



Designation: F1407 – 12 (Reapproved 2017)

Standard Test Method for Resistance of Chemical Protective Clothing Materials to Liquid Permeation—Permeation Cup Method¹

This standard is issued under the fixed designation F1407; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

Workers involved in the production, use, and transportation of chemicals can be exposed to numerous compounds capable of causing harm upon contact with the human body. The deleterious effects of these chemicals can range from acute trauma, such as dermatitis or burns, to chronic degenerative disease, such as cancer or pulmonary fibrosis. Since engineering controls may not eliminate all possible exposures, attention is often given to reducing the potential for direct skin contact through the use of protective clothing that resists degradation, penetration, and permeation.

This test method provides a simple, gravimetric means for measuring the resistance of clothing materials to permeation by liquid chemicals. Permeation testing by a more sophisticated procedure and penetration testing are addressed by separate methods: Test Methods F739 and F903, respectively. Test Methods for measuring the effects of chemicals on the physical properties of rubbers, plastics, and coated fabrics may be found in Test Method D471, Practices D543, and Test Methods D751, respectively. Guide F1001 designates 21 chemicals for use with these tests.

1. Scope

1.1 This test method measures the barrier effectiveness of a specimen of protective clothing upon continuous contact with a liquid.

1.1.1 *Procedure A*—For use when a value for the cumulative amount of chemical permeated in 1 h is desired.

1.1.2 *Procedure B*—For use when breakthrough detection time and permeation rate values are desired.

1.2 Although not addressed herein, the effect of the test chemical on the clothing material can be determined by comparing the weight or other physical properties of the specimen before and after the permeation test.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions to inch-pound units that are provided for information only and are not considered standard.

1.4 *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 applica-*

bility of regulatory limitations prior to use. Specific precautionary statements are given in Section 7.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D471 Test Method for Rubber Property—Effect of Liquids
D543 Practices for Evaluating the Resistance of Plastics to Chemical Reagents

D751 Test Methods for Coated Fabrics

E105 Practice for Probability Sampling of Materials

F739 Test Method for Permeation of Liquids and Gases through Protective Clothing Materials under Conditions of Continuous Contact

F903 Test Method for Resistance of Materials Used in Protective Clothing to Penetration by Liquids

F1001 Guide for Selection of Chemicals to Evaluate Protective Clothing Materials

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.

2.2 Federal Standard:
No. 191, Method 5030.2 Measurement of the Thickness of Materials³

3. Terminology

3.1 Definitions:

3.1.1 *breakthrough detection time, n*—the elapsed time measured from the initial exposure to the test chemical to the sampling time that immediately precedes the sampling time at which the test chemical is first detected.

3.1.1.1 *Discussion*—In this method, the sampling time is the time of weighing.

3.1.2 *cumulative permeation, n*—the total mass of chemical that permeates a specific area of the protective clothing material during a specified time from when the material is first contacted by the test chemical.

3.1.3 *degradation, n*—a 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 item 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 material 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 items 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 *protective clothing, n*—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.

3.1.7 *steady-state permeation rate, n*—a constant rate of permeation that occurs after breakthrough when all forces affecting permeation have reached equilibrium.

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

3.1.8.1 *Discussion*—In this test method, the test chemical is a liquid and can be either one component (that is, a neat liquid) or have several components (that is, a mixture).

3.1.9 *time interval, n*—the time between weighings of the permeation cup.

³ Available from Standardization Documents Order Desk, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098, <http://dodssp.daps.dla.mil>.

4. Summary of Test Method

4.1 The resistance of a protective clothing material to permeation by a test chemical is assessed by measuring the cumulative permeation, breakthrough detection time, and permeation rate through replicate specimens of the material.

4.2 A clothing material specimen is secured over the mouth of a shallow cup that holds the test chemical. The normally outside surface of the material faces the chemical; the other side is open to the atmosphere. See Fig. 1.

