



CHEMICAL WARFARE SERVICE  
Edgewood Arsenal, Maryland

Manufacturing Process  
Directive No. 16

MANUFACTURING PROCESS DIRECTIVE FOR IMPREGNATING  
PERMEABLE PROTECTIVE CLOTHING

CONTENTS

- I. PURPOSE
- II. GENERAL
- III. APPARATUS
- IV. PROCEDURE
- V. PRECAUTIONS
- VI. TESTS

EDGEWOOD ARSENAL  
MANUFACTURING PROCESS  
DIRECTIVE NO. 16

MANUFACTURING PROCESS DIRECTIVE FOR IMPREGNATING PROTECTIVE  
PERMEABLE CLOTHING

I. PURPOSE.

To impregnate service clothing as listed in C.W.S. Drawing No. C5-31-1 with materials which will render the fabric resistant to the penetration of HS and other similar chemical agents.

II. GENERAL.

The impregnation process consists essentially in the immersion of the garments to be treated, in a heated solution of impregnating materials, allowing time for complete saturation and finally removing the excess solution and drying the articles of clothing. After impregnation, the garments are tested for Impregnate I content and resistance to HS.

III. APPARATUS.

1. The following apparatus was used at Edgewood Arsenal, and found satisfactory in principle:

- a. Mixing tank, copper lined, 200-gallon capacity, with rotating steam coil, copper covered.
- b. Impregnator, washer 28-in. x 40-in., with rotating drum, American Laundry Machinery Company.
- c. Centrifuge, extractor, 30-in. diameter, under driven.
- d. Dryer, drying tumbler, 40-in. x 64-in., American Laundry Machinery Company.
- e. Storage Tank, iron, 300-gallon capacity for storing used (deteriorated) solution.
- f. Pump, LaBour centrifugal.
- g. Thermometers 3, precision, metal shield (2), 25-in. stem, 10-110°C. and (1) angle 10-140°C.

February 7, 1955

- 1 -

Appendix C

#### IV. PROCEDURE.

##### 1. Preparing the Solution:

a. Material. The following materials are used in making up the solution:

- (1) Impregnite I, U.S. Army Specification No. 97-54-51.
- (2) Chlorinated paraffin, U.S. Army Specification No. 4-503-127.
- (3) Acetylene Tetrachloride, U.S. Army Specification No. 4-502-12.

b. Mixing. The Impregnite I and chlorinated paraffin in equal weights shall be mixed with acetylene tetrachloride (see Table 1). The three materials are placed in a tank, and steam at about 5 pounds per square inch pressure is passed through a steam coil which is rotated to keep the material thoroughly mixed. When the mixture is thoroughly dissolved and heated to the desired temperature (60°C.) it is ready to be passed into the impregnator.

c. Testing the Solution in Chlorine Content. During the impregnating process, the impregnating solution may be tested for available chlorine to insure the maintenance of proper concentration. Five cc. of the solution is weighed in a glass stoppered Erlenmeyer flask. Fifty cc. of acetylene tetrachloride, 40 cc. of 5 per cent potassium iodide solution and 10 cc. of glacial acetic acid are added. The mixture is titrated immediately against 0.10 N sodium thiosulfate solution, using starch as the indicator.

$$\frac{\text{cc. 0.10 N thiosulfate} \times 0.0122 \times 100}{\text{weight of sample}} = \text{per cent Impregnite I in solution.}$$

##### 2. Impregnation:

a. Saturating. The garments to be impregnated are placed in the impregnator and the impregnating solution run in, in sufficient quantities to completely saturate the garments. After standing for about 10 minutes, the solution is drawn off and the garments are rotated and tumbled and allowed to drain until the fabric is free from unabsorbed liquid. This operation also requires about 10 minutes.

PROCEDURE (CONT'D)

b. Centrifuging. The garments are next transferred to the centrifuging machine and rotated at 350 r.p.m. until all the solution in excess of the required amount is removed from the garments.

c. Drying. The garments are next rotated and tumbled in a current of heated air until dry. Before drying, the impregnated garments must not be subjected to sun or other strong light, as such exposure tends to discolor the clothing.

TABLE 1

The following table shows the percentage composition by weight of the impregnating solution and the time of centrifuging for the various materials or garments listed:

Garment or Material	Impregnite I %	Chlorinated Paraffin %	Acetylene Tetrachloride %	Time of Centrifuging Minutes
Gloves, cotton, olive drab.	: 8.0	: 8.0	: Remainder	: 5
Gloves, woolen.	: 10.0	: 10.0	: "	: 2
Breeches, cotton.	: 9.5	: 9.5	: "	: 1
Breeches, woolen.	: 11.5	: 11.5	: "	: 1
Shirt, Cotton.	: 9.5	: 9.5	: "	: 1
Shirt, woolen.	: 10.0	: 10.0	: "	: 1
Underwear, cotton.	: 9.5 to 10.5	: 9.5 to 10.5	: "	: 5 to 10
Underwear, woolen.	: 9.0	: 9.0	: "	: 2
Stockings, light woolen.	: 10.0	: 10.0	: "	: 4
Stockings, heavy woolen.	: 7.0	: 7.0	: "	: 5
Leggins, canvas.	: 12.0	: 12.0	: "	: 1
Hoods, cotton.	: 9.0	: 9.0	: "	: 3
Coverall, cotton.	: 10.0	: 10.0	: "	: 1
Belts, Web	: 9.0	: 9.0	: "	: 2



V. PRECAUTIONS.

As the acetylene tetrachloride is toxic, every precaution should be taken to prevent an accumulation of the fumes of the impregnating solution. This can be done by means of fans, hoods, tanks, coverings or other protective measures.

