

Development of a new test cell to measure cumulative permeation of water-insoluble pesticides with low vapor pressure through protective clothing and glove materials

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Abstract: A collaborative approach, involving resources and expertise from several countries, was used to develop a test cell to measure cumulative permeation by a solid-state collection technique. The new technique was developed to measure the permeation of pesticide active ingredients and other chemicals with low vapor pressure that would otherwise be difficult to test via standard techniques. The development process is described and the results from the final chosen test method are reported. Inter-laboratory studies were conducted to further refine the new method and determine repeatability and reliability. The revised test method has been approved as a new ISO/EN standard to measure permeation of chemicals with low vapor pressure and/or solubility in water.

Key words: Permeation, Pesticides, Chemical-protective garments, Gloves, Chemical protective clothing

Introduction

Penetration and permeation tests are typically used to measure the protection provided by protective clothing and glove materials against chemicals. In both tests, the amount of chemical that is collected on the inner side of the material after a specified duration is used to determine the level of protection provided by the material. In general, penetration tests are conducted to assess the movement of chemical through voids and imperfections, and permeation tests are conducted to measure the ingress of chemicals at a molecular level.

Permeation is measured by clamping a specimen in a cell with test chemical in contact with the outside of the material and a collection medium on the inner side. The test chemical found in the collection medium is used to determine permeation; it is reported either as breakthrough time or cumulative permeation. Permeation standards published by ASTM International, the European Committee for Standardization (CEN) and International Organization for Standardization (ISO) measure permeation of liquids and gases through materials used for chemical protection^{1–3}. These standards are typically used to measure permeation of chemicals that are soluble in water, or volatile substances that can be collected in gaseous collection media. Based on the scope of these standards, they are not suited to measure permeation of pesticides and other mixtures in which the test chemical being measured has low

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vapor pressure and/or is water insoluble.

In the 1980s, research funded by the Office of Pesticide Programs, United States Environmental Protection Agency (US EPA) was conducted to determine permeation of solvents and a few active ingredients in pesticide formulations through glove materials. The ASTM F739 procedure and test cell was modified to allow for the use of solid collection media⁴. Glove data from the studies was published by EPA in 1993 in the guidance manual on personal protective equipment (PPE)⁵. In 1992, Pinette *et al.* compared the method used for the EPA study with an intermittent splash method that was developed as a possible alternative to measure permeation of non-volatile chemicals that are not soluble in water⁶. As noted in the guidance manual and paper by Pinette *et al.*, ASTM Subcommittee F23.30 was considering the development of a new standard method for measuring the permeation of water-insoluble compounds with low volatility^{5,6}. After more than twenty yr, there is still a need for such a standard.

With occupational health in mind, the development of this new permeation test method and test cell was critical. Normalised breakthrough times at low permeation levels have enabled risk assessors to select protective clothing materials based on the permeation of hazardous chemicals that are detected typically in aqueous media or by being collected as a volatile gas during the test itself. With most pesticides, such tests cannot be conducted to determine the permeation of active ingredient. Therefore it makes appropriate PPE selection very difficult, and would typically be based on expert knowledge of expected barrier properties for a given protective clothing material against a given chemical or active ingredient, for example. This paper describes the development of a test method to evaluate chemical protective clothing and gloves used by pesticide workers.

Preliminary Tests

Preliminary tests were conducted to compare test results using the ASTM F739 test cell as well as modifications in methodology reported by other researchers. For the tests described below, permeation of pendimethalin in Prowl[®] 3.3 EC formulation (5% active ingredient) through 0.20 mm thick nitrile gloves was measured. First tests were conducted according to ASTM F739, using the constant contact method for four h with water as the collection media¹. Although the test samples were visibly yellow on the collection side after four h, no active ingredient was detected when air or water were used as collection media.

Clear colorless chemicals would not have revealed the permeation. When acetonitrile was used as the collection medium, 6,564.6 $\mu\text{g}/\text{cm}^2$ of pendimethalin permeated through the nitrile glove sample after four h. As reported in previous studies, constant contact with an organic solvent as the collection medium is known to overestimate chemical permeation results. This may occur due to reverse permeation of the organic solvent which then carries chemical to the collection side.

These preliminary tests show that collection media typically used for neat industrial chemicals cannot be used to measure permeation of pendimethalin, a water insoluble chemical with low vapor pressure. Tests also show that constant contact with a solvent as the collection medium can result in grossly overestimating permeation.