4.3 The cup assembly is weighed, inverted, and reweighed at predetermined time intervals to determine the amount of chemical that permeates the material and subsequently evaporates to the atmosphere.

4.4 Based on the amount of chemical lost from the cup during the exposure period, the breakthrough detection time, permeation rate, and cumulative mass of chemical permeating the clothing material are calculated.

4.5 Detection of permeation requires sufficient volatility of the test chemical, and the appropriate combination of analytical balance and weighing interval.

4.5.1 See Section 10 for volatility test.

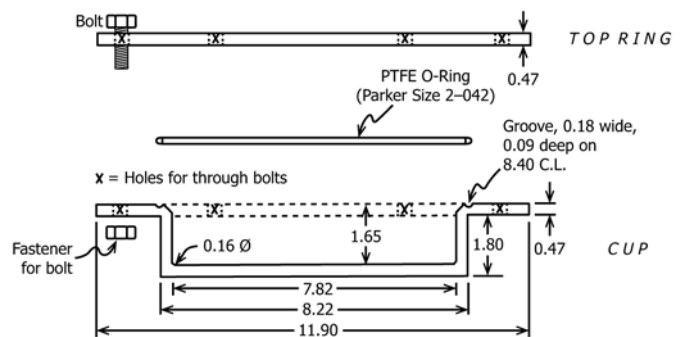
4.5.2 The likelihood of detecting chemical permeation increases as (1) the sensitivity of the balance is increased, and (2) the time between weighings and the length of the test are increased.

4.6 In extreme cases the chemical could so severely degrade the clothing material that the chemical will drip or otherwise flow from the inverted cup.

5. Significance and Use

5.1 This test method establishes a standard procedure for rapidly (in 1 h or less) determining the chemical resistance of specimens of protective clothing materials. This test method can be used to rank materials as to their suitability for use with liquids of known or unknown composition.

5.2 The breakthrough detection time, permeation rate, or cumulative permeation can be used to identify protective clothing materials that are more likely to limit potential exposures to chemicals. Longer breakthrough detection times and lower cumulative amounts permeated and permeation rates are characteristics of materials that are better barriers to the test chemical.



NOTE 1—Dimensions in centimetres.

FIG. 1 ASTM Permeation Cup

5.3 In general this test method is less sensitive than Test Method **F739** coupled with sensitive analytical procedures. In cases where the chemical of concern is highly toxic and contact of even a very small amount with the skin may be detrimental to health, the permeation cup method is not recommended. Use Test Method **F739**.

5.4 Upon permeating the clothing material, the chemical must evaporate in order for a weight loss to occur and permeation to be detected. Consequently, the test method may not be applicable for chemicals having low volatility (that is, vapor pressure). The vapor pressure below which this test method is not applicable has not been determined.

5.4.1 A procedure for assessing volatility is described in Section **10**.

5.5 The results of this test method are highly dependent on the test temperature. If the objective is to compare different clothing materials, all tests shall be conducted at the same temperature (± 3 °C).

6. Apparatus

6.1 *Thickness Gauge*, suitable for measuring thickness to the nearest 0.02 mm (0.001 in.), as specified in Fed. Std. No. 191, Method 5030.2, shall be used to determine the thickness of each material specimen tested.

6.2 *Analytical Balance*, readable and reproducible to at least ± 1 mg, is required for measuring the weight loss of the permeation cup. The capacity of the balance should be at least 50 g more than the weight of an empty permeation cup.

6.3 *Permeation Cup*, shown in **Fig. 1**, is constructed of metal (aluminum is recommended) and is composed of the cup, O-ring (PTFE is recommended), retaining ring (aluminum is recommended), and six fasteners (stainless steel is recommended) for securing the retaining ring to the cup. The circular opening in the retaining ring has a diameter of 7.8 ± 0.05 cm, corresponding to an area of 47.8 cm².

6.3.1 Materials other than aluminum may be used in the construction of the permeation cup. Such materials may be desirable for tests involving chemicals which are incompatible with aluminum (for example, acids).

