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Protective clothing — Protection against liquid chemicals — Measurement of repellency, retention, and penetration of liquid pesticide formulations through protective clothing materials

Vêtements de protection — Protection contre les produits chimiques liquides — Mesurage de la répulsion, de la rétention et de la pénétration des formulations de pesticides liquides à travers les matériaux des vêtements de protection

Reference number
ISO 22608:2004(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 22608 was prepared by Technical Committee ISO/TC 94, *Personal safety — Protective clothing and equipment*, Subcommittee SC 13, *Protective clothing*.

Introduction

The health and safety of 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 environments where 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 be used as personal protective equipment (PPE) for workers.

The movement of liquid pesticides through these materials is primarily due to penetration through spaces between fibres and interstices between yarns. As these materials provide protection either by repelling or retaining liquid pesticide, the measurement of these properties are also important. This test method is used to measure repellency, retention, and penetration of liquid pesticides through protective clothing materials.

Protective clothing — Protection against liquid chemicals — Measurement of repellency, retention, and penetration of liquid pesticide formulations through protective clothing materials

1 Scope

This International Standard specifies a test method to measure 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.

The degree of contamination depends on numerous factors such as type of exposure, application technique, and pesticide formulation. As the level of exposure can vary considerably, this method is designed to rate relative performance of personal protective equipment (PPE) 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.

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

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

This International Standard is applicable to the evaluation of materials that are new or those that have undergone treatment such as laundering, or simulated abrasion. Details of the treatment shall be reported. This test method can also be used to determine the resistance provided by protective clothing materials against penetration of new pesticide formulations.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2859-1:1999, *Sampling procedures for inspection by attributes — Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
analytical technique
procedure whereby the concentration of the test chemical in a collection medium is quantitatively determined

NOTE The procedure selected is based on the test liquid to be analysed. Applicable techniques include, but are not limited to, gas chromatography, high pressure liquid chromatography, gravimetric analysis, and radionuclide tagging/detection counting.

3.2
coated fabric
flexible material composed of a textile fabric and an adherent polymeric or other material applied to one or both surfaces

3.3
test liquid
mixture of raw materials, including but not limited to, active ingredients, inert ingredients and a base solvent used in pesticide formulation

NOTE Additional ingredients could include emulsifiers and surfactants. Solvents used in the formulation could be water, isopropyl alcohol, or petroleum distillate. Solid materials (powders, granules, etc.) 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.

3.4
penetration
flow of a chemical through closures, porous materials, seams, and holes or other imperfections in a protective clothing material on a non-molecular level

3.5
permeation
process by which a chemical moves through a protective clothing material on a molecular level

NOTE Permeation involves

a) sorption of molecules of the chemical into the contacted (outside) surface of a material,

b) diffusion of the sorbed molecules in the material, and

c) desorption of the molecules from the opposite (inside) surface of the material.

3.6
liquid retention
liquid retained in the protective clothing material under the conditions of this test

3.7
protective clothing material
any material or combination of materials used in an item of clothing for the purpose of isolating parts of the body from a potential hazard

NOTE For the purposes of this International Standard, protective clothing materials include those materials used in the construction of the suit or clothing that serve as the primary barrier for the wearer. Protective clothing materials do not include materials used in the construction of integral visors, gloves, and footwear.

3.8
liquid repellency
characteristic to resist wetting and penetration by a liquid

4 Principle

A test liquid is applied using a pipettor to the surface of the test assembly, which consists of single or multiple layer protective clothing material (test specimen) and an absorbent paper backed by polyethylene film (collector layer).

After a specified time, another absorbent paper backed by polyethylene film (top layer) is placed on the surface of the test specimen to remove the remaining liquid.

The top layer, contaminated test specimen and the collector layer are separated.

The amount of test liquid in each layer is measured either by gravimetric analysis (weighing) or by other appropriate analytical techniques.

Method A is a gravimetric method that measures the mass of the test liquid in each layer, whereas Method B is an analytical method that requires extraction of the test liquid and measures the mass of the active ingredient.

Data is obtained to calculate percent repellency, pesticide retention, and penetration.