Tests were then conducted using the single splash method similar to that used by Pinette *et al.*⁶. To collect the pendimethalin that permeated through the material, 2 ml of acetonitrile was pipetted on the collection side of the cell and the cell swirled for 30 s. The solvent with pendimethalin was then collected for analysis. Although initial test using the single splash method provided reasonable results, difficulty in standardizing the swirling action for 30 s was observed. Therefore, the method was not considered for further development. A report published by D'Onofrio in 2013 showed that solid collection media can be used to measure permeation of chemical warfare agents, chemicals with low vapor pressure and low water solubility⁷. A meeting with the author was held to discuss our challenges with tests conducted using solid collector media. Test repeated with suggested procedure for preconditioning of solid collection disc was promising. Therefore, the decision was made to continue testing with preconditioned solid collection media.

This paper includes information on tests conducted with three types of cells developed to measure permeation of chemical with low vapor pressure and low solubility in water. Preconditioned collector discs (considered to be a solid collection medium) were used for all three cells. Testing was conducted with one pesticide test chemical using a wide range of glove and textile materials.

Materials and Methods

Test chemical

Prowl[®] 3.3 EC, with 5% active ingredient, was selected as the test chemical for methodology development. Prowl[®] 3.3 EC is an emulsifiable concentrate pesticide product that includes pendimethalin, a yellow colored active ingredient

Table 1. Properties of pendimethalin (CAS# 40487-42-1)

Molecular weight	281.31 g/mol
Specific gravity	1.17 at 25 °C
Solubility in water	0.0003 g/l
Solubility in other solvents	Readily soluble in organic solvents such as acetone, xylene, etc.
Vapor pressure	Negligible; 3.0×10^{-5} mmHg at 25°C

Table 2. Description of the materials used for testing

Code	Material Description
1	Nonwoven fabric coated on the outside
2	Woven fabric with a repellent finish laminated to a microporous membrane
3	Nonwoven fabric coated on the outside with thin polyethylene
4	Knit fabric coated on the outside with thick polyurethane
5	Woven fabric coated on the outside with thick polyvinyl chloride (PVC)
6	Nonwoven fabric coated on the outside
7	Nonwoven fabric coated on the outside with thin polyethylene
8	Nonwoven fabric coated on the outside
9	Woven fabric with a repellent finish and a thin coating on the inside
10	Woven fabric with a repellent finish and a thin coating on the inside
11	Nonwoven fabric on two sides, laminated to a microporous membrane
12	Nonwoven fabric laminated to a microporous membrane on the outside
13	Nonwoven fabric laminated to a microporous membrane on the outside
14	Nonwoven fabric laminated to a microporous membrane on the outside
15	Nonwoven fabric on two sides laminated to center layer
16	8 mil (0.20 mm) nitrile glove material
17	10 mil (0.25 mm) latex sheet
18	~1 mil (~0.025 mm) polyethylene glove material
19	5 mil (0.13 mm) polyvinyl chloride (PVC) glove material

with low vapor pressure and negligible solubility in water. The bright yellow color of the active ingredient was very important in the methodology development process since it enabled qualitative visual detection of the active ingredient. The properties of pendimethalin are given in Table 1.

Test materials

A total of four elastomeric/polymeric and fifteen multi-component textile materials were tested. The description of materials is given in Table 2. Material 17 (0.25 mm thick latex sheet) was used for the initial testing of each cell. The first design of the test apparatus was tested with only elastomeric materials. Subsequent designs were tested with both elastomeric and multicomponent materials.

Collector discs

Preconditioned collector discs (solid collection medium) were used for all three cells. Precorequired wetting the disc with acetonitrile to allow the collector disc's surface to accept the permeant. Tests were conducted to determine the uptake efficiency of preconditioned 3M™ Empore™ C18 discs, and 0.25 mm and 0.51 mm latex, and Benchkote™ Plus. Preconditioned Benchkote™ Plus and 3M™ Empore™ C18 discs had high uptake efficiency as well as high extraction efficiency with acetonitrile. Benchkote™ Plus, a polyethylene-backed absorbent paper, was selected as it is relatively inexpensive and is also used as the collection layer for the pipette test, which is used to measure pesticide penetration. Tests were conducted to refine the preconditioning of the collector disc such that the acetonitrile used was just enough to wet the disc. Too little solvent does not allow good contact, and too much solvent poses the risk of reverse permeation.