6.3.2 A chemically resistant coating may be applied to the permeation cup to increase its range of chemical compatibility. Polytetrafluoroethylene (PTFE) has been used successfully as a coating.

6.3.3 In place of the O-ring, either the cup or the retaining ring may be machined such that a seal is achieved when the retaining ring is fastened to the cup.

6.3.4 The volume available for the test liquid is approximately 80 mL, although this amount of chemical is not required nor recommended for performing the test.

6.4 *Torquing Device*, to seal the retaining ring to the cup.

6.5 *Lubricant*, to prevent galling between the fasteners and the cup.

7. Safety Precautions

7.1 Before carrying out this test method, safety precautions recommended for handling any potentially hazardous chemical should be identified and reviewed to provide full protection to all personnel.

7.1.1 For carcinogenic, mutagenic, teratogenic, and other toxic (poisonous) chemicals, the work area should be isolated, well ventilated, and meticulously clean. Involved personnel should be outfitted with appropriate protective clothing and equipment.

7.1.2 For corrosive or otherwise hazardous chemicals, involved personnel should, at a minimum, be outfitted with protective clothing and equipment.

7.2 Provisions should be made for handling spills of the test chemical or splashes to the eyes or skin.

7.3 Appropriate procedures for the disposal of chemicals should be followed.

7.4 The test should be performed in a laboratory fume hood or outdoors.

NOTE 1—In order to obtain stable readings from the analytical balance, it may be necessary to shield the balance or at least its pan from air currents.

8. Test Specimen

8.1 A protective clothing material specimen may consist of either a single layer or a composite of multiple layers that is representative of an actual protective clothing construction with all layers arranged in proper order. In each test, the specimen's normally outer surface shall contact the test chemical.

8.1.1 If, in a proposed design of an article of protective clothing, different materials or thicknesses of materials are specified at different locations, specimens from each location shall be tested.

8.1.2 If, in a proposed design of an article of protective clothing, seams are specified, additional specimens containing such seams shall be tested. Care must be taken to ensure that the permeation cup can be properly sealed when specimens of nonuniform thickness are tested.

8.2 Each material specimen shall have a diameter at least 5 mm greater than the outside diameter of the O-ring or sealing mechanism.

8.3 A minimum of three specimens shall be tested for each material, composite, area (in the case of a heterogeneous design), or other condition. Random specimens may be generated as described in Practice **E105**.

9. Conditioning

9.1 No conditioning of the test specimen is required.

10. Volatility Test

10.1 Weight loss from the cup is the result of chemical diffusion to and evaporation from the surface of the clothing specimen that faces the air. If the volatility of the chemical is too low, the diffusing chemical will not evaporate from this surface of the specimen as rapidly as it arrives there. In the

extreme case, there might be no weight loss even though the chemical has diffused through the specimen.

10.2 Evaporation rate increases with temperature.

10.3 Detection of evaporation is dependent on the sensitivity of the balance and the surface area available for evaporation.

10.4 To determine whether the volatility of the test chemical is sufficient for the permeation cup method to be applicable, use the following test:

10.4.1 Place about 20 g of the test chemical into the cup.

10.4.2 Place the cup (uncovered) on the balance, being careful not to spill the chemical.

10.4.3 Record the weight of the cup.

10.4.4 Leave the cup on the balance for the shortest weighing time interval that will be used during the test. The shortest time interval may be the entire duration of the test if cumulative weight loss is the test objective (see Procedure A).

10.4.5 At the end of the period, record the weight of the cup. If the weight loss is >10 mg, then the chemical has sufficient volatility for the permeation cup test to be performed.

10.4.5.1 Because volatility is dependent on temperature and the detection of evaporation is dependent on the sensitivity of the balance, it may be necessary to rerun the volatility test each time any one of these parameters is changed.

10.4.5.2 If no evaporation is detected, consider lengthening the minimum time interval and rerunning the volatility test.

