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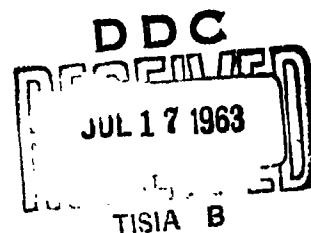
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EVALUATION OF THE IMPREGNITE ANALYZING KIT M-26

24 June 1963



U. S. NAVAL CIVIL ENGINEERING LABORATORY

PORT HUENEME, CALIFORNIA

EVALUATION OF THE IMPREGNITE ANALYZING KIT M-26

Y-F015-99-063

Type C

by

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ABSTRACT

The assigned task was initiated by the Bureau of Yards and Docks by Code 50, and it was recommended by U. S. Naval Research Laboratory (6140-18A GHF 31 January 1962) that the Bureau of Yards and Docks request scientific personnel of its Civil Engineering Laboratory to carry out the evaluation of Impregnite Kit M-26.

A series of tests were performed on the CC2 suspensions to evaluate the M-26 Analyzing Kit. With certain modifications the M-26 Kit can provide a very accurate and convenient way of determining the percent of the active CC2 in suspension.

Swatches of herringbone twill were dipped into the above suspension which contained dye, air dried, and a series of quantitative analyses were performed on discs of the cloth for CC2 content. With certain modifications the M-26 analyzing kit is a suitable device for the determination of CC2 content.

User trials were performed by Navy enlisted men and results were checked for precision.

INTRODUCTION

The impregnate content of treated clothing is degraded rather rapidly in service, sometimes in a few weeks. Consequently, a relatively simple but accurate kit for measuring the active CC2 content of chemical warfare protective clothing and impregnating suspensions is imperative.

Laboratory tests were performed to establish the precision of results using the M-26 kit and following the procedures exactly as outlined in Technical Manual TM 3-6665-202-12 supplied with the kit.

Another series of laboratory tests was performed with some modifications of both procedure and apparatus.

Field tests were performed utilizing enlisted personnel of the Disaster Recovery Training Department, Construction Battalion Center. To compare the precision which can be obtained from this kit, Navy enlisted personnel performed the same tests using the procedure and apparatus with the suggested modifications. By this method a comparison could be made in respect to the precision of the results obtained by a chemically trained person and nonchemically trained personnel.

EXPERIMENTAL

Preparation of Impregnate Suspensions

A chemical set, Clothing Impregnation M-3, Stock #4230-368-6145, packed January 1953, was obtained from NCBC. It contained the following packages:

Package #1	CC2 + ZnO	18	lbs
Package #2	Chlorinated Paraffin	4.25	lbs
Package #3	PVA	1.75	lbs
Package #4	Dye (OD)	1	lb

A suspension of the impregnating material was prepared using a one-hundredth part of the above proportions, weighed in grams. It was mixed per instructions in the packing case (Suspension #1). Another suspension was made per instructions and in the same proportions, except that the dye was omitted (Suspension #2).

Evaluation of the M-26 Kit

Laboratory Tests for the Determination of Percent of CC2 in Clothing Impregnating Suspension

Procedure 1. Determination of percent CC2 in Suspension #1 was made using the apparatus in the M-26 kit and the procedure in Section II of TM 3-6665-202-12. However, the OD dye is so intense that it makes the end point color changes in the analysis very difficult to see and, therefore, this method is not recommended for the determination of CC2 content when dye is included in the suspension. If the CC2 content of a suspension is needed, the sample should be taken before the dye is added. This will work very well since the dye is the last component added.

Procedure 2. In this procedure, Suspension #2 was used. The procedure and apparatus used were from the M-26 kit and TM 3-6665-202-12 with the following exception: The preparation of the Indicator (Starch) Solution described in section 8, page 4 of TM 3-6665-202-12 was modified by triturating the tablet from bottle D in a beaker, adding cold water, and boiling for one minute. The tablet will not go into solution in cold water. The end point used in this procedure and all subsequent procedures is a milky white color and not milky pink as stated in the TM 3-6665-202-12. The results of the analyses are shown in Table I.

Table I. Determination of Percent CC2
in a Suspension of Impregnite not Containing Dye

ml of Test Solution	Percent CC2 in Suspension
2.45	9.3
3.00	11.5
2.50	9.5
3.05	11.8
2.60	10.0

Note: Readings of percent of CC2 in suspension were all taken from the graph, Figure 6 in TM 3-6665-202-12. The graph for percent CC2 in the lid of the kit gives higher readings than Figure 6 in the TM.