Collector discs for cell 2 and the first set of cell 3 trials were conditioned as follows. The collector discs were placed in a glass petri dish and 1.5 ml of acetonitrile was pipetted to the absorbent side of the disc ensuring that the entire surface is covered. Tweezers were then used to carefully lift the collector disc and place it in the center of 8 cm × 8 cm Benchkote® Plus absorbent paper with the absorbent side in contact with the absorbent side of the collector disc. A 100 g weight was placed on top of the collector disc for one min to remove excess solvent. The weight of the collector disc was recorded prior to application of acetonitrile, after pipetting and the amount in the preconditioned disc to monitor for consistency.

A second set of tests was conducted with cell 3 after changes were made to the collector disc. The size of the collector disc was increased from 3.5 cm to 5 cm with the center 2.5 cm center pre-cut to allow separation of the center piece for extraction. For this set, 1 ml of acetonitrile was pipetted and the weight placed to remove excess was increased to 200 g.

Test cells and procedures

For clarity, this section includes a description (including design principles) and the test procedure for each cell. For cells 1 and 2, shortcomings, which drove the methodology development process, are included. Diagrams and images are included for clarification.

Cell 1

Description. The first prototype cell was based on a drawing provided by C. Mekeel, which included some



Fig. 1. (a) Left: Side view of cell 1 mounted on a stand. (b) Right top: Collector disc being placed on top of the elastomeric material (yellow chemical in the chamber below the sample is visible through the translucent material). (c) Right bottom: Weight placed on top of the collector disc to ensure contact with material.

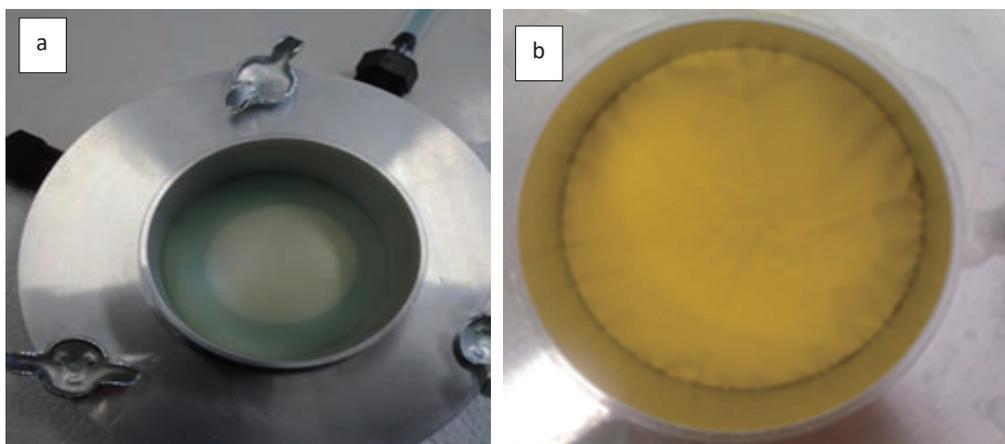


Fig. 2. Images showing the two problems with the cell. Fig. 3(a). Left: The image is of the original cell with colored water showing an air bubble on the surface that was difficult to remove. Fig. 3(b). Right: The image shows the stretching and associated puckering of the elastomer when the test chamber was filled with challenge chemical.

modifications to the cell used by Ehntholt *et al.*⁴⁾. The base of cell consisted of a 2 mm deep reservoir to hold the test chemical below the test sample. The reservoir had two openings, an inlet that was attached to a plastic syringe to fill the cell, and an outlet that was connected to plastic tubing to empty the cell. An O-ring placed in the groove of the metal base was used to prevent leakage. The metal cover had a cylindrical opening in the center to place the collector disc and a 225 g weight over the sample. Three wing nuts were used to secure the test assembly. The inlet and outlet mechanism of the original design was modified and

the cell mounted on the stand to make it easier to fill and empty the cell (see Fig. 1a).

