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GERARD C. COLETTA^a, S.Z. MANSDORF^b & S.P. BERARDINELLI^c

^a Tillinghast, a Towers Perrin Company, 333 Bush Street, San Francisco, CA 94104

^b S.Z. Mansdorf & Associates, 2000 Chestnut Blvd., Cuyahoga Falls, OH 44223

^c National Institute for Occupational Safety and Health, Division of Safety Research, 944 Chestnut Ridge Road, Morgantown, WV 26505-2888

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Chemical Protective Clothing Standard Test Method Development: Part II. Degradation Test Method*

GERARD C. COLETTA,^A S.Z. MANSDORF^B and S.P. BERARDINELLI^C

^ATillinghast, a Towers Perrin Company, 333 Bush Street, San Francisco, CA 94104; ^BS.Z. Mansdorf & Associates, 2000 Chestnut Blvd., Cuyahoga Falls, OH 44223; ^CNational Institute for Occupational Safety and Health, Division of Safety Research, 944 Chestnut Ridge Road, Morgantown, WV 26505-2888

A "round-robin" interlaboratory evaluation of a proposed American Society for Testing and Materials (ASTM) standard test method was conducted for measuring the resistance of chemical protective clothing materials to degradation by liquid chemicals. The objective of this project was to determine the relative precision of the method and, where appropriate, recommend modifications that would improve reliability. In the round-robin format, eight laboratories used the proposed method to test each of five protective clothing materials against five liquid chemicals that are widely used commercially. The resulting data revealed that the proposed test method was not stringent enough to generate acceptable levels of accuracy and precision. Both intra- and interlaboratory standard deviations showed a high degree of variability in changes for the three physical properties evaluated. Changes in the method were identified which, if implemented, should improve accuracy and precision considerably.

Introduction

This study presents the results of an eight laboratory, round-robin evaluation of a proposed American Society for Testing and Materials (ASTM) standard test method developed by ASTM's Committee F-23 on Protective Clothing, entitled "Test Method for Evaluating Protective Clothing Materials for Resistance to Degradation by Liquid Chemicals."⁽¹⁾ The quantitative evaluation described herein represents only an initial phase in the rigorous process of developing a standard test method for protective clothing. Such testing and review are essential in developing methods that are reproducible, accurate and without significant bias.

Evaluation of this proposed degradation test method was the second of a two-part project on developing chemical protective clothing standard test methods and was funded by the National Institute for Occupational Safety and Health (NIOSH) under contract No. 200-84-2702. The first part of this project was devoted to evaluating a test method for measuring the resistance of protective clothing materials to penetration by liquid chemicals.

This second part of the NIOSH project was designed to thoroughly evaluate the proposed ASTM degradation test method and, where appropriate, recommend changes that would lead to improvement.

Background

Standard test methods for measuring the resistance of chemical protective clothing materials to degradation and penetration are essential in generating performance data for proper selection and use of this equipment. Without such

performance data, workers cannot be assured of adequate protection when exposed to liquid chemicals. The need for accurate, meaningful performance data has become especially apparent with the recent publication of information showing measurable permeation of hazardous liquid chemicals through protective clothing materials heretofore thought to be impervious. An increasing emphasis on the use of chemical protective clothing to provide worker protection at hazardous waste sites and other uncontrolled environments has added significantly to this need. Further, the historical lack of performance data for use in the selection process is underscored by the fact that skin disease and disorders have accounted for more than 40 percent of all reported occupational illnesses every year from 1972 to 1980.⁽²⁾

Testing Strategy

The proposed degradation test method, as developed by ASTM's Committee F-23 on Protective Clothing, provides an initial screening for chemical protective clothing materials. It is intended to be the first in a hierarchy of three test methods measuring important performance parameters: resistance to degradation, resistance to penetration and resistance to permeation. Testing by this triad of performance parameters presents candidate clothing materials with challenges of increasing sophistication and rigor. With a degradation test scheduled first, only the best performing materials will be taken through the entire testing protocol. In this way, only a limited number of candidate materials should be put through the more complex, time consuming and expensive permeation test method.

It is important to note that the degradation test method is not intended to be, nor should it be used as, a substitute method for measuring penetration or permeation resistance.

*Mention of company names or products does not constitute endorsement by the National Institute for Occupational Safety and Health (NIOSH).