Coefficient of Variation from the Data in Table I.

$\bar{x}$	$(x - \bar{x})^2$	$S^2 = \frac{\sum(x - \bar{x})^2}{n - 1}$
9.3	1.21	
11.5	1.21	
9.5	0.81	
11.8	1.96	
10.0	0.16	
5 <u>52.1</u>	<u>5.35</u>	$S^2 = \frac{5.35}{4} = 1.34$
$\bar{x}$ 10.4		$S = \sqrt{1.34} = 1.16$ Standard Deviation

$$\text{Coefficient of Variation } C = 100 \frac{S}{\bar{x}} = 100 \left(\frac{1.16}{10.4} \right) = 11.2\%$$

Procedure 3. The same procedure was followed as procedure 2, except that a 1 ml tuberculin syringe was substituted for the automatic pipette supplied with the M-26 kit. The results are shown in Table II.

Table II. Determination of Percent CC2 in a Suspension of Impregnate Substituting a 1 ml Tuberculin Syringe for the Automatic Pipette

ml of Test Solution	Percent CC2 in Suspension
3.35	13.1
3.35	13.1
3.30	12.8
3.30	12.8
3.35	13.1

Coefficient of Variation from the Data in Table II.

Mean Value of CC2 = 13.0
Standard Deviation = 0.17
Coefficient of Variation = 1.3%

The method used for obtaining these values for this and succeeding tables is the same as that presented after Table I.

Procedure 4. The procedure is the same as #3 except that 1 ml of CC2 suspension was used instead of the 0.5 ml required by the method in the TM. The percentages were obtained by dividing the ml of test solution used by 2 in order that the percentage CC2 readings could be taken directly from the graph.

The results are shown in Table III.

Table III. Determination of Percent CC2 in a Suspension of Impregnite using 1 ml Test Sample and Double Quantities of Reagents

ml of Test Solution	Percent CC2 in Suspension
6.55	12.6
6.50	12.5
6.50	12.5
6.45	12.4

Coefficient of Variation from the Data in Table III.

Mean Value of CC2	= 12.5
Standard Deviation	= 0.1
Coefficient of Variation	= 0.8%

Laboratory Tests for the Determination of CC2 Content in Impregnated Herringbone Twill Cloth

Procedure 1. Samples of herringbone twill cloth were dipped into suspension #1 and air dried. The procedure and apparatus were exactly as outlined in TM 3-6665-202-12. The "Exact Method" (Paragraph 14c, Section IV) was followed. The CC2 content was determined by using Figure 7 in the TM.

Table IV. Determination of CC2 Content in Herringbone Twill Cloth

ml of Test Solution	mg CC2/cm ²
2.3	9.5
1.9	7.8
2.5	10.5
2.4	10.0
2.5	10.5
2.3	9.5

Coefficient of Variation from the Data in Table IV.

Mean Value of CC2	=	9.6
Standard Deviation	=	1.0
Coefficient of Variation	=	10.4%

Procedure 2. The "Exact Method" outlined in TM 3-6665-202-12 was followed, except that double quantities were used and bottle F was replaced by one twice its size. The CC2 content of a Herringbone Twill sample taken from an impregnated stock garment was determined by dividing the Impregnate in fabric, mg/cm² obtained from Figure 7 in the TM, by a factor of two. The results are shown in Table V.

Table V. Determination of CC2
Content in Herringbone Twill Cloth

ml of Test Solution	mg CC2/cm ²
2.40	5.0
2.80	5.7
2.60	5.4
2.50	5.1

Coefficient of Variation from the Data in Table V.

Mean Value of CC2	=	5.3
Standard Deviation	=	0.32
Coefficient of Variation	=	6.0%

Procedure 3. The method used in procedure 2, page 5 of this report, was used except that the stock garment had been impregnated more recently. The results are shown in Table VI.

Table VI. Determinations of CC2 Content in mg/cm²
of Herringbone Twill Cloth, Impregnated More Recently, Using
Double Quantities

ml of Test Solution	mg CC2/cm ²
3.45	7.1
3.40	7.0
3.40	7.0
3.50	7.3
3.30	6.9

Coefficient of Variation from the Data in Table VI.