Shortcomings. Air trapped between the test chemical and sample was difficult to remove (see Fig. 2). Since the test chemical was added to the chamber after the sample was mounted, some elastomeric materials stretched, thus changing the physical characteristics of the material. The surface of the material became dome-shaped instead of remaining flat as a result of the pressure from the test chemical beneath it. Therefore, the decision was made to design a new cell to address the problem with the stretch-

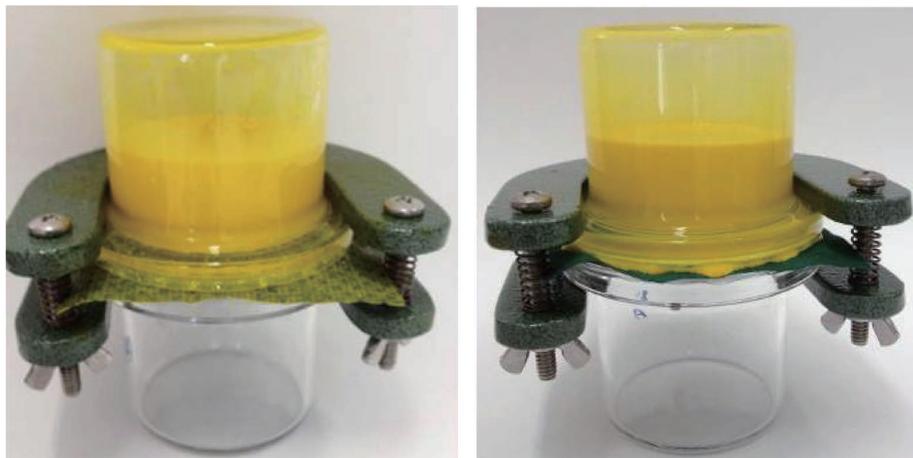


Fig. 3. Leakage in cell 2.

ing elastomeric material.

Cell 2

Description. The second prototype cell was developed to address the deficiencies in the first cell. This cell was designed such that the test chemical was on top of the material and the collector disc below on a firm surface that prevented the elastomeric material from stretching. The cell consisted of two cup-shaped glass pieces, each with a 5 cm inner diameter and 5 cm depth, with rims clamped together with a horseshoe clamp. The cells were tested using water with blue dye to determine leakage; none was found.

The cup-shaped pieces had flat as well as grooved rims. These pieces were connected with different rim configurations (one flat and one grooved rim; two flat rims; two grooved rims) to evaluate for the most effective arrangement. The cup with the flat rim in contact with the material and grooved rim with O-ring as the other half was found to be most effective and therefore was used for the testing of glove materials. Once it was determined that the issues with elastomeric materials (latex sheet and glove materials) observed in Cell 1 were addressed, testing was continued with multicomponent materials.

Test procedure

The cup with the flat rim was filled with 50 ml of test chemical. The 8×8 cm test specimen was placed on top of the rim. A preconditioned collector disc (4.7 cm diameter) was placed in the center of the test specimen such that absorbent side was in contact with the back of the test specimen. The stiff backing material was then placed on top of the collector disk and the second cup-shaped piece

with O-ring aligned, the two glass halves being clamped together with the horseshoe clamp. The test cell was then inverted so that the test chemical was in contact with the test specimen. After one h the cell was inverted again prior to carefully disassembling the cell to remove the collector disc for extraction.

Shortcoming. Most materials could be tested with this cell without any problem. However, leakage was difficult to control for materials that required a higher pressure to seal the edges (see Fig. 3). The glass cell cracked when pressure was increased.

Cell 3

Description. To address the shortcomings of the glass cell, the decision was made to use PTFE following an in-depth discussion to address the challenges with the glass cell (see Fig. 4). For this cell, three holes are drilled through the top and base, and bolts pass through both pieces. A torque wrench is used to tighten the nuts. A small spout on top of the cell allows for easy removal of the test chemical. The raised surface pushes the sample into intimate contact with the collection medium.

Test Procedure. The following methodology was used to conduct the tests with the PTFE cell. The collector discs were preconditioned prior to testing and the amount of acetonitrile in the disc monitored for consistency. For pre-cut discs, the amount of acetonitrile in the discs ranged from 0.19 g to 0.24 g (0.24 g equates to only 0.31 ml of acetonitrile).

