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Technical Report

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DEVELOPMENT OF AN IMPROVED
CW IMPREGNATING PLANT



U. S. NAVAL CIVIL ENGINEERING LABORATORY
Port Hueneme, California



DEVELOPMENT OF AN IMPROVED
CW IMPREGNATING PLANT

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7 September 1959

by

J. E. Halton

U. S. NAVAL CIVIL ENGINEERING
LABORATORY
Port Hueneme, California

SUMMARY

OBJECT OF THE PROJECT

To develop chemical warfare decontamination equipment.

OBJECT OF SUBPROJECT

To improve chemical-handling procedures and equipment for use in converting laundry equipment to a clothing impregnation unit.

OBJECT OF REPORT

To present the development, fabrication, and testing of new chemical equipment to be used in conjunction with a portable laundry unit for impregnation purposes.

RESULTS

Operational difficulties encountered previously in the preparation of impregnation chemicals can be greatly reduced through the utilization of a turbine-type mixer and a baffled mixing tank. Mixing time and maintenance required are greatly reduced.

It is shown that a dual-speed washer-extractor is not necessary for the proper impregnation of clothing, and that the extraction procedures can be adjusted to utilize the normal washer-extractor speeds on the portable laundry. Wet clothing can be impregnated for further timesaving in the process.

ABSTRACT

The Laboratory was directed by the Bureau of Yards and Docks to improve present equipment and procedures for the impregnation of protective clothing for chemical warfare. Such clothing is treated in modified laundry equipment. The project was designed to develop, fabricate, and test improved auxiliary chemical-handling equipment to be used in conjunction with a portable laundry unit.

Experimental chemical-processing equipment was designed and constructed by the Laboratory to convert a 100-pound combination laundry to impregnation use. Through a series of tests, a new chemical-handling unit was developed and procedures were simplified. Tests disclosed that a turbine mixer utilized in a baffled mixing tank could be successfully used in the proper preparation of impregnation chemicals. A unit design incorporating this mixer is recommended for use with a combination washer-extractor for clothing impregnation.

CONTENTS

	page
INTRODUCTION	1
DESCRIPTION OF EQUIPMENT	2
Laundry Unit	2
Chemical Impregnation Equipment	3
IMPREGNATION CHEMICALS	3
Chemicals Used	3
Chemical Mixing	4
TEST PROCEDURE	4
First Test Series (with NPL)	4
Concentrate Preparation	5
Clothing Impregnation	5
Extraction of Impregnite	6
Drying of Clothing	6
Process Control Tests	6
Discussion of First Test Series Results	6
Second Test Series	7
Mixing Equipment Experiments	7
Impregnite and Hot Impregnite	10
Extraction and Rinsing Experiments	10

	page
Drying Experiments11
Dyes11
Wet or Dry Impregnation12
Discussion of Second Test Series Results12
NEW MIXING AND HANDLING KIT.12
Description of New Kit13
Checkout of New Unit.13
CONCLUSIONS.17
RECOMMENDATIONS18
REFERENCES19
DISTRIBUTION LIST41
LIBRARY CATALOG CARD43
	page
APPENDIX - OPERATING AND MIXING INSTRUCTIONS FOR IMPREGNITE MIXING UNIT.20
TABLI	
	page
1 - Composite Data Sheet Test Results16
ILLUSTRATIONS	
figure	page
1 - Laundry unit and experimental chemical-handling equipment	2
2 - Experimental mixer with variable speed drive	8

figure	page
3 - Baffled tank with experimental turbine blade mixer	9
4 - New chemical mixing kit with skid A on left and skid B on right:	14
5 - Recessed tank discharge	14
6 - Mixer boom and tank piping	15
7 - Hose connection to laundry washer	15

INTRODUCTION

Fulfillment of the BuDocks passive defense mission requires the preparation of permeable impregnated clothing for chemical warfare protection. Although certain items of laundry equipment primarily intended for other purposes are usable for clothing impregnation, special equipment is required for effective mixing of the impregnation chemicals. The Naval Civil Engineering Laboratory was therefore assigned by the Bureau of Yards and Docks the project of developing and testing auxiliary equipment that could be used in conjunction with a portable laundry unit to produce a complete clothing impregnation plant.

A new combination portable laundry unit was designed for the Bureau by the American Laundry Machinery Corporation, Cincinnati, Ohio, incorporating features which allowed ready conversion to impregnation use. This unit did not include the chemical preparation equipment necessary in impregnation operation, and the primary purpose of the subject project was the development of chemical-handling equipment to be used with the new portable laundry. This report presents the design, development, and testing of the auxiliary equipment.

As a starting point for the project tests, the Laboratory constructed a tentative experimental impregnation plant using the new laundry unit in conjunction with chemical-handling equipment designed by NCEL. A group of tests were run with the technical cooperation of NRL (Naval Research Laboratory) to determine the general performance of the plant and normal operating difficulties. A second group of tests was run to determine causes of mixing difficulties and the effect or variation in the drying and impregnation procedures on the uniformity of impregnation and color. As a result of these initial experiments, a new chemical-handling unit was designed, fabricated, and tested.

DESCRIPTION OF EQUIPMENT

The original experimental impregnation plant constructed by NCEL consisted of the American Laundry Machine Corporation portable laundry unit and auxiliary equipment designed by the Laboratory comprising three chemical mixing tanks, three mixers, and two circulating pumps (Figure 1).



Figure 1. Laundry unit and experimental chemical-handling equipment.