Mean Value of CC2	= 7.1
Standard Deviation	= 0.16
Coefficient of Variation	= 2.3%

Field Test of the Determination of the Percent of CC2 in Clothing Impregnating Suspension

Procedure 1. This was a field trial of the M-26 kit performed by enlisted personnel after a period of instruction by a chemist. The procedure was that described in TM 3-6665-202-12 except that a 1 ml syringe was used to draw the 0.5 ml sample of suspension from bottle E. The results are shown in Table VII.

Table VII. Field Trial Determination of the CC2 Content in a Suspension of Impregnite Using a 1 ml Syringe and Performed by Enlisted Personnel

ml of Test Solution	Percent CC2 in Suspension
3.30	13.0
3.45	13.4
3.25	12.5
3.50	13.5

Coefficient of Variation from the Data in Table VII.

Mean Value of CC2	= 13.0
Standard Deviation	= 0.6
Coefficient of Variation	= 4.6%

Field Test for the Determination of CC2 Content in Impregnated Herringbone Twill Cloth.

Procedure 1. The "Exact Method" in the TM 3-6665-202-12 was followed with the exception that all quantities were doubled and titrated to an end point of milky white. The OD dye was present but the milky white color could be easily observed. The value of mg/cm² obtained from Figure 7 was divided by a factor of 2. The results are shown in Table VIII.

Table VIII. Field Trial Determinations of the
CC2 Content in mg/cm² of Herringbone Twill Cloth, Impregnated
more recently, Using Double Quantities and Performed by Enlisted
Personnel

ml of Test Solution	mg CC2/cm ²
3.45	7.1
3.60	7.4
3.40	7.0
3.20	6.6

Coefficient of Variation from the Data in Table VIII.

Mean Value of CC2	= 7.0
Standard Deviation	= 0.33
Coefficient of Variation	= 4.7%

DISCUSSION

General Remarks Concerning the Operation, Design and Components of the
Impregnite Analyzing Kit M-26

1. The graphs, Figures 6 and 7, on pages 8 and 9 of the TM 3-6665-202-12 are too small for accurate and fast reading. This deficiency could be corrected preferably by expanding the graphs or presenting the information in tabular form.
2. After a few titrations the seal in the cap of bottle F decomposes and there is loss of solvent A (chlorobenzene and acetic acid). This solvent is volatile, flammable and toxic.
3. The TM 3-6665-202-12 does not explain either the location of, or use of, the plastic holder for bottle A. It is located on top of the foam packing and is the first component seen when the lid is removed. The function of this holder is to hold bottle A during the titration.
4. The automatic burette found in the kit is the smallest standard size listed in any scientific catalogue.

5. Instructions for the correct method of reading a burette are not contained in the TM 3-6665-202-12. Readings should be obtained by reading the values from the bottom of the meniscus. Example:

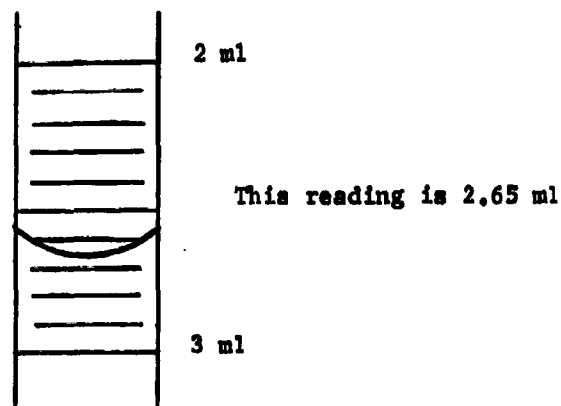


Figure 1. Section of a burette

Precision of the Results Obtained in the Various Procedures

The CC2 content of an Impregnate suspension as outlined in procedure 2, page 2 of this report was obtained with a coefficient of variation of 11.2. In procedure 3, page 3 of this report, the automatic pipette was replaced by a 1 ml tuberculin syringe and the same procedure was followed. The coefficient of variation was decreased from 11.2 to 1.3. This increase in precision more than justifies the substitution of the tuberculin syringe for the automatic pipette.

Using procedure 4, page 4 of this report the quantities were doubled to obtain a better sampling and the coefficient of variation was reduced from 1.3 to 0.8. This small increase in precision does not justify the doubling of quantities. Procedures in the TM 3-6665-202-12 concerning the quantities of reagents and materials for the determination of the CC2 content of an Impregnate suspension are adequate.

In the determination of CC2 in impregnated clothing, procedure 2, page 5 of this report, the coefficient of variation was reduced from 10.4 to 6.0. Thus, when the CC2 content of impregnated clothing is to be obtained, the doubling of quantities may be justified in order to increase the precision.

