high-pressure liquid chromatography can be used for formulations with an active ingredient. This requires extraction (in most cases) and analysis of the active ingredient.

8.1.3 Use Method A if the gravimetric method is used for analysis. Use Method B if the procedure requires extraction and analysis of active ingredient.

9. Preparation of Test Apparatus and Materials

9.1 *Calibration of the Pipettor*

9.1.1 *Calibration with Distilled Water*

9.1.1.1 Calibrate the pipettor by weighing 0.1 mL (0.2 mL for higher contamination level) of distilled water. Take ten readings. The values shall be within the 2 % tolerance limits.

9.1.2 *Calibration with Liquid Test Chemical*

9.1.2.1 The pipettor shall be calibrated by each operator before conducting the tests. Use the same tip to dispense the test aliquot (0.1 or 0.2 mL) and record the weight to the third decimal place (0.001). Take ten readings. Each value shall be within the 2 % tolerance limits. Calculate the mean of ten readings. The mean value shall be used as the value for total amount of the liquid test chemical applied for Method A in 10.2.

NOTE 4—Experience of the operator in pipetting the liquid test chemical according to the procedure provided by the manufacturer is crucial. Inexperience in aspirating and dispensing the liquid test chemical can result in errors. Electronic pipettors may reduce the error caused by operator experience.

NOTE 5—The viscosity of the liquids may affect the amount dispensed. Liquid buildup in the tip may occur for liquids that are more viscous. In case of buildup, use fresh tip for each application or change as required, based on the results of the ten consecutive readings taken in 9.1.2.

9.2 *Preparation of the Test Assembly*

9.2.1 Mount the pipettor on the support stand and place under the fume hood.

NOTE 6—If the height of the container with the liquid test chemical is greater than 25 mm, place the specimen holder on a raised platform so the test chemical can be aspirated with ease.

9.2.2 Place the collector layer with the absorbent side up on the base plate of the specimen holder. Then place the test specimen, outside face uppermost, followed by the cover plate. Place the specimen holder with the specimen and collector layer horizontally below the pipettor. Adjust the height of the pipettor to a distance of 30 ± 5 mm above specimen.

NOTE 7—A polymethyl methacrylate (PMMA) cover plate prevents the edges of the material from curling and also ensures contact between the layers.

9.2.3 Mark the position of the specimen holder on the stand or raised platform.

9.3 *Conditioning and Testing Conditions*

9.3.1 Unless otherwise specified, test specimens and absorbent paper shall be conditioned and tested at $(20 \pm 5^\circ\text{C})$ and 60 ± 10 % relative humidity for 24 h before testing.

10. Method A

10.1 *Contamination of Specimen*

³ Whatman Benchkote Plus absorbent paper is available through scientific products suppliers or from Whatman Company. Information on the paper and suppliers can be obtained from www.whatman.com