The three bolts and washers were inserted through the base of the test cell, and the base was placed on a flat surface. The preconditioned disc was carefully centered on the cell with the polyethylene side of the collection layer

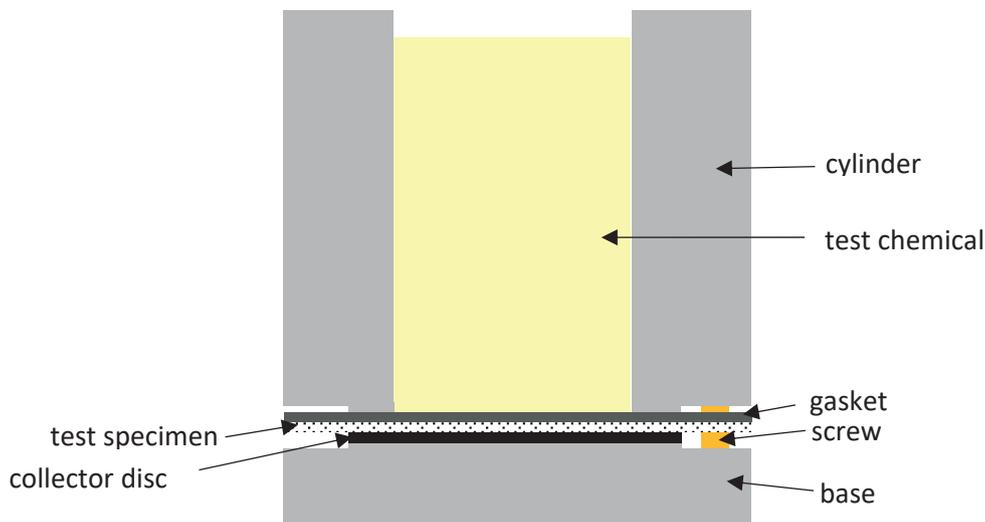


Fig. 4. Schematic of cell 3, made of PTFE.

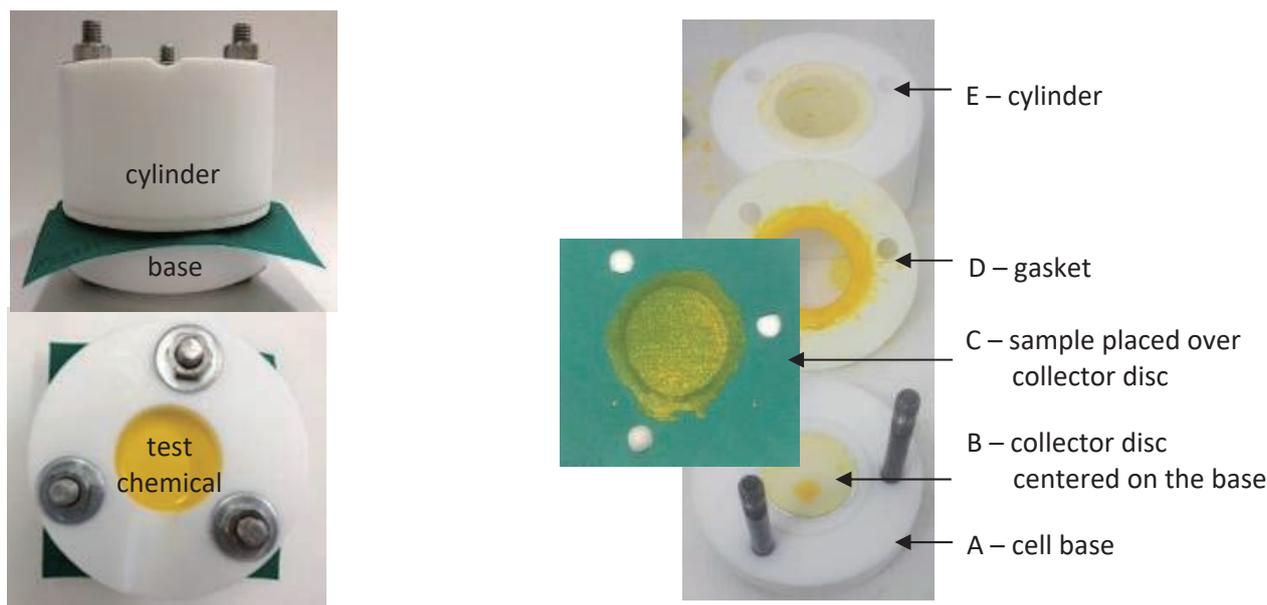


Fig. 5. (a) Left: Side and top view of the cell 3 assembly. (b) Right: Disassembled cell to show the order (A–E) in which the parts are assembled.

facing the base. The test sample was placed on top of the preconditioned disc with the material inner surface in contact with the disc, followed by the gasket. A 100 g weight was placed on the 35 mm opening to improve contact by removing the air between the collector disc and test sample. The cylinder (top of the test cell) was placed with the spout-side facing up (see Fig. 5). The nuts were first tightened by hand, and then a torque wrench set at 5 Nm was used to achieve a liquid-tight seal. Use of the torque wrench also gives consistency from test to test since the

pressure applied around the edge of each sample is the same. Since the test chemical is an emulsion, it was shaken well, and then 35 ± 1 g was weighed and poured into the test chamber.