5 Apparatus

5.1 Apparatus and materials for contamination of test specimen

5.1.1 Test liquid, to contaminate the test specimen.

Magnetic or other stirrer shall be used for liquids that may settle during application.

5.1.2 Pipettor, with disposable pipette 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 (see Figure 1).

Multidispensing shall not be used for test liquids that may settle during application.

NOTE Liquid viscosity, user experience and temperature can affect accuracy and precision. Additional information on selection of pipettor can be obtained from the manufacturer.

5.1.3 Test specimen holder, consisting of a base plate 100 mm × 100 mm (see Figure 2) and a cover plate 100 mm × 100 mm with a 60 mm × 60 mm opening in the centre (see Figure 3).

The test specimen holder is made of polymethyl methacrylate (4 mm thickness) or other suitable material. The mass of the cover plate shall be 30 g to 35 g.

5.1.4 Timer, accurate to nearest 1 s.

5.1.5 Absorbent paper, two 80 mm × 80 mm squares backed by polyethylene film per test specimen, one square of which is used to measure penetration, and the other to measure repellency, both having the following characteristics[1)]:

— base mass: 160 g/m^2

— thickness 590 µm at 53 kPa and 710 µm at 10 kPa

— typical water absorption value 75 mg/cm^2

Substitutions are not recommended, because due to differences in sorptive properties; use of absorbent papers other than that specified may affect the test results.

5.1.6 Container, to discard contaminated materials.

5.2 Apparatus for analysis using Method A

5.2.1 Balance, accurate to the nearest 0,001 g.

5.2.1 Tweezers.

5.3 Apparatus and materials for analysis using Method B

5.3.1 Solvent, appropriate for extraction of pesticide.

NOTE Selection of the solvent is dependent on the test liquid and the analytical method used. A minimum extraction efficiency of 95 % is required. Procedure to calculate extraction efficiency is given in 10.2. Solvent with high volatility may not be appropriate, as there may be evaporation loss during handling operations.

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1) Benchkote® Plus is the trade name of a polyethylene-backed absorbent paper manufactured by Whatman, available through scientific product suppliers or directly from Whatman (www.whatman.com). This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

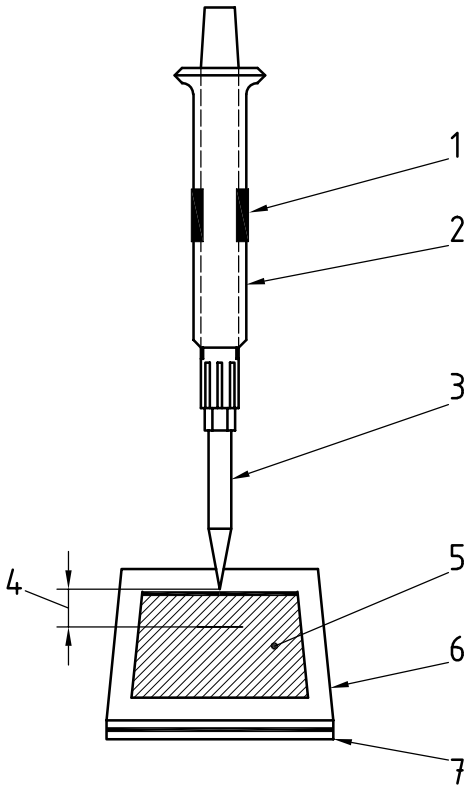
5.3.2 Bottles, airtight chemically resistant flasks or bottles suitable for extraction of pesticides. Airtight chemically resistant bottles are also used for storage.