Laundry Unit

The portable laundry plant consists of three units mounted on steel skids; one 40-inch by 30-inch washer-extractor, a 37-inch drying tumbler, and a steam generator. The plant requires weather protection and a water supply ample for both the steam generator and washer. Approximately 17 kilowatts of electrical power at 208 volts,

60 cycle, and 3 phase is prescribed. The plant can handle loads of 100 pounds dry weight of towels, or 60 pounds dry weight of clothing (coveralls, jeans, etc.).

The washer-extractor is equipped with an additional drum switch and drive, an impregnite-suspension inlet valve, and an impregnite-suspension drain valve to allow use of the washer-extractor as an impregnator. The additional drum switch is marked for impregnation operation and switches the extractor speed to 220 rpm for extracting the impregnation slurry. For impregnation operation, the capacity is reduced to 65 to 75 pounds of clothing per load.

Chemical Impregnation Equipment

The original chemical-handling equipment for the preparation of the impregnite suspension, designed to mix the required chemicals in concentrated and diluted suspensions and to pump the dilute suspension in and out of the washer during the impregnation operation, consisted of a 150-gallon concentrate mixing tank, two 200-gallon dilute-suspension tanks, three medium-speed mixers (430 rpm), two Gould gear pumps, and appropriate valves and piping for the transfer of the concentrate and dilute suspensions from tank to tank.

IMPREGNATION CHEMICALS

Standard military impregnation chemicals were used throughout the various tests. These chemicals are used in both Army and Navy impregnation plants with slight variations in mixing and application procedures.

Chemicals Used

The impregnation suspension is designed to deposit the active ingredient, impregnite (CC-2), on the fibers of the clothing throughout the thickness of the fabric. Chlorinated paraffin (CP) is used as a binder for the CC-2, and zinc oxide is used to prevent the deterioration of the chlorine strength of the CC-2 and to protect the cloth. For impregnation prior to storage, additional zinc oxide to give an 80 to 20 ratio of CC-2 to ZnO is used. To insure penetration and deposition

of these materials on the fabric fibers, it is necessary that they be suspended as particles of approximately 4 microns in diameter. Larger particles are filtered out on the surface of the fabric and are unsightly and ineffective. The CC-2 and zinc oxide are normally purchased as a 90-to-10 percent mixture of "micronized" material called XXCC-3, but the compound is sometimes packaged as XXCC-4, a 95-to-5 mixture of CC-2 and zinc oxide. XXCC-4 was used in the NCEL tests. The XXCC-3 is composed principally of particles of the proper size, but suspension of the materials as individual particles requires vigorous mixing to break down agglomerates of the small particles that normally occur in the packaged material. The chlorinated paraffin is a viscous fluid and must be emulsified to secure the proper particle size.

PVA (polyvinyl alcohol, a dispersing agent), "Daxad" (a stabilizer), and "Duponal" (a wetting agent) are used in preparing the suspension to aid in emulsifying and stabilizing the chemicals. Dyes are used with the impregnation suspension to give the clothing the proper color.

Chemical Mixing

Proper preparation of the concentrate is the most critical phase of the impregnation operation and depends on suspending the CC-2, zinc oxide, and chlorinated paraffin as 4-micron particles. Various mixing procedures are used by different military impregnation plants. Some procedures dissolve the PVA and prepare the CP emulsion separately; others start with a PVA and XXCC-3 mixture and progressively add the other materials. The mixing equipment available determines to a large extent the procedure most suitable for a given situation. The methods used for the NCEL tests were based on an NRL operation instruction.

TEST PROCEDURE

First Test Series (with NRL)

Since previous Navy impregnation procedures were based on NRL instructions and the chemical equipment had been designed primarily on that basis, a cooperative test arrangement was made with NRL. The tests described under this heading were conducted during July 1956, at Port Hueneme, with the cooperation of Mr. G. H. Fielding of NRL

Concentrate Preparation. The initial step in the test series was the preparation of an impregnation-suspension concentrate. The equipment used for this initial make-up was a 150-gallon round-bottomed tank equipped with a medium-speed mixer, and two Gould gear pumps. PVA and XXCC-4 were mixed dry, then added to water in the mixing tank, and the mixture was recirculated through the pumps for 55 minutes. Daxad and Duponal were then added and the mixture was pumped for 25 minutes more. The next morning CP was added to the mixture by using the pump to draw the compound from the drum over a 40-minute period, and the mixture was circulated for an additional 45 minutes. The necessary water was then added. The XXCC-4 powder was added by sprinkling on the surface of the suspension, but as the addition was being completed, the circulating pump began to stall, despite frequent lubrications with CP. Continuing trouble then occurred with the pumps stalling and with the mixing drum used on the mixer becoming clogged with a pasty mass. The particle size of the XXCC-4 remained too large, being about 40 to 50 microns. A second batch was then prepared, although difficulty with the gear pumps prevented recirculation of the mix to achieve desired particle-size suspension. Nevertheless, a dilute suspension was prepared from this concentrate, dye was added to the dilute impregnate and a batch of clothing was processed. Several more batches of concentrate were made before the conclusion of testing; none completely satisfactory, although stirring of the concentrate without recirculation seemed to be nearly adequate with at least one of the batches. Unexplained variations in the CC-2 strength of the concentrate occurred, probably because of poor mixing. Foaming occurred at times, but was controllable with Dow-Corning Anti-Foam A.