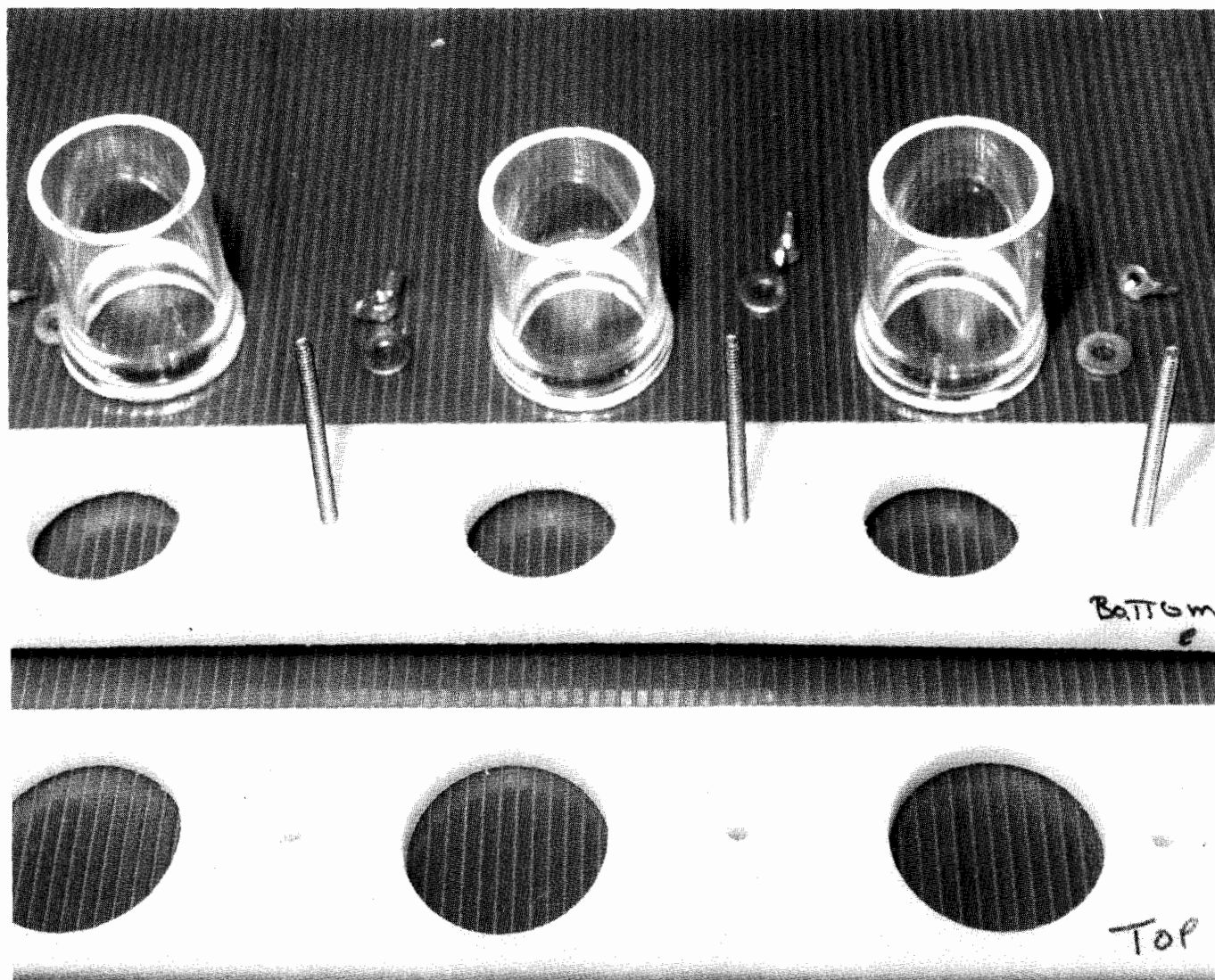


Figure 1 — Chemical degradation test apparatus.

Round-Robin Evaluation

Each participating laboratory was provided with an identical copy of the proposed degradation test method to ensure that, as much as could be anticipated, the labs would each follow the same set of procedures. The draft method was structured in a detailed, step-by-step format.