5.3.3 Tweezers.

5.3.4 Timer, accurate to nearest 1 s.

5.3.5 Graduated cylinder, (50 ± 0,2) ml bottle-top dispenser or other apparatus for accurate measurement of solvent.

5.3.6 Orbital shaker, capable of (200 ± 20) r/min.

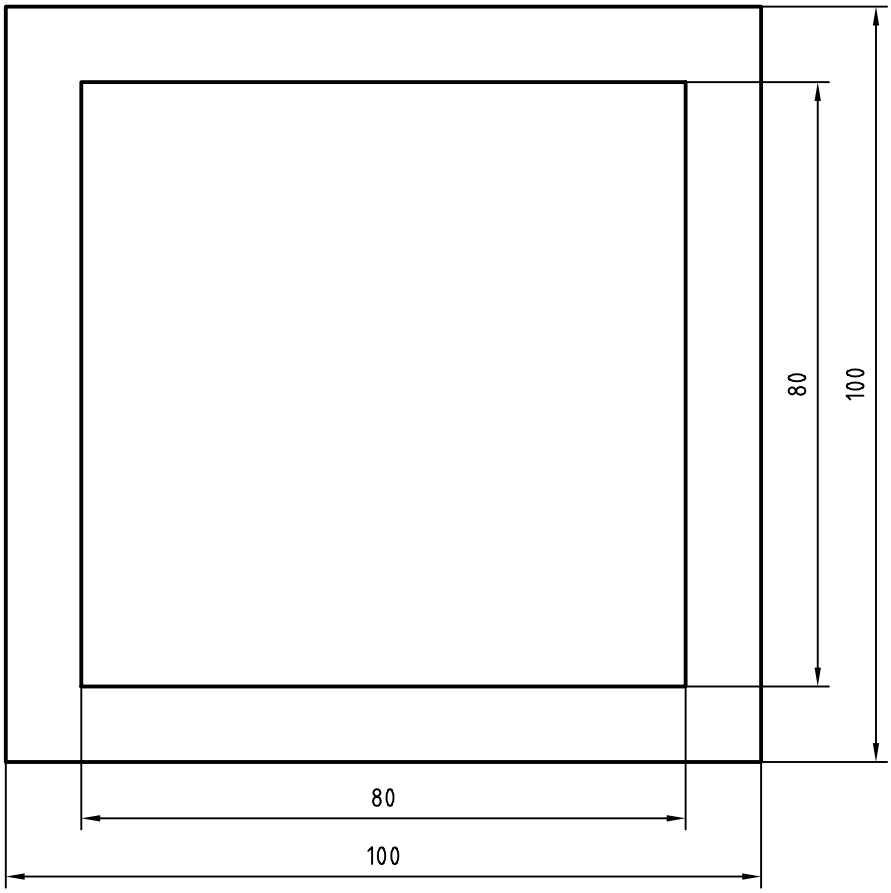


Key

1 clamp for support stand
2 pipettor
3 disposable tip
4 distance between the tip and the centre of test specimen
5 test specimen
6 100 mm × 100 mm cover plate with 60 mm × 60 mm opening
7 100 mm × 100 mm base plate