Clothing Impregnation. The first batches of clothing were impregnated using a 9-percent CC-2 suspension, with resulting deposition of about 14-percent CC-2 in the clothing fabric. This is considerably higher than the required 10-percent and additional experimental runs were made with less concentrated suspensions. Results continued high, however, and the appearance of most of the clothing treated was poor because of mottling and streaking caused by nonuniform deposition of impregnate, possibly because of excess quantities of larger than 4-micron particles in the suspension. Sizing remaining in the material may also have been a cause of the streaking. Some improvement was noted when the more dilute suspensions were used. Experimental variation of the impregnation time seemed to indicate that the exposure time was a more controlling factor than the suspension strength.

Extraction of Impregnite. Extraction of the first batch of clothing was carried out at 220 rpm for 3-1/2 minutes and seemed to be inadequate. A second batch was extracted at 660 rpm for periods of 1 to 10 minutes. No definite improvement was noted. The extraction method given by NRL was then used; i.e., short periods of low-speed extraction combined with short periods of tumbling. A slight improvement was noted in the general appearance of the clothing.

Drying of Clothing. The NRL instruction manual¹ procedure was used without difficulty and with good results. This procedure requires preheating of the dryer to 100 F. The wet impregnated clothing is placed in the dryer and the temperature is slowly raised to 180 F. Fifty minutes is used to raise the temperature to 180 F and the temperature must not exceed this level. After 5 minutes at 180 F, the temperature is reduced over a 15-minute period to about 140 F and the clothing can be removed when it is cool enough to handle. Only about 30 pounds (dry weight) of clothes should be dried in one load.

Process Control Tests. Microscopic tests were made frequently during the preparation of the concentrate to determine whether proper mixing was being attained, as attested to by the micron size of the particles during various stages of the operation. The particle size of the initial mixture of PVA and CP should show 95 to 100 percent of particles 4 microns or below before adding the zinc oxide and the balance of the XXCC-4. The final mixture should show 95 percent of the particles below 8 microns.

Cloth swatches and tubes were attached to various items of clothing to check on correlation of CC-2 deposits of such samples with the CC-2 deposits on the clothing. Correlation was poor and test swatches and tubes showed lower strength deposits.

Discussion of First Test Series Results. Results throughout this series of tests showed the difficulties of obtaining uniform impregnation. This was not unexpected since much of the clothing had been received in an already impregnated condition from a standard impregnation plant, and showed nonuniform depositions of impregnite and dye. The American Laundry Machinery Corporation portable laundry proved quite adequate for its part of the function, but many difficulties were observed in the chemical-handling process. The chemicals obtained from Navy stores seemed somewhat old, but test batches of concentrate produced good quality impregnation suspensions. Mixing and pumping the suspension was most troublesome and further study of this aspect was indicated.

Second Test Series

Because of the poor results of the first tests, it was decided to obtain as much information as possible from operating impregnation plants, and to conduct the following tests and experiments at Port Hueneme in the hope of establishing more reliable information.

Mixing Equipment Experiments. Investigations of the fundamentals of mixing for reduction of agglomerated small particles to the ultimate particle size of the compound indicated that a high shear type of mixer was most desirable for this type of operation. Since this is the desired function of the concentrate mixer, while handling the XXCC-3 or -4 suspension, a turbine-type mixer was selected as yielding the best high-shear mixing conditions. This type is also suitable for dispersing the CP. A small mixer of this type was fabricated and tested with good results. Consequently, a larger mixer (laboratory-type) with variable-speed drive was built and tested. A satisfactory concentrate suspension was prepared in less than one hour, with particle size less than 4 microns, and no difficulty was experienced in pumping the concentrate. The laboratory mixer proved a valuable tool in determining the rpm and horsepower requirements for the desired turbine-type mixer.

The milling action of the gear pumps is not necessary for preparing the concentrate, and more suitable pumps for pumping the concentrate and dilute suspensions were investigated. Finally, a Worthington internal-bearing positive-displacement pump was tried and found to be very adequate. The bearings in this pump are carbon and will withstand very high temperatures, whereas the bearings in the Gould pumps used in the first tests were brass and had a tendency to expand under temperature conditions other than ambient, with resultant pump seizures during the course of mixing operations.

The proper shape and baffling of the concentrate mixing tank were also logical areas of investigation for improving the mixing process, and a tank with four vertical side baffles was constructed and used with a "Lightnin" mixer. Results were good, but dead areas existed at the base of the baffles. This was remedied in a second tank by leaving vertical slots at the base of the baffles. Subsequent mixing was improved and this type of tank was used with the turbine mixer described previously. The tank and mixer are shown in Figures 2 and 3.