Test Method Design

With the proposed degradation test method, the resistance of a protective clothing material is determined by the following:

1. measuring the thickness, weight and elongation of fresh specimens of the candidate clothing material that have not been contacted by the liquid chemical of interest;
2. measuring the thickness, weight and elongation of specimens of the clothing material that have been contacted by the liquid chemical; and
3. comparing the results of steps 1. and 2. to identify the changes resulting from contact by the liquid chemical.

The resistance to degradation of a protective clothing material is evaluated further by noting changes in the visual appearance of the surface of specimens that have been contacted by the chemical. This could include discoloration, cracking, flaking, or stiffening.

Changes in weight, thickness and elongation are only three of many possible indicators of material degradation. They were selected because of their ability to be measured and thereby quantified. Other parameters, such as tensile or tear strength, could be used just as easily.

Materials and Challenge Liquids

Protective clothing materials and challenge liquid chemicals discussed in Part I were used throughout this study (*materials*: polyvinyl chloride, polyvinyl alcohol, Neoprene, butyl, Tyvek; and *liquids*: water, toluene, MEK, hexane, HCl).

Apparatus and Procedures

The test fixture is a simple "sandwich" configuration consisting of three identical one-chamber glass pipe cells held between top and bottom polyethylene boards (Figure 1). Material specimens are positioned on the bottom board. The test cells

are placed directly on top of the specimens and the apparatus assembly completed. Liquid chemical is then poured into the cells to initiate contact with the material specimens.

For this project, the glass cells were sized at a nominal 5.1 cm (2.0 in.) inside diameter to provide a contact area of approximately 20.3 cm² (3.1 in.²). For each liquid chemical, six random specimens of each of the clothing materials were selected, cut to 7.0 cm × 7.6 cm (2.8 in. × 3.0 in.) and tested.

The thickness and weight were measured for three specimens selected from the six for contact by the liquid chemical. From each of the remaining three, rectangular samples [7.6 cm × 1.3 cm (3.0 in. × 0.5 in.)] were cut for initial elongation measurements. One end of each strip was clamped and attached to a ring stand. Elongation was then measured by hanging weights on the free end until approximately 20% (1.0 cm) stretch was achieved. The value of the load for this elongation was recorded.

The first three specimens were placed in the test fixture and liquid chemical added to a depth of 1.0 - 1.5 cm (0.4 - 0.6 in.). Liquid chemical contact was continued for one hour. At the end of the hour, the liquid chemical was poured out, the fixture disassembled and the thickness and weight of the contacted specimens measured.

From each of these three contacted specimens, a rectangular sample was cut to 7.6 cm × 1.3 cm (3.0 in. × 0.5 in.). Elongation was accomplished using the weight identified in the earlier elongation of noncontacted specimens to 20% stretch.

Results from each test were recorded. Thickness, weight, elongation and the percent (%) change in each were tabulated. The thickness and weight changes are representative of the area in each specimen that was contacted by the liquid chemical. Observations of surface changes were noted also.

TABLE I
Chemical Protective Clothing Degradation Test Results:
Polyvinyl Chloride Laboratory Summary

Laboratory	Thickness (% Change)					Weight (% Change)				
	Water	Toluene	MEK	Hexane	HCl	Water	Toluene	MEK	Hexane	HCl
1	1.9	66.7	dissolved	-0.8	0.9	0.0	22.0	dissolved	-18.8	0.0
2	-2.3	1.6	dissolved	0.0	0.9	0.1	-20.4	dissolved	-12.0	0.0
3	0.0	5.3	dissolved	-2.8	7.0	2.2	-48.9	dissolved	-68.7	3.7
4	-8.2	-17.8	dissolved	1.0	0.3	0.0	-35.1	dissolved	-14.3	1.4
5	4.5	4.4	dissolved	5.9	0.0	0.1	11.2	dissolved	2.4	187.0
6		2.2	dissolved	-5.9			32.7	dissolved	-71.9	
7	0.0	0.4	dissolved	1.9	2.2	0.7	-4.8	dissolved	-12.9	9.9
8	4.8	9.0	dissolved	0.8	-3.7	0.5	10.3	dissolved	-4.4	0.5
Average	0.1	9.0	dissolved	0.0	1.1	0.5	-4.1	dissolved	-25.1	28.9
Within-lab SD ^A	8.6	11.7		4.9	3.7	0.4	8.3		5.9	4.1
Between-lab SD	4.1	23.1		3.2	3.0	0.7	26.7		26.8	64.6
Coefficient of Variation	95.3	288.0		--	433.0	161.0	682.0		109.0	224.0