The precision obtained in the field tests, as outlined in procedure 1, page 6 of this report clearly indicates that Navy enlisted men can operate this kit with efficiency and accuracy. In the determination of CC2 in an

Impregnite suspension, the coefficient of variation was 4.6 in comparison to 1.3 obtained by a chemist under laboratory conditions.

By doubling the quantities of reagents and material, Navy enlisted men were able to obtain the CC2 content of impregnated clothing with a coefficient of variation of 4.7 in comparison to 2.3 obtained by a chemist.

CONCLUSIONS

After carrying out the various laboratory and field tests described in the procedures, certain observations were made and conclusions reached as presented below...

1. After extended storage, starch tablets (bottle D) are difficult to dissolve in cold water and therefore must be ground to a powder and put in solution with boiling water.
2. The automatic pipette can be replaced by a tuberculin syringe of either a 1 ml or 2 ml capacity. The automatic pipette is too expensive and complicated, and when set for a certain volume of suspension it will not withdraw consistently the same volume. The syringe is much more accurate, simpler, less expensive, and easier to operate and clean. There is no adjustment necessary. The increase in precision of the results in Table II over Table I justifies the change. If broken or damaged, the syringe would be easier to replace in a remote area than the automatic pipette.
3. The graphs in TM 3-6665-202-12, Figures 6 and 7, are too small to get a fast and accurate reading.
4. The cap of bottle F should have a liner (seal) which is resistant to the solvent (chlorobenzene and acetic acid solution). The present seal soon decomposes and upon shaking there is some loss of contents.
5. Except for the deficiencies noted above, the components of the kit are used to good advantage. Ample reagents are included.
6. The instructions are clear and adequate for all normal service with the exception of paragraph "a" of topic 12, "Test Procedure," which should read, "Stop adding test solution from the burette when the liquid in bottle F suddenly turns from dark purplish black to a milky white," not "milky pink."
7. The kit and instructions are sufficiently versatile and adaptable to maintain reliable production of information under unforeseen and unfavorable circumstances such as loss of parts and shortage of standard replacements with the exception of the automatic burette. This would be most difficult to improvise if it were broken.

8. In the determination of CC2 content in clothing, the dye present will not interfere with the determination.
9. In the determination of CC2 content in a suspension containing the OD dye, the color is so intense that the end point changes in the analysis cannot be detected and therefore this method is worthless.
10. In the determination of CC2 content of clothing doubling the quantities decreases the coefficient of variation.
11. Results of field trial determinations, Table VII and Table VIII, are convincing evidence that enlisted personnel can perform these tests with precision.

RECOMMENDATIONS

It is recommended that the Impregnite Analyzing Kit M-26 be accepted for field use if the following requirements are met:

1. The starch tablet from bottle D be ground and dissolved by a volume of boiling water equal to the volume of dropping bottle C. (Note: only cold water should be poured into bottle C to prevent breakage).
2. In the determination of CC2 content of an Impregnite suspension, a 1 ml tuberculin syringe should be used in place of the automatic pipette to withdraw the 0.5 ml test sample from bottle E.
3. Bottle F should be doubled in size and the seal in the cap should be made of material such as Teflon (DuPont trademark), which is resistant to the solvent (chlorobenzene-acetic acid solution).
4. Paragraph e, page 7, of the test procedure in TM 3-6665-202-12 should state that the end point is milky white and not milky pink.
5. In the determination of the CC2 content of clothing, double quantities of the clothing and reagents listed in TM3-6665-202-12 should be used.
6. In the determination of CC2 content of a suspension of Impregnite, the sample for testing should be withdrawn before the dye is added. This presents no problem since the dye is the last component added in preparing the suspension.
7. The minimum and maximum CC2 content of protective clothing should be established. A search of available literature revealed vague statements on this.

8. The differences in readings of percent of Impregnite by weight obtained from Figure 6, TM 3-6665-202-12 and those obtained from the graph in the lid of the kit should be resolved.

9. The correct graphs should be expanded so that readings would be more easily obtained. The graphic information might be more easily obtained if put in tabular form.

10. The solvent mixture should be labeled "volatile, flammable, toxic."

11. A statement in the TM 3-6665-202-12 should be included to explain the use and position of the plastic holder for the test solution bottle A, as presented in item 3, page 7 of this report.

12. Instructions should be included in the TM 3-6665-202-12 for the correct method of reading a burette as presented in the General Remarks, item 5, page 8 of this report.