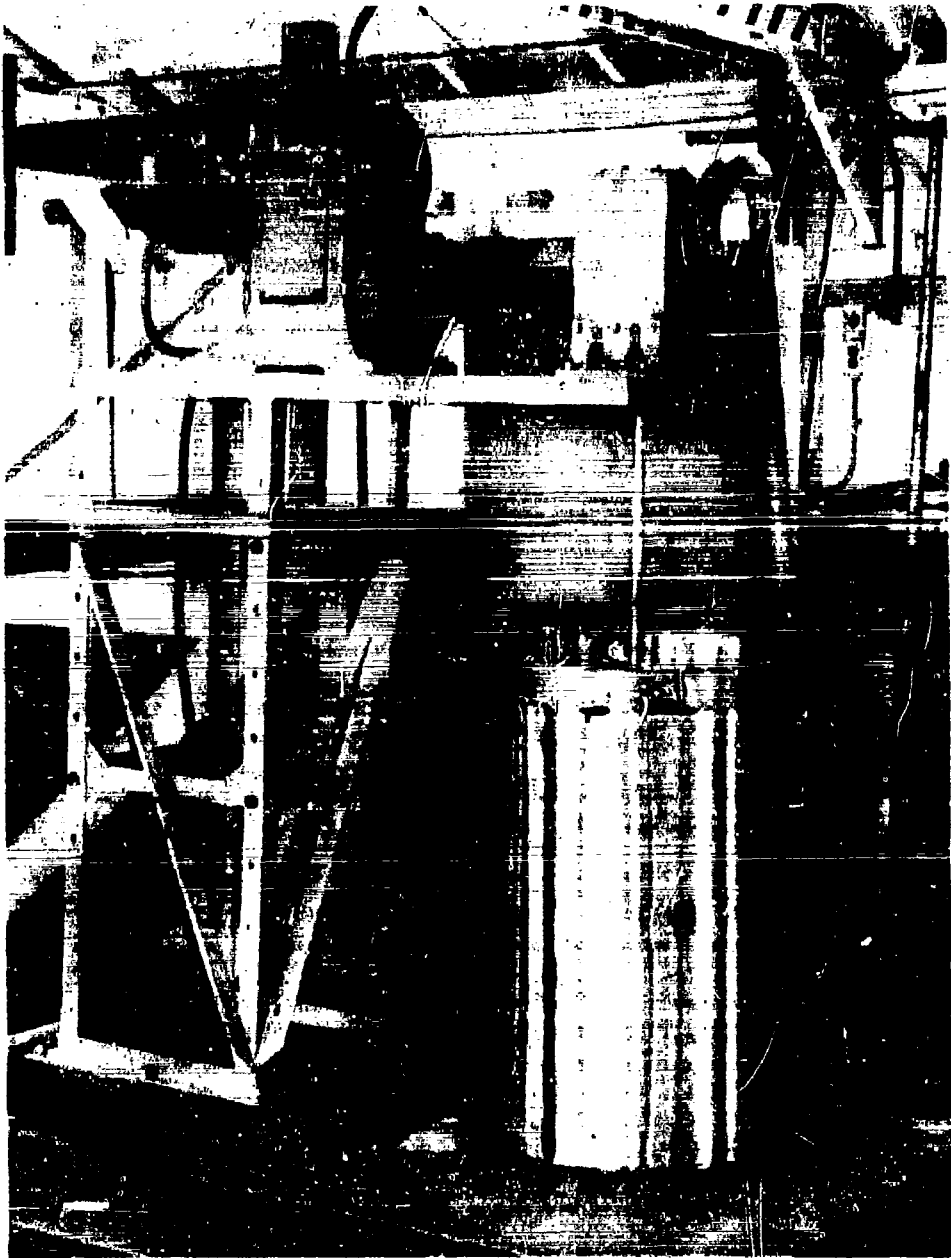


Figure 2. Experimental mixer with variable speed drive.



Figure 3. Turbine blade mixer in baffled tank.

Impregnite and Hot Impregnite. A fresh batch of concentrate, conforming to the NRL formula, was made utilizing the turbine-type mixer with the baffled tank. The CC-2 strength of the concentrate was 28 percent, and 95 percent of the particles were under 4 microns. A dilute slurry suspension, utilizing the freshly prepared concentrate, was then made up, averaging 6.3 percent CC-2. The impregnator was then loaded with 50 pounds of clothes, with test swatches attached, and the dilute suspension was pumped in. The clothes were then impregnated, extracted, and dried. The results were excellent in every way. The test swatches showed a definite correlation with respect to the amount of CC-2 contained in the impregnated clothing as determined by the chemical analyses of the swatches and test patches cut from the impregnated clothing. The test swatches averaged 10.50 percent CC-2 by weight as against 10.90 percent CC-2 for the test patches taken from the clothing. During the course of the earlier tests (first series) it had been impossible to get such close correlation.

A trial run using impregnite heated to 133 F resulted in heavier impregnation, indicating the possibility of speeding up the process by using hot impregnite. However, since the time required for impregnation is already short compared to other steps in the process, the additional expense of heating the impregnite is not likely to be worthwhile.

Extraction and Rinsing Experiments. The results of the initial tests had indicated a slight advantage in using the 220-rpm extraction interrupted by short periods of tumbling, and further tests were run using this method and others to compare results. Other methods tried were 660-rpm extraction and the Brooklyn impregnation plant method. The Brooklyn method also uses 220-rpm extraction speeds interrupted with periods of tumbling, but the time intervals are somewhat longer. Results indicated that there was little to choose between the three methods tried, and that all were fairly satisfactory even though the color and chemical dispersal were slightly uneven throughout the clothing impregnated. In an effort to improve upon the general appearance of the impregnated clothing, a series of post-rinsing operations were conducted after extracting at 660 rpm. The extraction cycle utilized was as follows:

Extract - 20 seconds at 660 rpm
Tumble - 3 minutes
Extract - 20 seconds at 660 rpm
Tumble - 3 minutes

11

This was followed by a rinsing operation for 3 minutes in 120 F water. A final extraction of 20 seconds duration followed by a two-minute tumbling period concluded the operations. The rinse water used was returned to the dilute slurry tanks to be strengthened with concentrate for future impregnation operations.