Laboratory	Elongation (% Change)				
	Water	Toluene	MEK	Hexane	HCl
1	0.0	253.5	dissolved	-63.4	0.0
2	-33.7	-69.9	dissolved	-27.2	-30.4
3	10.1	60.1	dissolved	-63.4	-18.7
4	-7.4	58.9	dissolved	-35.1	-14.3
5	18.1	133.7	dissolved	-54.7	28.8
6		282.6	dissolved		
7	3.5	112.5	dissolved	-40.0	-6.0
8	3.1	237.8	dissolved	-40.8	7.3
Average	0.1	118.9	dissolved	-50.7	-6.4
Within-lab SD	13.6	27.2		8.4	13.2
Between-lab SD	15.3	127.5		13.8	19.2
Coefficient of Variation	22.7	107.0		32.0	364.0

^ASD = standard deviation.

TABLE II
Chemical Protective Clothing Degradation Test Results:
Polyvinyl Alcohol Laboratory Summary

Laboratory	Thickness (% Change)					Weight (% Change)				
	Water	Toluene	MEK	Hexane	HCl	Water	Toluene	MEK	Hexane	HCl
1	-18.7	-0.5	5.9	-0.5	-35.1	64.0	94.3	0.0	-1.3	35.7
2	11.9	4.8	-2.4	-0.8	-8.6	75.5	125.3	75.8	4.2	29.7
3	-7.9	0.0	5.0	-1.3	dissolved	155.0	31.0	-6.7	4.5	dissolved
4	-29.3	-6.3	-0.6	-9.7	-48.3	84.5	6.3	34.1	1.8	43.5
5	12.6	-10.0	-2.1	1.4	-41.6	303.8	500.5	338.9	237.4	301.6
6						150.9	153.5	0.0	0.0	
7	-12.0	-2.3	2.4	-0.8	-25.5	45.2	180.5	128.2	60.8	197.8
8	-4.9	-5.7	6.7	1.5	-5.9	40.4	9.2	43.3	4.1	87.5
Average	-6.9	-2.9	2.1	-1.5	-27.5	114.9	137.6	76.7	38.9	116.0
Within-lab SD ^A	4.2	1.7	1.2	2.6	7.5	38.4	48.2	49.5	39.0	34.2
Between-lab SD	14.2	4.5	3.6	3.5	15.9	82.2	150.4	107.8	77.5	100.9
Coefficient of Variation	215.0	166.0	181.0	291.0	64.0	79.0	115.0	155.0	223.0	92.0

Laboratory	Elongation (% Change)				
	Water	Toluene	MEK	Hexane	HCl
1	47.0	0.0	0.0	0.0	0 to 93
2	8.0	-0.0	0 to 15	0 to 8	tore
3	460.0	50.6	-34.8	5.6	dissolved
4	1453.5	-53.6	0 to 3	-77.8	7788.9
5	662.5	187.7	511.7	-14.4	tore
6					
7	1395.6	163.3	56.7	103.3	5945.1
8	466.8	21.9	-8.0	44.1	3910.1
Average	887.7	52.9	105.1	10.1	5881.3
Within-lab SD	534.8	99.9	324.5	52.9	2457.8
Between-lab SD	444.7	83.1	205.5	55.2	1584.2
Coefficient of Variation	78.0	246.0	365.0	757.0	50.0

^ASD = standard deviation.

Results

Seven of the eight selected laboratories completed the degradation tests using all protective clothing materials and all liquid chemicals. The eighth lab conducted a limited number of tests and submitted only summary data for those tests it conducted. Summary data for the complete evaluation are provided in Tables I through V. One table has been generated for each of the five tested clothing materials.

In addition to the average percent change measured by individual laboratories for each physical property (thickness, weight and elongation) for each test chemical, the tables include aggregate average percent changes for each parameter. Also, the tables contain calculated intralaboratory (within-laboratory) and interlaboratory (between-laboratory) pooled standard deviations and the coefficient of variation for all data categories.

The data show wide variations in all statistical parameters. In many cases, the direction of change (*i.e.*, + or -)

differs among the laboratories. Furthermore, most of the standard deviations are well in excess of values generally accepted in standardized test methods. A coefficient of variation for a laboratory test method such as this should be less than 10%.