NOTE 2—When the test liquid is a mixture, weight loss during the volatility test may be due to the evaporation of a component that may not permeate the clothing specimen.

11. Procedure A – Cumulative Permeation

11.1 Cut the test specimen from the clothing material as described in 8.2. Smaller sizes will result in leaking; larger sizes will hinder sealing the cup.

11.2 Measure the thickness of the specimen to the nearest 0.02 mm (0.001 in.) and record.

11.3 Add approximately 25 mL of the test chemical to the cup.

11.4 Center the material specimen over the O-ring, taking care that the specimen does not contact the chemical. The normally outside surface of the chemical protective material shall face the test chemical.

NOTE 3—To avoid obtaining an erroneously high cumulative permeation, the procedures described in 11.4 – 11.6.2 should be performed as quickly as possible.

11.5 Place the retaining ring over the specimen. Tighten the fasteners in a uniform manner in order to avoid bending the ring due to uneven stresses. A torque of 6 in.-lb is recommended.

NOTE 4—A small amount of lubricant may be applied to the threads of the fasteners to reduce the likelihood of galling.

11.6 *Weighing Sequence:*

11.6.1 Weigh the cup immediately. This weight is the initial weight and is used in the calculation of the cumulative permeation.

11.6.2 Invert the cup and begin timing. This starts the test.

NOTE 5—During the test, the inverted cup assembly should be positioned so that the material specimen is free to swell, sag, or pucker. The inverted cup should be elevated off the counter, floor, or ground at least 2.5 cm (1 in.) by a stand that allows air to freely pass under the cup. Air flow is necessary for evaporation to occur. Ideally the test will be performed in a laboratory fume hood or outdoors (see **Note 1**).

11.7 After 1 h return the cup to its upright orientation and reweigh the cup. This weight will be used along with the initial weight to calculate the cumulative permeation.

11.8 Disassemble the cup.

11.9 Remove the material specimen and note any changes in its appearance.

11.10 A minimum of three specimens per condition, as detailed in 8.1, shall be tested.

12. Procedure B – Breakthrough Detection Time and Permeation Rate

12.1 Cut the test specimen from the clothing material to the dimension prescribed in 8.2. Smaller sizes will result in leaking; larger sizes will hinder sealing the cup.

12.2 Measure the thickness of the specimen to the nearest 0.02 mm (0.001 in.) and record.

12.3 Add approximately 25 mL of the chemical to the cup.

12.4 Center the material specimen over the O-ring, taking care that the specimen does not contact the chemical. The normally outside surface of the chemical protective material shall face the test chemical.

NOTE 6—To avoid obtaining an erroneously short breakthrough detection time, the procedures described in 12.4 – 12.6.2 should be conducted as quickly as possible.

12.5 Place the retaining ring over the specimen. Tighten the fasteners in a uniform manner in order to avoid bending the ring due to uneven stresses. A torque of 6 in.-lb is recommended.

NOTE 7—A small amount of lubricant may be applied to the threads of the fasteners to reduce the likelihood of galling.

12.6 *Weighing Sequence:*

12.6.1 Weigh the cup immediately. This weight is the initial weight on which calculations of permeation will be based.

12.6.2 Invert the cup and begin timing. This starts the test.

NOTE 8—During the test, the inverted cup assembly should be positioned so that the material specimen is free to swell, sag, or pucker. The inverted cup should be elevated off the counter, floor, or ground at least 2.5 cm (1 in.) by a stand that allows air to pass freely under the cup. Air flow is necessary for evaporation to occur. Ideally the test will be performed in a laboratory fume hood or outdoors (see **Note 1**).

12.6.3 At predetermined times, return the cup to its upright orientation and reweigh the cup.

12.6.3.1 For a 1-h test, the recommended elapsed times from the start of the exposure (that is, from the time the cup is inverted) at which the cup should be reweighed are 3, 6, 9, 12, 15, 20, 25, 30, 40, 50 and 60 min.