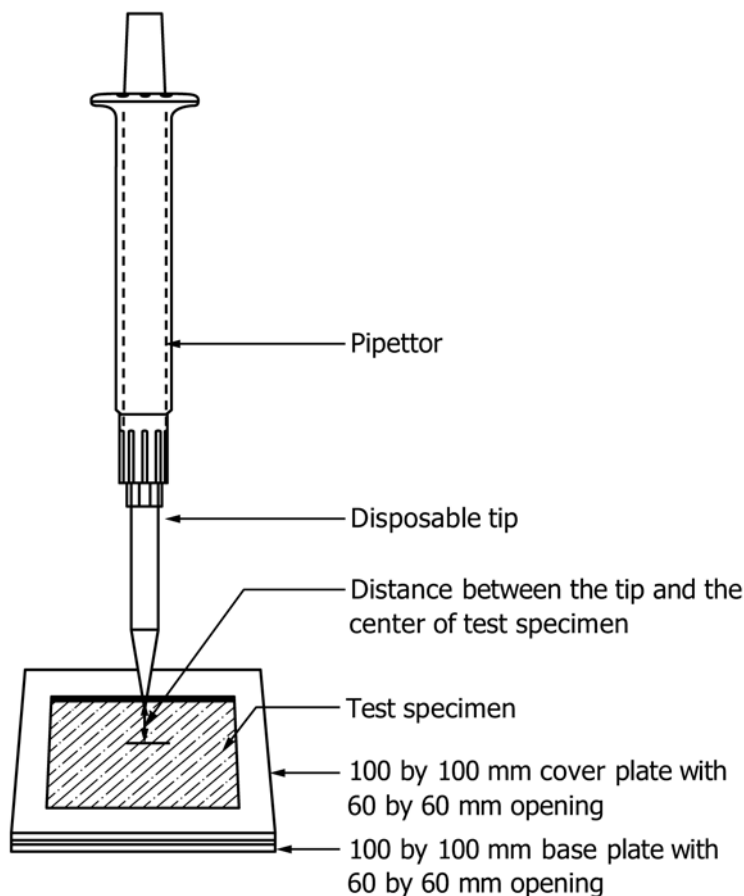


FIG. 1 Pipettor with a Disposable Pipet Tip, Mounted on a Support Stand

10.1.1 Weigh the test specimen and the two absorbent papers and record the readings in grams to the third decimal place (0.001).

NOTE 8—To avoid contamination, the pre-weighed test specimen and corresponding absorbent papers can be placed on aluminum foil.

10.1.2 Prepare the test assembly as given in Section 9.

NOTE 9—Corners of test specimens that have a tendency to curl shall be taped to the base plate. Use small pieces of tape so that contact between the test specimen and the collector layer below the test specimen is not changed.

10.1.3 Place the test assembly and the corresponding pre-weighed top absorbent layer next to the pipettor.

NOTE 10—If more than one specimen is being tested at one time, for efficiency, arrange the test assemblies and corresponding top absorbent layer next to the pipettor.

10.1.4 Shake liquid test chemical and carefully aspirate the test aliquot.

NOTE 11—A magnetic stirrer is recommended to stir liquids that may settle during application.

10.1.5 Position the raised platform with the test assembly below the pipettor as marked in 9.2.3. Carefully dispense the test aliquot within 5 s to the center of the test specimen, start the timer, and then place the transparency film over the cover plate.

NOTE 12—The transparency film is placed over the cover plate to reduce evaporation.

10.1.6 After 10 min, remove the cover plate of the specimen holder. Use tweezers to place the 80- by 80-mm polyethylene-backed absorbent paper on the surface of the test specimen with the absorbent side in contact with the test specimen. Place the cover plate back on the test assembly.

10.1.7 After an additional 2 min, use tweezers to separate the three layers. Handle the test specimens and absorbent papers from the edges.

10.1.8 Weigh each layer and record the readings to the third decimal place.

10.2 Computation of Results

10.2.1 Subtract the weight of each layer recorded in 10.1.1 from the corresponding weight recorded in 10.1.8 to calculate mass of liquid test chemical in each layer, M_r , M_{lr} , and M_p .

Where:

- M_r = mass (mg) of liquid test chemical in absorbent paper used to remove excess liquid pesticide after 10 min,
- M_{lr} = mass (mg) of liquid test chemical in the protective clothing material specimen, and
- M_p = mass (mg) of liquid test chemical in the collector layer.

10.2.2 Calculation of Mass Balance

10.2.2.1 Calculate the mass balance for each test by adding the respective M_r , M_{lr} , and M_p . For each specimen tested, the value shall range between 95 to 105 % of M_r , where M_i is the total amount of liquid test chemical applied determined in 9.1.2. Repeat the test if the mass balance is not within the range.

10.2.3 Calculation of Repellency, Retention, and Penetration

10.2.3.1 For each test specimen, calculate percentage of repellency, liquid retention, and penetration of the liquid test chemical using the following formulae:

$$\text{Percent Repellency (PR)} = M_r/M_i \times 100$$

$$\text{Percent Retention (PLR)} = M_{lr}/M_i \times 100$$

$$\text{Percent Penetration (PP)} = M_p/M_i \times 100$$

NOTE 13—Calculate evaporation loss for each test specimen given by: $100 - (PR + PLR + PP)$.

11. Method B

11.1 Verification of Amount of Active Ingredient in Liquid Test Chemical Applied

11.1.1 To verify the amount of active ingredient in the liquid test chemical, pipette test aliquots (three replications of the 0.1 or 0.2 mL) into 100 mL of the solvent. Shake well and analyze the liquid using the analytical technique selected in Section 8. The mean value will be used as total amount of active ingredient applied in 11.6.

11.2 Determination of Extraction Efficiency

11.2.1 Calculate the extraction efficiency using the solvent selected before testing the test specimen. To measure the extraction efficiency, contaminate and extract three test specimens using procedures in 11.4 and 11.5. Analyze the extracts using the analytical technique selected in Section 8. Calculate extraction efficiency using the formula given in 11.6.2. A minimum extraction efficiency of 95 % is required. Repeat the procedure to determine extraction efficiency with another solvent if the extraction efficiency is lower than 95 %.