These results indicate that both the experienced and inexperienced laboratories had some difficulty in interpreting the written test method and/or in conducting the actual test procedure. Interestingly, several of the more experienced labs appeared to have had the most difficulty. Feedback from the laboratories and a review of raw test data highlighted the following problem areas:

1. Thickness measurements

- A specific technique for thickness measurements was not indicated. This led to the use of several approaches that generated differing results.

- Furthermore, the configuration of a thickness gauge was not specified. Without foot pads to bridge peaks and valleys in a material specimen's surface, measurements could have a high variability. This is especially important in measuring swollen specimens after chemical contact.

2. Weight measurements

- Because the test method did not unequivocally state that weight measurements and calculated changes should pertain only to the area contacted by a liquid chemical, several labs reported values for the entire material specimen. (These weight changes were recalculated to represent only areas contacted by liquid chemical.)
- Several laboratories experienced difficulty in handling and weighing specimens contacted by a liquid chemical immediately after removal from the test apparatus. Specifically, the use of tared weighing bottles was considered awkward and time consuming.

- The time suggested for drying wet specimens by blotting was not specified. Laboratories were inconsistent in the amount of drying accomplished.

3. Elongation measurements

- Rectangular strips used for contacted (by the liquid chemical) elongation measurements were not taken from the same specimens as the strips for noncontacted elongation measurements. This may have led to significant variations in the results.
- Several labs incorrectly varied the noncontacted and contacted elongation load to maintain a constant 20% elongation rather than use the same "before" load to obtain directly a change in elongation. (New elongations based on fixed "before" loadings were estimated using Hooke's Law).
- The length of rectangular strip to be made available for stretch was not specified precisely. Therefore, the available length between clamps depended on each lab's preference and led to some variability.

TABLE III
Chemical Protective Clothing Degradation Test Results:
Neoprene Laboratory Summary

Laboratory	Thickness (% Change)					Weight (% Change)				
	Water	Toluene	MEK	Hexane	HCl	Water	Toluene	MEK	Hexane	HCl
1	0.0	55.7	15.1	6.6	0.8	2.7	295.9	91.2	12.3	0.9
2	0.0	17.5	4.5	0.0	-2.2	3.2	159.8	36.6	0.0	0.5
3	0.0	47.0	22.3	2.4	13.1	9.5	364.2	101.0	24.3	8.0
4	0.2	31.5	12.4	6.4	-0.7	0.4	105.4	43.3	20.7	0.1
5	0.0	34.4	15.6	2.8	-2.7	1.1	163.4	47.3	10.2	1.7
6		34.9	9.1	4.5			390.6	109.6	29.9	
7	2.6	37.2	14.3	3.3	0.0	2.5	128.4	45.9	5.1	0.4
8	3.9	59.0	20.4	6.0	0.1	3.3	171.0	44.8	6.1	2.1
Average	1.0	39.6	14.2	4.0	1.2	3.2	222.3	65.0	13.6	2.0
Within-lab SD ^A	1.8	4.7	3.0	2.0	2.5	1.4	18.4	17.3	5.3	1.7
Between-lab SD	1.5	12.7	5.4	2.2	5.0	2.7	103.9	28.1	9.7	2.5
Coefficient of Variation	234.0	34.0	44.0	74.0	466.0	95.0	47.0	51.0	81.0	151.0

Laboratory	Elongation (% Change)				
	Water	Toluene	MEK	Hexane	HCl
1	21.2	tore	341.4	79.3	17.9
2	40.8	41.1	2.9	12.7	-7.2
3	6.5	162.3	89.8	20.2	1.3
4	17.3	80.8	103.0	77.4	4.8
5	11.4	280.9	104.6	13.3	7.2
6					
7	0.8	tore	126.3	-3.0	-12.3
8	-0.3	tore	263.0	36.7	4.6
Average	14.0	141.3	147.3	33.8	2.3
Within-lab SD	24.7	44.7	24.5	23.7	8.4
Between-lab SD	13.2	91.7	106.5	30.2	9.1
Coefficient of Variation	200.0	72.0	74.0	114.0	538.0

^ASD = standard deviation.