The results obtained from the 660-rpm high-extraction tumbling and rinsing operation were superior to the earlier extraction methods tried. Uniformity of color and chemical dispersal was attained. Utilization of the above procedure eliminates the requirement for a dual-speed extractor-type laundry unit. Since the operating characteristics of laundry extractors vary, it is probable that experimentation of extraction and tumbling times will be required to yield the best results for individual plants.

Drying Experiments. One test was made in which results were compared on impregnated suits which were: (1) drip-dried, (2) extracted and air-dried; and (3) extracted and dried in the rotary drier to check effects and suitability of these methods. The rotary drier yielded superior drying results. The other methods are suitable for emergency conditions. Although the rotary drier yielded superior drying results, it was noted that the clothing was all knotted up in a ball and not evenly dried throughout. This could easily be rectified through the utilization of reversible tumbling action type of dryers, prevalent in commercial practices, or possibly by slowing down the speed of the existing rotary-type dryer and also by the addition of extra cross members (bars) to the basket.

Dyes. It was difficult to obtain uniform results with the dyes used with the impregnation process. The use of three separate dye compounds seems an unjustified complication to the process. Inasmuch as the Army successfully uses a single compound, with its large M2 plant, the use of a single compound seems a worthwhile reduction in the process. As there are considerable existing stocks of the three dyes to be used up, it would seem desirable to blend a single compound from the existing materials and make subsequent purchases of a single dye of the proper color.

Wet or Dry Impregnation. A run was made using washed and extracted clothing that was still wet. Impregnation was satisfactory. Success is also reported³ with this method by the Army Chemical Corps.

Discussion of Second Test Series Results. The portable laundry machinery proved adequate for impregnation operation in both the first and second test series, with the exception of a tube failure in the steam generator. The impregnation procedures recommended in the NRL manual were greatly improved by the modified experimental chemical-handling equipment; though the concentrate and dilute-suspension storage tanks designed for these tests were too high for convenient operation. The turbine mixer and the baffled tank eliminated troubles from the mixing and pumping standpoints, since they reduced the required time of use of the pumps and insured that the pumps would handle the concentrate only after all materials had been reduced to the proper size. A boom-type davit seemed desirable for positioning the turbine blade mixer. This innovation would result in savings, inasmuch as a single turbine blade mixer could be utilized in all four tanks of the impregnation mixing kit.

As a result of the foregoing developmental and testing work accomplished at the Laboratory, a new experimental mixing kit has been designed, fabricated, and tested.

NEW MIXING AND HANDLING KIT

The new experimental chemical-handling kit was designed and fabricated as a complement for advanced base laundry units for impregnation operations. Bases for the new design were the experimental results of mixer design, tank design, and tests of mixing and handling techniques. Results of tests indicated that turbine-type mixers utilized in baffled tanks could be successfully used in the proper preparation of the concentrated impregnite. This was accomplished with a minimum amount of time and maintenance requirements as compared to the methods utilized in the NRL manual. Proper particle size was easily attainable and pump problems were nonexistent.

Description of New Kit

The new experimental kit is permanently mounted on two 54-inch by 96-inch by 8-inch skids, connected by a single flexible coupling. This kit can easily be attached to any laundry unit for impregnation operations through the utilization of two flexible couplings for the transfer of the impregnite slurry to and from the laundry machine. The component parts of this kit (see Figure 4) are: (a) four galvanized steel tanks; (b) two Worthington rotary pumps with 1.5-hp electric motors, one for transfer of concentrate and one for the dilute slurry; (c) one "Lightnin" mixer with turbine-type blades, driven by a 1.5-hp electric motor at 431 rpm; (d) piping and valves; and (e) an electrical control panel.

The concentrate tanks on skid A have recessed discharges (Figure 5) to allow a side discharge for accessibility of piping. The mixing tank has a volume of 9.5 cubic feet, which allows mixing of a 40-gallon batch of concentrate. The concentrate storage tank has a capacity of 110 gallons. The storage tanks for diluted suspensions have a 148-gallon capacity.

The size and shape of the tanks makes them easily accessible, and the piping is compact and kept to a low level throughout. Figure 6 shows the mixer boom and the piping. Figure 7 shows the hose connection to the laundry washer. The mixer boom swings to allow the same mixer motor to be used for any of the four tanks.

Checkout of New Unit

The new unit was used to prepare several batches of impregnite suspension with which clothing was impregnated. Results were satisfactory and a mixing time of less than one hour was required to prepare the concentrate suspension.

Clothing impregnated showed a higher concentration of impregnite than expected. This was probably a result of the new mixing technique giving a better percentage of small particles in the suspension. Consequently, some adjustment in the recommended dilute impregnite suspension strength is required. Recommended changes in NRL procedure are included in the Appendix. Table 1 is a composite data sheet showing test results for the old and the new unit.



Figure 4. New chemical mixing kit with skid A on left and skid B on right.