Figure 1 — Schematic diagram for placement of pipettor and test assembly

Dimensions in millimetres

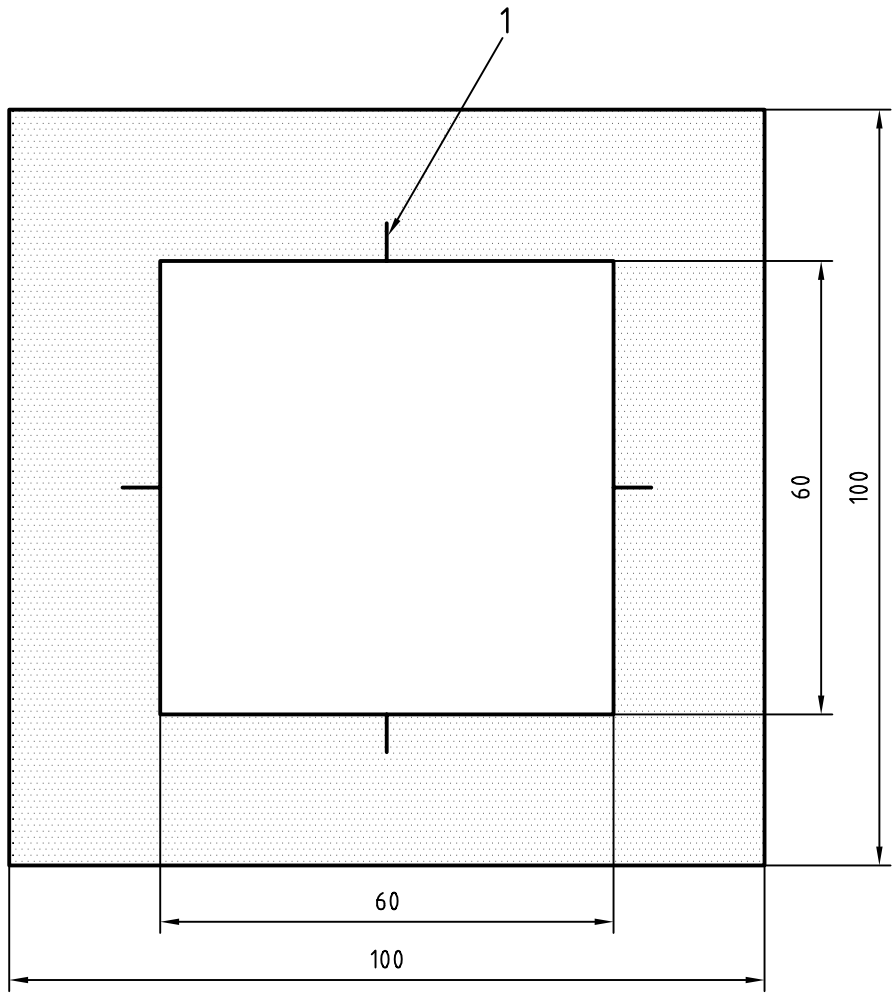


Characteristics

100 mm × 100 mm plate
80 mm × 80 mm marking inscribed on the plate
thickness 4 mm

Figure 2 — Plexiglas base plate

Dimensions in millimetres



Key

1 centre mark

Characteristics

100 mm × 100 mm with a 60 mm × 60 mm opening in the centre

thickness 4 mm

mass 30 g to 35 g

centre marks at 60 mm × 60 mm opening

Figure 3 — Plexiglas cover plate

6 Test specimens

Protective clothing material test specimens may consist of a single layer or a composite of multiple layers that is representative of an actual protective clothing garment. Test specimens may also include seams, closures or other unions used in the construction of the garment. In each test, the outer surface shall be contaminated with the pesticide formulation.

Each protective clothing material test specimen shall measure 80 mm × 80 mm. For Method A, carefully examine the samples and remove any loose fibres or yarns that may be protruding from the edges or sticking to the fabric surface.

A minimum of three test specimens shall be tested for each test material. Random sampling procedures described in ISO 2859-1 shall be used for the selection of test specimens.

7 Selection of analytical technique

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

Gravimetric analysis can be used if the test liquid has

a) low evaporation rate, and

b) no filtration or selective retention of ingredients.

Typically, pesticide formulations that are categorized as emulsifiable concentrates (relatively small particle size) and liquid concentrates (water-based solution concentrate with no particles) meet the criteria.

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

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

8 Preparation of test apparatus and materials

8.1 Calibration of the pipettor

8.1.1 Calibration with distilled water

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.

8.1.2 Calibration with test liquid

The pipettor shall be calibrated by each operator prior to conducting the tests. Use the same tip to dispense the test aliquot (0,1 ml or 0,2 ml) and record the mass to the nearest 0,001 g. Take ten readings. Each value shall be within the 2 % tolerance limits. Calculate the mean of 10 readings. The mean value shall be used as the value for total amount of test liquid, m_t, expressed in milligrams, applied in Method A in 9.2.

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

The viscosity of the test liquids may affect the amount dispensed. Liquid build-up in the tip may occur for liquids that are more viscous. In the case of build-up, use a fresh tip for each application or change as required, based on the results of the ten consecutive readings taken above.

8.2 Preparation of test assembly

Prepare the test assembly as follows.

a) Mount the pipettor on the support stand. Place under the fume hood, if the test liquid to be used for testing is hazardous. If the height of the container with the test liquid is greater than 2,5 cm, place the test specimen holder on a support jack or raised platform so the test liquid can be aspirated with ease.

b) Prepare the test assembly by placing the collector layer with the absorbent side up on the base plate of the test specimen holder. Then add the test specimen, outside face uppermost, followed by the cover plate.

c) Place the test assembly below the pipettor. Centre it below the tip and adjust the height of the pipettor to a distance of (30 ± 5) mm above test specimen (see Figure 1).

d) Mark the position of the test specimen holder on the stand or raised platform.