12.6.3.2 The time interval between weighings shall never decrease during the test.

12.6.4 Record the weight and time.

12.6.5 Invert the cup.

12.7 Repeat the weighing sequence until one or more of the following conditions is met:

12.7.1 A predetermined time has passed; 1 h is suggested as the minimum test duration.

12.7.2 The weight losses per unit time have stopped increasing and are approximately the same from interval to interval (that is, steady-state permeation has been reached).

12.7.3 The weight losses per unit time continue to increase from interval to interval, eventually surpassing a predetermined value.

12.7.4 The weight loss per unit time for each interval reaches a maximum and decreases by more than 50 %.

12.8 Disassemble the cup.

12.9 Remove the material specimen and note any changes in its appearance.

12.10 A minimum of three specimens per condition, as detailed in 8.1, shall be tested.

13. Calculation

13.1 Calculate the method sensitivity as follows:

$$\text{Method Sensitivity} = (R/At) \times 10^6 \quad (1)$$

where:

R = value representing the reproducibility of the balance, g,
 A = surface area of the opening in the retaining ring, cm²,
 and

t = time; in Procedure A, the duration of the test, min, and in Procedure B, the length of the time interval between the breakthrough detection time and the weighing time immediately following it. If no permeation is detected, the duration of the longest time interval, min.

13.2 Calculate the cumulative permeation as follows:

$$CP = \frac{W_i - W_f}{A} \times \frac{10^6 \mu\text{g}}{\text{g}} \quad (2)$$

where:

CP = cumulative permeation for a specified test material and chemical, μg/cm²,

W_i, W_f = weight measurement, i = initial, f = final, g, and
 A = surface area defined by the opening in the retaining ring, cm².

13.3 Calculate the average permeation rate during each time period as follows:

$$PR_i = \frac{W_{gi} - W_{ei}}{At_i} \times \frac{10^6 \mu\text{g}}{\text{g}} \quad (3)$$

where:

PR_i = average permeation rate for a time interval, t_i , μg/cm²/min,

t_i = duration of time interval, min,

W_{gi} = weight of cup assembly at start of interval, g,

W_{ei} = weight of cup assembly at end of interval, g, and

A = surface area defined by the opening of the retaining ring, cm².

NOTE 9—The following factors are useful in converting permeation rates: 1 μg/cm²/min = 0.17 mg/m²/s = 10 mg/m²/min.

13.4 Steady state permeation rate is defined as the average value of three successive PR_i s obtained with time intervals of at least 5 min and with the third PR_i no more than 20 % greater or less than the first PR_i .

13.5 The breakthrough detection time is determined based on the definition in 3.1.2 and from inspection of the PR_i calculated in 13.3. As examples, consider the two following sets of data for tests performed with a retaining ring having an open area of 50 cm².

13.5.1 *Set 1*—The breakthrough detection time is 3 min.

Time, min	0	3	6	9	12	15	20
Cup weight, g	100	100	99	97.5	95.5	93	88
Permeation rate, μg/cm ² /min	0	0	6667	10 000	13 333	16 667	20 000

13.5.2 *Set 2*—The breakthrough detection time is 9 min.

Time, min	0	3	6	9	12	15	20
Cup weight, g	100	100	99	100	99	97.5	94
Permeation rate, μg/cm ² /min	0	0	6667	-6667	6667	10 000	14 000

14. Report

14.1 Identify and report the material tested, including generic name, manufacturer, product name, nominal thickness, measured thickness, and a general description of the location from which the specimen was taken (for example, palm or back of glove).

14.2 Identify the test chemical, including generic name, source, and lot number in cases where the test chemical is of known composition.

14.3 Report the test temperature (°C) (see 5.5).

14.4 Report the test duration in hours.

14.5 *Procedure A*:

14.5.1 Report the average of the three cumulative permeations measured for each combination of clothing material and chemical.