TABLE IV
Chemical Protective Clothing Degradation Test Results:
Butyl Laboratory Summary

Laboratory	Thickness (% Change)					Weight (% Change)				
	Water	Toluene	MEK	Hexane	HCl	Water	Toluene	MEK	Hexane	HCl
1	0.6	32.1	4.3	24.8	0.0	1.2	110.1	2.1	75.9	0.3
2	-1.1	26.5	-0.4	3.9	0.0	0.0	63.0	0.5	66.6	0.0
3	0.0	30.1	7.7	16.4	7.0	0.0	215.6	5.5	93.4	0.0
4	-0.8	3.1	0.7	2.0	0.2	0.0	84.0	2.3	67.4	0.0
5	10.2	17.8	1.3	10.6	0.0	20.8	79.5	3.4	60.0	-0.1
6		24.6	2.7	15.3			241.7	0.0	143.7	
7	0.0	23.8	1.2	22.6	2.2	0.8	99.5	1.6	69.4	0.4
8	-0.5	23.5	0.5	29.2	1.1	0.1	81.9	1.1	41.9	0.6
Average	1.2	22.7	2.2	15.6	1.5	3.3	121.9	2.1	77.3	0.2
Within-lab SD ^A	2.9	3.8	2.6	3.5	1.3	5.7	12.6	0.5	10.7	0.8
Between-lab SD	3.7	8.5	2.5	9.1	2.4	7.2	63.3	1.6	28.5	0.2
Coefficient of Variation	392.0	41.0	164.0	62.0	182.0	278.0	53.0	80.0	39.0	412.0

Laboratory	Elongation (% Change)				
	Water	Toluene	MEK	Hexane	HCl
1	-3.6	261.4	48.9	142.5	-7.2
2	-14.1	47.7	-12.1	4.5	-10.5
3	11.7	174.8	36.9	186.2	5.2
4	9.1	233.9	20.3	357.6	-24.9
5	10.7	482.2	-11.0	205.3	18.9
6					
7	-5.8	181.8	-9.6	141.0	-58.6
8	10.4	225.3	-25.8	483.8	-28.6
Average	2.6	229.6	6.8	217.3	-15.1
Within-lab SD	17.2	77.1	23.1	86.5	11.7
Between-lab SD	9.6	121.5	26.4	145.6	23.4
Coefficient of Variation	758.0	63.0	516.0	78.0	173.0

^ASD = standard deviation.

Each of these problem areas alone probably would not account for wide variation in test data. Together, however, they have the potential for causing the large disparity in results encountered in this round-robin evaluation.

Discussion

A review of the ranges of both within- and between-laboratory standard deviations quickly revealed that the degradation test method, as written, does not generate acceptable levels of accuracy and precision. As shown for polyvinyl chloride, in Table VI, large variations in the standard deviations (SDs) were recorded. For example, the within-laboratory SD for elongation ranged from 0 (for dissolved PVC) to 27.2. The between-laboratory SD for elongation ranged from 0 (for dissolved PVC) to 127.5. Thickness and weight changes had similar wide ranges. Furthermore, test results for the other four materials demonstrated as wide or wider SD ranges. The coefficients of variation were all greater than ten percent.

Within-laboratory standard deviations, between-laboratory standard deviations, and coefficients of variation between laboratories were determined by following the procedure outlined in *ASTM Standard Practice (E 691-79)*, "Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods."⁽³⁾

In an effort to reduce the variability found in the raw data as generated by this test method, weights and elongations were normalized by dividing by material thickness, and new percent changes were calculated. This technique had little effect in reducing the standard deviations and was discounted in the final analyses.

Conclusions

This method, as used throughout the round-robin evaluation, did not demonstrate within- and between-laboratory accuracy and precision expected of a statistically valid test procedure.

The test method does appear valuable, however, because it permits measurement of important physical properties, is

relatively easy to perform, and does not require a commitment of costly apparatus or manpower. Improvements recommended by the participating laboratories and drawn from the review test data may well eliminate most of the accuracy and precision problems encountered in the project. This is a case in which considerable benefit can be drawn from apparently negative test results.

Recommendations

A number of improvements for the degradation test method have been identified as follows:

1. Thickness measurements

- One technique for measuring the thickness of materials should be cited. This will eliminate variability in areas such as specimen compression during measurements. Federal Test Method 5030.2 (No. 191A), which specifies compression weight, was used by one laboratory successfully and should be considered a viable candidate.