VI. TESTS.

1. Requirements for Impregnation and Mustard Resistance:

a. Protective Treatment. The amount of Impregnite I remaining in the cloth after impregnation of the various articles of clothing shall be as follows:

(1)	<u>Gloves, Cotton or Woolen.</u> Impregnite I - - - - -	8.7 + 1%
(2)	<u>Breeches</u> Impregnite I in cotton - - Impregnite I in woolen - -	9.2 + 1% 8.7 + 1%
(3)	<u>Shirts.</u> Impregnite I in cotton - - Impregnite I in woolen - -	9.2 + 1% 9.8 + 1.1%
(4)	<u>Underwear.</u> Impregnite I in cotton - - Impregnite I in woolen - -	8.7 + 1% 7.4 + 1%
(5)	<u>Stockings, Light or Heavy Woolen.</u> Impregnite I in Light weight - - Impregnite I in heavy weight - -	9.8 + 1.1% 8.7 + 1%
(6)	<u>Leggins, Canvas.</u> Impregnite I - - - - -	7.8 + 1%
(7)	<u>Hood, Cotton.</u> Impregnite I - - - - -	9.2 + 1%
(8)	<u>Coverall, Cotton.</u> Impregnite I - - - - -	9.2 + 1%
(9)	<u>Belts, Web.</u> Impregnite I - - - - -	7.8 + 1%

b. Mustard Resistance. The impregnated cloth shall resist penetration by mustard vapor for at least the duration of time stated for the various articles of clothing when mustardized air at a concentration of 0.50 mg. of mustard per liter is drawn through the cloth at a linear velocity of 4.4 cm. per minute;

TESTS (CONT'D)

- |     |                             |                    |
|-----|-----------------------------|--------------------|
| (1) | <u>Gloves.</u>              |                    |
|     | Cotton - - - - -            | 7-1/2 hr.          |
|     | Woolen - - - - -            | 9-1/4 hr.          |
| (2) | <u>Breeches.</u>            |                    |
|     | Cotton - - - - -            | 5 hr.              |
|     | Woolen - - - - -            | 7-1/4 hr.          |
| (3) | <u>Shirts.</u>              |                    |
|     | Cotton - - - - -            | 5 hr.              |
|     | Woolen - - - - -            | 4-1/2 hr.          |
| (4) | <u>Underwear.</u>           |                    |
|     | Cotton - - - - -            | 3-1/2 hr.          |
|     | Woolen - - - - -            | 6-3/4 hr.          |
| (5) | <u>Stockings, Woolen.</u>   |                    |
|     | Light Weight - - -          | 9-3/4 hr.          |
|     | Heavy Weight - - -          | 7-1/4 hr.          |
| (6) | <u>Leggins, Canvas</u> -    | 5 hr.              |
| (7) | <u>Hood, Cotton</u> - -     | 5 hr.              |
| (8) | <u>Coverall, Cotton</u> - - | 5 hr.              |
| (9) | <u>Belts, Web</u> - - - -   | No test specified. |

2. Method of Tests:

a. Samples for Impregnate I and Mustard Resistance Tests:

(1) Gloves, Cotton or Woolen. A sample, one foot square, of the knitted goods, of the same material as the gloves, shall be obtained and impregnated at the same time with each number (size) of 500 pairs of gloves or fraction thereof. The samples of glove fabric shall be treated during the impregnation process, exactly as the gloves, and shall receive no other treatment than the gloves. At least two mustard resistance test samples 4 inches in diameter shall be taken from each one foot square sample of impregnated glove fabric. Each sample shall be tested for mustard resistance. Two additional test samples, each three inches square, shall be cut from the one foot square sample. Each sample shall be tested for Impregnate I.

TESTS (CONT'D)

(2) Breeches, Cotton or Woolen. A sample, one yard square, of the cotton or woolen cloth, of the same material as the breeches, shall be obtained and impregnated at the same time with each batch of 500 breeches or fraction thereof. The samples of the cotton or woolen cloth shall be treated during the impregnation process exactly as the breeches, and shall receive no other treatment than the breeches. Two test samples 3 inches square shall be cut from the yard square sample. Each sample shall be tested for Impregnite I. Two additional test samples 4 inches in diameter shall be cut from the same square yard sample and tested for mustard resistance.