8.3 Conditioning of test specimen

Unless otherwise specified, condition the test specimen at (25 ± 5) °C and (60 ± 10) % relative humidity for 24 h prior to testing. The testing shall commence within 10 min of removing the test specimens from the conditioning atmosphere.

8.4 Testing temperature

Unless otherwise specified, all testing shall be conducted at the same conditions used for conditioning, (25 ± 5) °C and (60 ± 10) % relative humidity.

9 Method A

9.1 Contamination of test specimen

The contamination of the test specimen is determined as follows.

a) Weigh the test specimen and the two absorbent papers and record the readings to the nearest 0,001 g. To avoid contamination, the pre-weighed test specimen and corresponding absorbent papers can be placed on aluminium foil.

b) Prepare the test assembly as given in Clause 8. 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.

c) Place the test assembly and the corresponding pre-weighed top absorbent layer next to the pipettor. If more than one test specimen is being tested at one time, for efficiency, arrange the test assemblies and corresponding top absorbent layer next to the pipettor.

d) Shake test liquid and carefully aspirate the test aliquot. A magnetic stirrer is recommended to stir liquids that may settle during application.

e) Position the raised platform with the test assembly below the pipettor as marked in 8.2 d). Carefully dispense the test liquid to the centre of the test specimen and simultaneously start the timer. The time taken to dispense the liquid shall be within 5 s. Apply (0,1 ± 0,002) ml of test liquid for lower contamination level and (0,2 ± 0,004) ml for higher contamination level. A 100 mm × 100 mm transparency film may be used to cover the opening to reduce evaporation loss.

f) After 10 min, remove the cover plate of the test specimen holder. Use tweezers to place the 80 mm × 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.

g) After 2 min more, use tweezers to separate the three layers. Handle the test specimens and absorbent papers from the edges.

h) Weigh each layer and record the readings to the nearest 0,001 g.

9.2 Calculation

9.2.1 Determination of masses of test liquid in each layer

Subtract the mass of each layer recorded in 9.1 a) from the corresponding mass recorded in 9.1 h) to calculate mass of test liquid in each layer, m_{ap}, m_{pc}, m_{cl},

where

m_{ap} is the mass, expressed in milligrams, of test liquid in 80 mm × 80 mm absorbent paper used to remove excess liquid pesticide after 10 min;

m_{pc} is the mass, expressed in milligrams, of test liquid in the protective clothing material test specimen;

m_{cl} is the mass, expressed in milligrams, of test liquid in the collector layer.

9.2.2 Mass balance

Calculate the mass balance for each test by adding the respective m_{ap}, m_{pc}, m_{cl}. For each test specimen tested, the value shall range between 95 % to 105 % of m_t, where m_t is the total amount of test liquid applied determined in 8.1.2. Repeat the test if the mass balance is not within the range.

9.2.3 Calculation of repellency, retention, and penetration

For each test specimen, calculate the percentage repellency (PR), the percentage retention (PLR) and the percentage penetration (PP) of the test liquid using Equations (1) to (3) respectively:

$$PR = m_{ap}/m_t \times 100 \quad (1)$$

$$PLR = m_{pc}/m_t \times 100 \quad (2)$$

$$PP = m_{cl}/m_t \times 100 \quad (3)$$

Calculate evaporation loss (EL) for each test specimen using Equation (4):

$$EL = 100 - (PR + PLR + PP) \quad (4)$$

10 Method B

10.1 Verification of amount of active ingredient in test liquid applied

To verify the amount of active ingredient in the test liquid, pipette test aliquots (three replications of the 0,1 ml or 0,2 ml) into 100 ml of the solvent. Shake well and analyse the liquid using the analytical technique selected in Clause 7. The mean value will be used as total amount of active ingredient, m_t, applied in 10.6.

10.2 Determination of extraction efficiency

Calculate the extraction efficiency using the solvent selected prior to testing the test specimen. To measure the extraction efficiency, contaminate and extract three test specimens using procedures in 10.4 and 10.5. Analyse the extracts using the analytical technique selected in Clause 7. Calculate extraction efficiency using the equation given in 10.6.2. 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 %.