- A thickness gauge with foot pads should be specified, as is done in the above-cited Federal Test Method. As an alternative, thin glass slides might be considered for use on either side of a material specimen in a sandwich configuration to act as foot pads.

2. Weight measurements

- A reworded method should clearly state that weight change measurements pertain only to the specimen area contacted by a liquid chemical. Applicable calculations should be described.
- An alternative to the tared weighing bottle, such as a zip-lock polyethylene baggie, can be used to minimize awkwardness in handling contacted specimens. Users should note that baggies may be susceptible to degradation when contacted by some chemicals.
- An alternative technique for blotting dry contacted specimens to ensure less variation in results should be developed. Specifically, a time for blotting should be stated.

TABLE V
Chemical Protective Clothing Degradation Test Results:
Tyvek Laboratory Summary

Laboratory	Thickness (% Change)					Weight (% Change)				
	Water	Toluene	MEK	Hexane	HCl	Water	Toluene	MEK	Hexane	HCl
1	-2.6	-2.4	-2.6	0.0	0.0	0.0	260.6	281.9	144.2	31.1
2	-8.3	0.0	-27.6	-8.3	-11.7	0.0	0.0	4.6	8.4	35.6
3	0.0	21.7	20.6	0.0	6.7	0.0	0.0	85.8	0.0	641.2
4	-5.5	-0.7	2.8	-15.6	6.7	0.5	30.7	11.0	7.7	131.0
5	0.0	-2.6	2.9	-2.6	-0.2	18.2	321.6	303.5	88.9	127.8
6										
7	10.2	-4.4	-9.6	18.2	0.7	6.7	243.2	22.3	20.0	133.8
8	-2.4	-7.1	-3.3	-11.9	-5.1	17.3	241.6	182.7	45.9	119.0
Average	-1.2	0.6	-2.4	-2.9	-0.4	6.1	156.8	127.4	45.0	174.5
Within-lab SD ^A	6.8	8.6	10.1	9.2	18.9	7.7	23.5	33.3	21.6	15.1
Between-lab SD	5.4	8.9	13.5	10.2	6.0	7.7	129.7	119.1	49.5	195.8
Coefficient of Variation	724.0	2063.0	703.0	474.0	4957.0	179.0	84.0	97.0	120.0	113.0
Laboratory	Elongation (% Change)									
	Water	Toluene	MEK	Hexane	HCl					
1	0.0	0 to 2	0 to 2	0 to 2	0 to 2					
2	0.0	0.0	0.0	0.0	0.0					
3	-4.3	40.8	-3.2	-6.1	-14.3					
4	-74.2	0 to 5	-27.8	33.5	123.3					
5	-27.7	86.7	78.0	62.1	12.0					
6										
7	-1.1	5.6	-38.9	15.6	16.7					
8	-16.9	65.6	33.3	142.9	27.8					
Average	-17.8	39.7	19.9	41.3	27.6					
Within-lab SD	28.1	62.1	60.4	64.3	45.3					
Between-lab SD	25.0	33.5	34.4	50.7	44.8					
Coefficient of Variation	211.0	178.0	349.0	198.0	231.0					

^ASD = standard deviation.

3. Elongation measurements

- Each original material specimen should be enlarged to at least 7.6 cm × 10.2 cm (3.0 in. × 4.0 in.). Then, from the edge of each original specimen, a 7.6 cm × 1.3 cm (3.0 in. × 0.5 in.) rectangular strip should be cut for the non-contacted elongation measurement. The remainder of each specimen should then be contacted by liquid chemical. This will ensure that the before and after elongation strips are from the same material specimen.
- That the elongation load is a variable should be stated more clearly. The load value determined for non-contacted specimens should be used for the contacted specimens.
- The length of each rectangular strip to be left between the clamps should be specified. This action will ensure consistent lengths available for elongation.

Apparatus

In addition, recommendations for improving the test fixture have been made. They are the following:

- The fixture should be modified to separate the three-cell configuration into single-cell units. This is an important point in pouring off excess liquid chemical at the conclusion of each test.
- The bottom portions of the test fixture should be blocked from direct airflow when set up in an air-exhausted hood. This will prevent volatile chemicals from evaporating and possibly affecting results of the test procedure.

A revision of the degradation test method incorporating most all of these recommendations has been generated.⁽⁴⁾ A smaller-scale, between-laboratory evaluation of this new version is currently underway.

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