After one h the cell was emptied and disassembled, and the test sample was removed carefully to prevent contamination of the collector disc. The first set of tests was conducted using a 35 mm collector disc with aluminum foil backing and the second set using a pre-cut collector disc in which only the center was extracted. The outer ring of the

Table 3. Cumulative permeation ($\mu\text{g}/\text{cm}^2$) of pendimethalin through multicomponent fabrics

Code	Cell 2 - Glass		Cell 3 - PTFE		Cell 3 - PTFE Pre-cut		Comments
	Mean	SD	Mean	SD	Mean	SD	
1	0.01	0.00	0.01	0.01	0.00	0.00	Not detectable/measurable or low permeation
2	0.03	0.02	0.11	0.01	0.04	0.03	Not detectable/measurable or low permeation
3	0.00	0.00	0.00	0.00	0.05	0.01	Not detectable/measurable or low permeation
4	0.01	0.00	0.11	0.10	0.17	0.30 ¹	¹ One reading of 0.52
5	0.04	0.02	0.19	0.06	0.26	0.21	Low permeation
6	0.46	0.08	1.17	0.49	0.83	0.27	Relatively low permeation
7	0.04	0.02	2.20	0.40	1.54	0.83	Relatively low permeation
8	0.52	0.06	8.34 ²	0.72	1.59	0.87	² High permeation around the edge that was addressed by pre-cutting the collector disc
9	5.78	0.6	8.80	3.39	53.50	42.56 ³	³ Variability due to pin holes
10 ⁴	42.01	6.88	83.16	36.12	126.73	64.85	⁴ Woven fabric with thin coating on the back
11	236.93	362.7 ⁵	8.97	6.93	7.40	2.45	⁵ High value in glass because of leakage that was addressed in the cell 3 design
12 ⁶	7.90	9.42	5.51	0.77	10.52	6.91	⁶ Spunbond fabrics with microporous membrane. Variability observed in materials with this fabric construction.
13 ⁶	3.64	2.68	103.22	60.69	34.86	30.95	
14 ⁶	41.21	29.90	329.35	27.56	38.69	17.56	
15	3,206.50	615.70	4,115.17	408.88	3,979.74	424.20	High movement of chemical due to penetration through imperfections in the material.

Superscript number corresponds to the comment in the right column.

disc, which may be affected by the pressure applied to seal the cell, was discarded.

Extraction and chromatographic analysis

The pre-cut collector disc was extracted with 20 ± 0.05 ml of acetonitrile using a wide-mouth bottle, placed in an orbital shaker at a speed of (200 ± 20) revolutions per min. After 30 min, the extract from the bottle was analyzed using HPLC with a UV detector. (*Instrument*: Chromquest, UV detector, autosampler; *80:20 ACN:H₂O Isocratic*; *Rate*: 1 ml/min; *Volume injected*: 20 μL ; *Wavelength*: 240 nm; *Column*: ACE C18 (4.6 mm \times 25 cm) and 5 μm particle size).

Calculation of results

The following equation was used to calculate cumulative permeation for all test cells.

$$CP = (C \cdot V) / A$$

where

CP = cumulative permeation, $\mu\text{g}/\text{cm}^2$

C = concentration of chemical of interest in extractant, $\mu\text{g}/\text{ml}$

V = volume of extractant, ml

A = area of the collector disc that was extracted, cm^2

The chemical of interest was pendimethalin and the extractant was acetonitrile for this study. For all tests the outer side of the material was in constant contact with the

challenge chemical for one h.

Results and Discussion

Table 3 shows mean and SD of fifteen multicomponent materials tested with the prototype cell 2, and prototype cell 3 (set with whole collector disc was extracted) and Cell 2 with pre-cut discs (set with only the center extracted). In addition to quantitative data, the bright yellow color of the pendimethalin (active ingredient) played a key role in methodology development. The distribution pattern of pendimethalin on the collector disc assisted in better understanding the possible reasons for variability. For some materials it was possible to determine whether the pendimethalin permeated through the material or whether the bright color on the disc was due to imperfections or defects in the material (i.e. penetration). Unfortunately this distinction is often not possible in clear colorless chemicals in which the results are based entirely on quantitative analysis. As with other permeation test methods, differentiation between penetration and permeation is difficult in materials with small pores, pinholes, and gaps in coating. In practice the differentiation between penetration and permeation is largely irrelevant; in both cases the chemical gets inside the PPE where it can come into direct contact with the wearer.