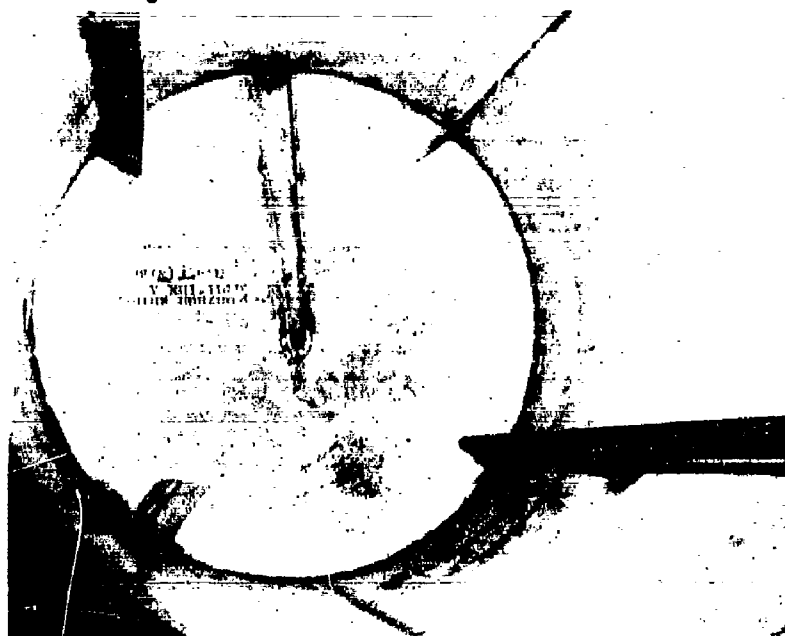


Figure 5. Recossed tank discharge.



Figure 7. Hose connection to laundry washer.

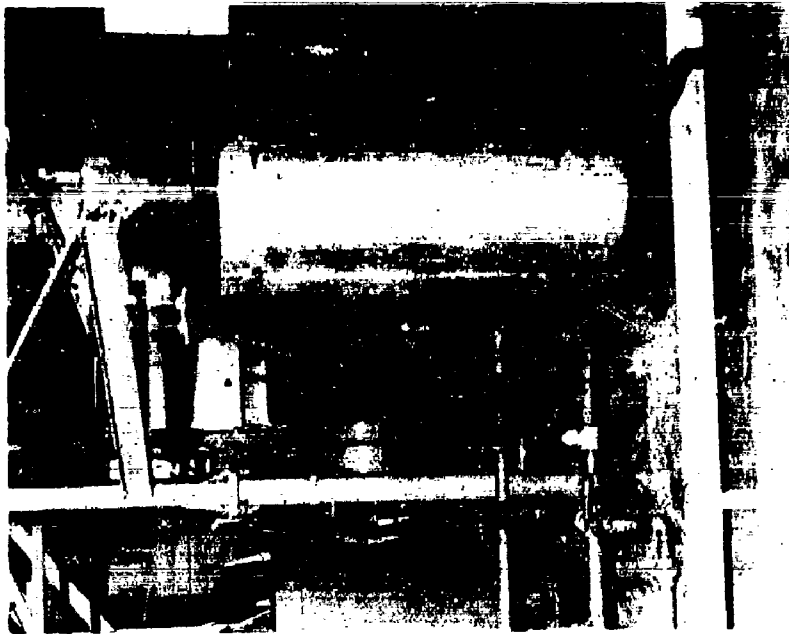


Figure 6. Mixer boom and tank piping.

Table I. Composite Data Sheet Test Results.

	Old Unit	New Unit
Average mixing time of concentrate	285 minutes	50 minutes
Suspended particle size (conc.)	95 percent in excess of 40 - 50 microns	95 percent less than 3 microns
Dilute suspension concentrate percent	9 percent	6.3 percent
Clothing - concentration percent CC-2	14 - 16 percent	10 percent
Swatches - concentration percent CC-2	10 - 12 percent	10 percent
Clothing appearance	mottled, nonuniform deposition of CC-2	excellent
Extraction cycle rpm	220 rpm	660 rpm and post rinse

The rapid mixing ability of the new unit allows 280 gallons of concentrate to be prepared per 8-hour day. This is sufficient to prepare 1550 gallons of dilute suspension necessary for impregnating 1550 pounds of clothes, based on the ratio of one gallon of dilute suspension per pound of clothes. The concentration of this dilute suspension would approximate 6.3 percent CC-2, which is strong enough to obtain a 10 percent CC-2 content in the clothes impregnated. If the dilute suspension is to be reclaimed after each impregnation operation, the original 280 gallons of concentrate would be sufficient to impregnate approximately 2300 pounds of clothes (based on impregnating new unimpregnated clothing). The figure of 2300 pounds would be considerably higher if this concentrate were to be used for reimpregnation purposes (because of reduced requirements for used impregnated clothing).

CONCLUSIONS

As a result of the tests and investigations, it is concluded that:

1. The 100-pound laundry can be converted for use in clothing impregnation by combining it with the new chemical-handling system described in this report.
2. The new equipment reduces the Impregnite mixing time; hence the NRL procedure for impregnation, extraction, and drying needs some modification.
3. With the turbine blade mixer and baffled tank, a batch of concentrate can be prepared in approximately one hour, a considerable timesaving over using gear pumps for mixing.
4. The dual-speed extractor-type laundry unit is not a necessity. Excellent results were attained with the spin-tumble extraction method even when a maximum speed of 660 rpm was used.
5. Results obtained using a 660-rpm extraction cycle followed by post-rinsing operations were far superior to any other methods tried with respect to uniformity of dispersal of impregnite and dye.
6. Post-dyeing (dyeing after completion of impregnation) improved the appearance of the clothing, and heating of the impregnite suspension hastened the process, but neither procedure is considered worthwhile because of additional time and equipment demands for a minor gain.
7. Wet clothing can be impregnated, resulting in considerable timesaving when the clothing requires laundering before impregnation.
8. Use of a single dye compound is desirable.
9. Best drying results are obtained with the rotary dryer, but drip-drying, i.e., extracted and air-dried, would be suitable in an emergency.

10. Test swatches pinned to the clothing can be used for determining the impregnate in the treated clothes, although occasional checks must be made to insure correlation.