11.3 Testing of Blanks

11.3.1 To ensure that there is no interference due to chemicals that may be present in the test material and absorbent paper, extract and analyze three 80 by 80-mm replicates of test specimen that have not been contaminated (blanks). Extract the blanks using procedures in 11.5. Analyze the extracts using the analytical technique selected in Section 8. The blanks shall be tested before testing the contaminated specimen.

11.4 Contamination of Specimens

11.4.1 Shake the liquid test chemical and carefully aspirate the test aliquot.

NOTE 14—Magnetic stirrer is recommended to stir liquids that may settle during application.

11.4.2 Position the raised platform with the test assembly below the pipettor as marked in 9.2.3.

11.4.3 Apply the liquid pesticide to the center of the test specimen and simultaneously start the timer. The time taken to dispense the liquid shall be within 5 s.

NOTE 15—Apply 0.1 ± 0.002 mL of liquid pesticide for lower contamination level and 0.2 ± 0.004 mL for higher contamination level.

11.4.4 After 10 min, remove the cover plate of the specimen holder. Use tweezers to place the 80 by 80-mm polyethylene-backed absorbent paper on the surface of the test specimen with the absorbent side in contact with the test specimen. Place the cover plate back on the test assembly.

11.4.5 After an additional 2 min, use tweezers to separate the three layers, handling the area that is not contaminated. Place each layer in separate flasks/bottles and secure the openings. Store the bottles in a freezer if the material is not extracted the same day.

11.5 Extraction of Liquid Test Chemicals

11.5.1 The following procedure will be used to extract pesticide from the test specimen and corresponding absorbent papers.

11.5.1.1 Add 50 ± 0.2 mL of the selected solvent to the flask/bottle using a graduated cylinder, bottle-top dispenser, or other suitable apparatus. Ensure that the sample is in contact with the solvent and the flask/bottle is secured tightly.

11.5.1.2 Set the orbital shaker speed to 200 rpm.

11.5.1.3 Place the bottles/flasks on the orbital shaker and set the timer for 30 ± 1 min.

11.5.1.4 Start the shaker and the timer and extract for 30 ± 1 min.

11.5.1.5 After 30 min, transfer the extract from the flask/bottle to the storage bottles. Tighten the caps of the opening of the storage bottles.

11.5.1.6 Extract the contaminated material in additional 50 mL of the solvent using procedure 11.5.1.1–11.5.1.5.

11.5.1.7 Combine the two aliquots. Do not add additional solvent to make up the volume to 100 mL. Store the extracts in a freezer for analysis.

NOTE 16—The total volume in the storage bottle will not be exactly 100 mL because of solvent remaining in the material. Since the concentration of the pesticide in the solvent retained in the material is at equilibrium with that collected as extract, it would be a representative sample.

11.5.1.8 Use good laboratory practices for the disposal of toxic substances and cleanup of laboratory glassware/apparatus.

11.6 Computational Results

11.6.1 Calculate percentage of repellency, pesticide retention, and penetration of the active ingredient using the following formulae:

$$\text{Percent Repellency (PR)} = M_r \times \frac{100}{M_i}$$

$$\text{Percent Retention (PPR)} = M_{pr} \times \frac{100}{M_i}$$

$$\text{Percent Penetration (PP)} = M_p \times \frac{100}{M_i}$$

M_r = mass (mg) of active ingredient extracted from the 80 by 80-mm absorbent paper used to remove excess liquid pesticide after 10 min,

M_{pr} = mass (mg) of active ingredient extracted from the protective clothing material specimen,

M_p = mass (mg) of active ingredient extracted from the collector layer, and

M_t = total mass (mg) of active ingredient applied to the test specimen.

NOTE 17—The amount verified by analytical technique is used for calculating M_t (see 11.1.1). Mass of pesticide for M_r , M_p , M_{pr} , and M_p is calculated by multiplying the concentration of active ingredient (mg/mL) by 100 mL (total volume of the solvent). Mass balance can be calculated by adding M_r , M_{pr} , and M_p . The total should account for a minimum of 95 % of M_t .

11.6.2 Extraction Efficiency (%) = $[M_r + M_{pr} + M_p / M_t] \times 100$.

12. Precision and Bias

12.1 *Precision and Bias*—The precision and bias for this test method is being determined and will be available within five years.

13. Keywords

13.1 contamination; extraction; liquid pesticide; liquid retention; penetration; repellency; textile materials

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