The quantitative data and visual observations of test

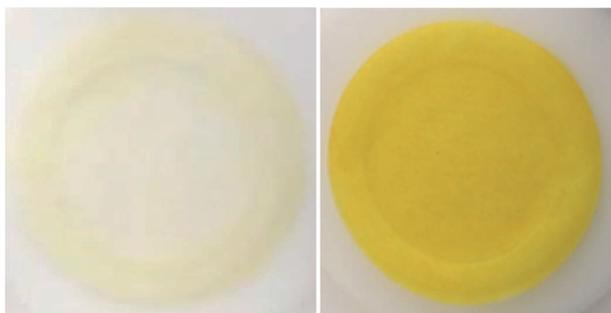


Fig. 6. Collector discs with uniform permeation.

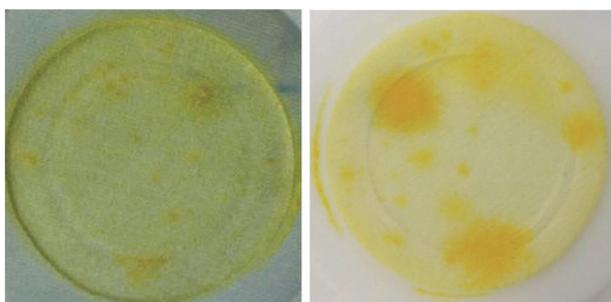


Fig. 7. Penetration around the edge due to fabric damage as a result of pressure from tightening the cell.



Fig. 8. High variability in a permeation observed in collector discs placed below material with microporous surface and spunbond backing.

specimens and collector discs demonstrate the progression of the cell development. Materials 1–7 had permeation below the detection limit or low permeation values regardless of the test cell and material 15 had consistently high mean and SD regardless of the test method. Relatively higher permeation for material 11 when tested with cell 2 was due to leakage. This shortcoming was addressed in cell 3. Similarly, for material 8, higher permeation observed and recorded around the edges was addressed by pre-cutting the disc and testing only the center.

Figures 6–8 include examples of the different types of distribution patterns that assisted in determining the possible reasons for variability. Figure 6 includes examples of

materials that passed the pressure penetration test; therefore, the pendimethalin observed as yellow coloration has permeated through the materials⁸). The distribution of the pendimethalin on these fabrics is fairly uniform. Figure 7 is an example of a coated fabric with pinholes resulting in penetration. Variability was observed in all materials with a microporous surface on spunbond backing. It was observed that the performance of these materials varied; some provide more protection than others. Figure 8 is an example of sample with high variability. The number of samples tested for these materials may have to be higher due to high variability.

The methodology, including the dimensions of collector disc, was further refined through inter-laboratory study conducted as part of ISO standard development. A second study inter-laboratory was conducted to determine repeatability and reliability of the test method⁹). The revised test method has been approved as a new ISO/EN standard to measure permeation of chemicals with low vapor pressure and/or solubility in water¹⁰).

Conclusion

The test cell developed as a result of this study is suitable for assessing the permeation of pesticides with low vapor pressure through protective clothing and gloves. The shortcomings of previous methodologies have been addressed in a three-stage development process. Issues addressed include the problem of challenge pesticide leaking from the test cell and the difficulty of ensuring intimate contact between the material under test and collector disc. Qualitative visual assessment of permeation by a brightly colored active ingredient provided information on distribution pattern that could not be determined by chemical analysis. Samples with uniform permeation had lower variability.

Usefulness of This Study

In the past, permeation data was available for materials tested with volatile organic chemicals but not for active ingredient in pesticides. Development of this permeation test method allows measurement of cumulative permeation of active ingredients in pesticide products. Cumulative permeation and knowledge of pesticide toxicity will allow risk assessors to make a judgment based on the knowledge of the test result which would previously have been unavailable, and of the working situation of those that may be exposed to the pesticide.

The new permeation standard is required for the performance standards for protective clothing and gloves used by pesticide operators^{11,12}. These performance standards provide a mechanism to assign protection factors for protective clothing, accessories such as aprons and gloves required for risk mitigation.

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