RECOMMENDATIONS

It is recommended that:

1. New chemical mixing and handling kits for converting laundry units into impregnating plants should be based on the turbine mixer and baffled tanks design.
2. Pumps in the new unit should be of the internal carbon-bearing positive-displacement rotary type.
3. Steps be taken to convert existing stocks of the three separate dyes to a single dye compound, and to make subsequent purchase of a single dye compound.
4. The dual-speed washer-extractors be replaced by a single-speed washer-extractor after existing stocks are depleted, inasmuch as an excellent job of extraction can be done with a high-speed extraction cycle.
5. The NRL operating manual¹ be revised to incorporate the information in the Appendix together with details of operations with the new mechanical equipment.

REFERENCES

1. Naval Research Laboratory. "Instructions for the Operation of the Navy Portable Clothing Impregnating Plant, "A635-B, revised by J. M. Davidson, Bureau of Ships.
2. Department of the Army, Technical Manual TM 3-281. "Impregnating Plant, Clothing, M2," February 1954.
3. Chemical Corps, Chemical Warfare Laboratories. "Determination of Method for Impregnation of Wet Clothing," by John L. Meredith, 2nd Lt., CRLR 532, Army Chemical Center, Maryland, 6 March 1956.

AD-89 418

APPENDIX

OPERATING AND MIXING INSTRUCTIONS FOR IMPREGNITE MIXING UNIT

The information in this appendix is based on the "Instructions for the Operation of the Navy Portable Clothing Impregnating Plant", Naval Research Laboratory, No. A635B, Revised 5-51, Washington, D. C. Changes have been made in the section "Concentrate Preparation" to conform to the new mixing unit designed by NCEL. The sections "Preparation of Dilute Impregnating Suspension," "Operation of Plant," "Laboratory Tests and Analysis Procedures," and "Inspection, Handling and Marking of Clothes" have been taken directly from that publication.

CONCENTRATE PREPARATION

The actual impregnation of clothing is carried out in the laundry-impregnator. Impregnating suspension of the proper strength is pumped into the impregnator which is loaded with the clothing. When the clothing has become thoroughly soaked, the solution is pumped out and the excess liquid removed from the clothes by centrifuging. In this step the drum of the impregnator revolves at a high speed. After this extraction process, the clothing is transferred from the impregnator to the drier. In the tumbler-drier air is sucked in over steam coils and passes through the clothing, driving out the water. Careful control of the drying temperature is necessary. This step completes the impregnation process.

The binding agent to hold the impregnite on the cloth is chlorinated paraffin (CP). This agent is a liquid which is not soluble in water. To apply this agent to clothing it must be broken down into very small globules or emulsified in the water. An emulsifying agent is required to stabilize the small globules against coalescing and separating into a separate layer from the water. One of the most effective agents for this process is polyvinyl alcohol (PVA). It has been found that the addition of a small amount of a wetting agent, Duponol, to the polyvinyl alcohol will greatly reduce the globule size of the chlorinated paraffin and, therefore, render the emulsion more nearly like a solution. DaXad, naphthaleneformaldehyde sodium sulfonate, is also added to stabilize the emulsion and aid in dispersing XXCC-3.

Zinc oxide is added as a component part of the impregnating suspension because of its property of preventing deterioration of cotton clothing. It is believed that its action is merely to react with hydrochloric acid released by hydrolysis of the impregnite. The amount recommended is twenty percent by weight of the XXCC-3.

Navy protective clothing is made of unbleached, undyed cotton. When dyed clothing is required for camouflage, it is necessary to add a dye to the impregnating suspension. The Navy has adopted a single shade of olive green for all protective clothing which requires using three dye powders.

Equipment

Four tanks of graduated sizes are used in mixing the concentrated and dilute impregnation suspensions. The smallest tank (tank No. 1) is used to prepare a concentrated suspension of impregnite. The next larger tank (tank No. 2) is used to store the prepared concentrated suspension. The two large tanks (Nos. 3 and 4) are used for mixing dilute suspensions used in the actual impregnation operation.

The concentrated suspension is prepared as follows:

A. Mix thoroughly, in a bucket or box, 3-1/2 pounds of dry PVA and 4 pounds of XXCC-3. Mix very well and add mixed powders to 12 gallons of water in tank No. 1. Start mixer and concentrate pump to recirculate contents in tank No. 1.

B. Dissolve 1 pound (454 grams) of DaXad and 0.2 pound (92 grams) of Duponol in 1 quart of water while stirring. Add to tank No. 1 when dissolving is complete. Stop gear pump.

C. Keep mixer running in tank No. 1 and add 52-1/2 pounds of chlorinated paraffin. This addition should be done slowly over a 5 to 10 minute period. After addition is completed, check micron size of the emulsion to see that globules are about 4 microns in diameter. This must be checked with a microscope.

D. Add 14 gallons of water and continue mixing.

E. Start the concentrate pump recirculating the tank contents and slowly add 162 pounds of XXCC-3 and 20 pounds of zinc oxide with the mixer operating. These ingredients should be sprinkled on the surface of the stirring liquid in small portions. About twenty minutes should be used for this addition. Continue operation of the mixer and concentrate pump until a microscopic check of the suspension shows that 95 percent of the particles are smaller than 8 microns in diameter.

F. Pump the concentrate over to tank No. 2 and flush tank No. 1 and the concentrate pump with fresh water. Another batch may then be prepared in tank No. 1 if desired.