14.5.2 If no permeation is detected, report the method sensitivity.

14.6 *Procedure B*:

14.6.1 Report the breakthrough detection time and method sensitivity for each test.

14.6.2 If no chemical permeation is detected, report the method sensitivity.

14.6.3 Report the average of the three cumulative permeations measured for each combination of clothing material and chemical.

14.6.4 Report a plot of PR_i versus exposure time for each specimen tested. For each time interval, use the midpoint of the

interval as time coordinate. For example, plot the PR_i for the interval of 30 to 40 min at 35 min.

14.6.5 If steady-state permeation is found (see 13.4), report the average steady-state permeation rate for the material tested along with the steady-state permeation rate for each specimen tested.

14.6.6 If steady-state permeation is not found, report the average permeation rate for the three specimens during the last time interval.

15. Precision and Bias

15.1 This test method has been the subject of an interlaboratory test involving ten independent laboratories. The test chemical was acetone (ACS grade) and the material was a 16-mil thick neoprene rubber sheet. The results are summarized in Table 1.

15.2 The average and within-laboratory standard deviation for measuring steady-state permeation rate of acetone through the neoprene was $249.77 \pm$ (to be calculated) $\mu\text{g}/\text{cm}^2/\text{min}$. The between-laboratory standard deviation was $\pm 31.6 \mu\text{g}/\text{cm}^2/\text{min}$.

15.3 The average and within-laboratory standard deviation for measuring cumulative amount of acetone permeating the neoprene in 1 h was $11\,575 \pm$ (to be calculated) $\mu\text{g}/\text{cm}^2$. The between-laboratory standard deviation was \pm (to be calculated) $\mu\text{g}/\text{cm}^2$.


15.4 The average and within-laboratory standard deviation for measuring breakthrough detection time for acetone permeating the neoprene was (to be determined) \pm (to be calculated) minutes. The between-laboratory standard deviation was \pm (to be calculated) min.

TABLE 1 Interlaboratory Test Using Acetone and Neoprene Rubber Sheet

Laboratory No.	Neoprene Thickness, in.	Temperature, °C	Breakthrough Detection Time, min	Method Sensitivity, $\mu\text{g}/\text{cm}^2/\text{min}$	Steady-State Permeation Rate, $\mu\text{g}/\text{cm}^2/\text{min}$	Cumulative Permeation in 1 h, $\mu\text{g}/\text{cm}^2$
1	0.015	21	12	0.71	196	8714
	0.015	21	9	0.71	199	8669
	0.015	19	12	0.71	211	8080
2	0.017	25	9	0.71	248	10 866
	0.016	25	3	0.71	249	11 224
	0.016	24	6	0.71	254	12 103
3	0.016	19	12	0.71	213	8919
	0.016	19	9	0.71	218	9778
	0.016	19	12	0.71	201	8785
4	0.016	23	9	0.71	247	11 216
	0.016	24	9	0.71	241	10 831
	0.016	23	6	0.71	244	11 173
5	0.016	25	3	0.71	316	15 230
	0.016	25	3	0.71	287	14 288
	0.016	25	3	0.71	293	14 194
6	0.017	22	6	0.71	243	9370
	0.016	22	9	0.71	261	11 451
	0.017	22	6	0.71	237	10 308
7	0.017	22	15	0.43	237	10 208
	0.016	21	6	0.71	234	10 479
	0.016	21	6	0.71	239	10 857
8	0.016	23	6	0.71	277	14 254
	0.016	23	6	0.71	287	14 307
	0.016	24	6	0.71	251	14 015
9	0.015	25	3	0.71	296	14 106
	0.015	25	9	0.71	274	12 417
	0.015	25	3	0.71	304	14 908
10	0.015	23	6	7.1	231	12 605
	0.015	23	9	7.1	238	10 660
	0.016	23	0	7.1	267	13 245

16. Keywords

16.1 chemical protective clothing; liquid permeation; permeation; permeation cup method

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