(3) Shirts, Cotton or Woolen. A sample, one yard square, of the cotton or woolen cloth, of the same material as the shirts, shall be obtained and impregnated at the same time with each batch of 500 shirts or fraction thereof. The samples of the cotton or woolen cloth shall be treated during the impregnation process, exactly as the shirts, and shall receive no other treatment than the shirts. Two test samples 3 inches square shall be cut from the square yard sample. Each test sample shall be tested for Impregnite I. Two additional test samples, 4 inches in diameter, shall be cut from the same square yard sample and tested for mustard resistance.

(4) Underwear, Cotton or Woolen. A sample, one yard square, of the cotton or woolen knitted goods, of the same material as the underwear, shall be obtained and impregnated at the same time with each batch of 500 undershirts or drawers or fraction thereof. The samples of the cotton or woolen knitted fabric shall be treated during the impregnation process exactly as the shirts or drawers and shall receive no other treatment. Two test samples 3 inches square shall be cut from the square yard sample. Each sample shall be tested for Impregnite I. Two additional test samples 4 inches in diameter shall be cut from the same square yard sample and tested for mustard resistance.

(5) Stockings, Light and Heavy Woolen. Samples of the impregnated stockings shall be taken in the ratio of one pair of stockings to 500 pairs of stockings or fraction thereof. Test samples 3 inches square shall be cut as follows: One each from the foot of each stocking and one each from the ankle of each stocking. Each test sample shall be tested for Impregnite I. Additional samples 4 inches in diameter of the impregnated stockings shall be taken in the same ratio, and tested for mustard resistance.

TESTS (CONT'D)

(6) Leggins, Canvas. A sample, one foot square, of the canvas cloth, of the same material as the leggins, shall be obtained and impregnated at the same time with each batch of 500 pairs of leggins or fraction thereof. The samples of the canvas fabric shall be treated during the impregnation process, exactly as the leggins and shall receive no other treatment than the leggins. Two test samples, 3 inches square, shall be cut from the foot square sample. Each test sample shall be tested for Impregnite I. Two additional test samples 4 inches in diameter shall be taken from the remaining foot square sample and tested for mustard resistance.

(7) Hood, Cotton. A sample, one foot square, of khaki cotton cloth, of the same material as the hood, shall be obtained and impregnated at the same time with each batch of 500 hoods or fraction thereof. The samples of the khaki cotton cloth shall be treated during the impregnation process, exactly as the hood, and shall receive no other treatment than the hood. Two test samples, 3 inches square, shall be cut from the foot square sample. Each sample shall be tested for Impregnite I. Two additional test samples, 4 inches in diameter, shall be taken from the remaining foot square sample and tested for mustard resistance.

(8) Coverall, Cotton. A sample, one yard square, of the cotton cloth, of the same material as the coverall, shall be obtained and impregnated at the same time with each batch of 250 coveralls or fraction thereof. The samples of cotton cloth shall be treated during the impregnation process exactly as the coveralls and shall receive no other treatment. Two test samples, 3 inches square, shall be cut from the square yard sample. Each test sample shall be tested for Impregnite I. Two additional test samples, 4 inches in diameter, shall be cut from the square yard sample and tested for mustard resistance.

3. Impregnite I Test. The accurately weighed test sample of the impregnated cloth is placed in a 750 cc. Erlenmeyer flask; 50 cc. of chloroform is poured in the flask, 40 cc. of water and 10 cc. of a 10 per cent solution of potassium iodide and 10 cc. of glacial acetic acid are added. The contents of the flask are then titrated with 0.1 normal sodium thiosulfate, using starch as the indicator.

$$\frac{\text{cc. 0.1 normal thiosulfate} \times 1.22}{\text{weight of sample}} = \% \text{ Impregnite I}$$

TESTS (CONT'D)

4. Mustard Resistance Test. The sample of impregnated cloth to be tested is tightly clamped between the two close fitting sections of a circular brass cup (see Dwg. EAE-S-796). The protruding edges of the cloth are closely trimmed and the joint is covered with paraffin to insure an air-tight seal. The mustard vapors in known volume and concentration are passed through the cloth for a measured length of time. The construction of the cup is such that mustard vapors enter the cup at the circumference of one section, pass through the cloth, and out of the cup at the center of the second section. This provides for uniform contact between the test sample of impregnated cloth and the mustard vapors.

Air is passed through a flowmeter to determine its rate of flow; through a small glass chamber carrying a thermometer to determine its temperature; through a glass bubbler containing liquid mustard (immersed in a water bath for temperature control) to saturate it with mustard vapors; through the cup containing the sample of impregnated cloth; and finally, through a bubbler (likewise in a water bath for temperature control) containing a solution of the dichromate indicator.

The temperature of the air of the bubbler, containing the liquid mustard, and of the mustard vapor, is maintained at 25°C. The dichromate indicator, consisting of 10 cc. of 0.002 normal potassium dichromate containing 20 per cent of sulfuric acid, is kept at 80°C. The test is completed when the color of the indicator is discharged.