10.3 Testing of blanks

To ensure that there is no interference due to chemicals that may be present in the test material and absorbent paper, extract and analyse three 80 mm × 80 mm replicates of test specimen that have not been contaminated (blanks). Extract the blanks using procedures in 10.5. Analyse the extracts using the analytical technique selected in Clause 7. The blanks shall be tested prior to testing the contaminated test specimen.

10.4 Contamination of test specimen

The contamination of the test specimen is determined as follows.

a) Shake the test liquid and carefully aspirate the test aliquot. A magnetic stirrer is recommended to stir liquids that may settle during application.

b) Position the raised platform with the test assembly below the pipettor as marked in 8.2 d). Carefully dispense the test liquid to the centre of the test specimen and simultaneously start the timer. The time taken to dispense the liquid shall be within 5 s. Apply $(0{,}1 \pm 0{,}002)$ ml of test liquid for lower contamination level and $(0{,}2 \pm 0{,}004)$ ml for higher contamination level.

c) After 10 min, remove the cover plate of the test specimen holder. Use tweezers to place the 80 mm × 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.

d) After 2 min more, use tweezers to separate the three layers. Handle the test specimens and absorbent papers from the edges. Place the three layers in separate flasks/bottles.

10.5 Extraction of test liquids

The following procedure shall be used to extract the test liquid from the test specimen and corresponding absorbent papers.

a) Add $(50 \pm 0{,}2)$ 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.

b) Set the orbital shaker speed to (200 ± 20) r/min.

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

d) Start the shaker and the timer and extract for (30 ± 1) min.

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

f) Extract the contaminated material in an additional 50 ml of the solvent following steps a) to e).

g) Combine the two aliquots.

h) Analyse the extracts using the analytical procedure selected in Clause 7. If the analysis is to be conducted later, store the extracts in a freezer for analysis.

The total volume in the storage bottle will not be exactly 100 ml, due to solvent remaining in the material. Use good laboratory practices for the disposal of toxic substances and cleanup of laboratory glassware/apparatus.

10.6 Calculation

10.6.1 Determination of masses of active ingredient in each layer

Multiply the amount (in mg/ml) of active ingredient by 100 (total volume of the solvent in each analysis) to determine m_{ap}, m_{pc}, m_{cl},

where

m_{ap} is the mass, expressed in milligrams, of active ingredient in 80 mm × 80 mm absorbent paper used to remove excess liquid pesticide after 10 min;

m_{pc} is the mass, expressed in milligrams, of active ingredient in the protective clothing material test specimen;

m_{cl} is the mass, expressed in milligrams, of active ingredient in the collector layer.

10.6.2 Calculation of extraction efficiency

Determine the percentage extraction efficiency (EE) using Equation (5):

$$EE = [(m_{ap} + m_{pc} + m_{cl})/m_t] \times 100 \quad (5)$$

where m_t is the total amount of active ingredient applied determined in 10.1.

10.6.3 Calculation of repellency, retention, and penetration

For each test specimen, calculate the percentage repellency (PR), the percentage retention (PLR) and the percentage penetration (PP) of the test liquid using Equations (6) to (8) respectively.

$$PR = m_{ap}/m_t \times 100 \quad (6)$$

$$PLR = m_{pc}/m_t \times 100 \quad (7)$$

$$PP = m_{cl}/m_t \times 100 \quad (8)$$

11 Precision and bias

The precision and bias for these test methods are currently being established.

12 Test report

The test report shall include the following information for each test:

a) a reference to this International Standard (ISO 22608:2004);

b) the identification of the test material, including supplier, trade name, and composition;

c) all details of the treatment (such as laundering and simulated abrasion) of test specimen (required only if the test specimen were subjected to a treatment prior to testing);

d) the information that describes the test liquid and solvent (if extraction procedure was used prior to analysis and if commercial product was used, include the trade name, active ingredient the concentration used for testing;

e) the level of contamination used in testing (either 0,1 ml or 0,2 ml);

f) the test method (A or B) used [include mean evaporation loss (EL) if Method A was used];

g) the analytical technique if Method B was used;

h) the mean and standard deviation of percentage repellency (PR), percentage retention(PLR), and percentage penetration (PP);

i) the conditioning of test specimens and test liquids.

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Price based on 11 pages