PREPARATION OF DILUTE IMPREGNATING SUSPENSIONS

(1) For Impregnating Airzen Suits

Impregnation of the white two-piece protective suits requires that they be dyed for camouflage purposes. Three water dispersible dyes are furnished to produce the shade of green used for all suits. These water dispersible dyes for aqueous impregnation are given below:

TLX - 42 Lithosol Yellow ARP
TLX - 20 Monastral Green G
TLX - 29 Carbon Black

Equal proportions of these dyes make the color desired.

Regardless of proportions of individual dyes, the same total weight of dyes is used. The amount is 9 percent by weight of CC-2. For example, 135 gallons of 9-percent suspension is to be made up to dye clothing the green shade. The suspension will contain 135×8.8 (from density table paragraph 4) $\times .09$ or 107 pounds of CC-2, which requires $107 \times .09$ or 9.6 pounds total weight of dye.

The dye powders should be stirred into 4-5 gallons of water in a bucket. When powders have become dispersed and no lumps remain, the paste is added to 80 gallons of water in one of the dilute suspension tanks. The bucket should be carefully rinsed out and the total water in the tank made up to 100 gallons. The dye dispersion is recycled in a few minutes and then 35 gallons of concentrate is pumped in.

Recycling and agitation are continued for 20 minutes to insure complete mixing. An analysis should now be made of the dilute suspension. It should contain 9 percent CC-2. A value of 8.5 percent or below requires that more concentrate be added whereas a value of 9.5 percent or above requires that more water be added.

Incorporation of dyestuff in the concentrate is possible if the dyes are first made into a paste and if the concentrate is recycled for at least an hour. Dyes may be added to dilute suspension already prepared. However, for maximum color value, it is believed best to disperse the dyes in as large a volume of water as possible. Note: A 9-percent suspension may not be the best for a particular portable plant. Many factors affect the CC-2 pickup of suits. If it is found that a 9-percent suspension gives high loadings, reduce the suspension strength until suits of 10-percent CC-2 content are obtained.

(2) For Reimpregnation of Arzen Suits

Clothing that is to be reimpregnated must first be tested for strength, analyzed for CC-2 content, and laundered if sufficiently dirty. It is important to know the CC-2 content of clothing that is to be reimpregnated.

The reimpregnation level for clothing is 3-percent CC-2. This value is based on protection against gas and applies to clothing being worn in the field.

Below 3 percent CC-2 content, clothing becomes much less effective against vesicants. For this reason, a value of 6 percent CC-2 has been established as the lower limit for clothing which is to be reissued from any impregnating plant.

It is not desirable to heavily reimpregnate clothing since it becomes heavier, greasy, and less comfortable to wear. The following table of suspension strengths is recommended for Arzen suits containing different CC-2 contents:

<u>CC-2 Content of Suits</u> percent	<u>CC-2 Content of Suspension to be Used</u> percent
5	4
4	5
3	6
below 3	8

Socks and gloves less frequently require reimpregnation unless they have been worn. Suspension strengths should be adjusted according to the CC-2 content of the socks or gloves as follows:

CC-2 Content of Socks or Gloves	CC-2 Content of Suspension to be used
percent	percent
5	2
3	2.5
below 3	3

When only a few impregnator loads of clothing are to be reimpregnated, make up only a small batch (50 gallons) of dilute suspension. For one or two loads, a calculated amount of water can be added directly to the impregnator and the regular 9 percent suspension pumped to give the desired concentration. Reanalyze the suspension in the tank after the run and adjust the concentration to 9 percent.

(3) Impregnation of Socks and Gloves

Besides the two-piece protective suit, two pair of part-wool socks and one pair of woolen gloves constitute a unit of protective clothing. Impregnation of these socks and gloves by the aqueous suspension process causes considerable shrinkage. No procedure has been found to overcome this difficulty.

The socks and gloves should be impregnated to contain 10-percent CC-2. However, because of their loose texture they can be impregnated in a more dilute suspension. The strength of the suspension required will be between 4 percent and 6 percent depending on the particular plant. 100 gallons of 6-percent suspension requires 85 gallons of water and 15 gallons of concentrate suspension. The dilute suspension should be well mixed and analyzed before use.

(4) Impregnation of Other Types of Clothing

It is likely that, in cases of emergency, portable plants will be called upon to impregnate other types of clothing. For example, the Navy may decide to issue protective short drawers or all-cotton socks and gloves. Impregnation or reimpregnation of Army protective clothing might be required.

In most cases, it will be necessary for each plant to work out exact procedures for any type of clothing encountered. However, certain general rules apply. A seven to nine ounce cotton twill will be done the same as Arzen suits. Knit cotton shorts or underwear will be similar to socks, and require a more dilute suspension. Coarse, heavy articles like canvas leggings will require higher suspension strength. The following table of suspension strengths for different types of clothing is given as a guide:

Knit underwear, long and short	4 percent
Army wool hoods	3 percent
Army or Marine herringbone twill garments	8 percent
Canvas leggings	15 percent

Whenever pre-dyed clothing is to be impregnated, reduce the amount of dye from 9 percent to 3 percent by weight of CC-2. This amount of dye is required to prevent whitening of the clothing.

G. Methods of Calculation

It is frequently necessary to be able to calculate how much concentrate or water must be added to a given tank of impregnating suspension to bring it to the correct strength. For this purpose it is necessary to know the densities of the suspensions involved. The following table is given of approximate densities:

<u>Material</u>	<u>lbs/gal</u>
Water	8.3
10 percent PVA solution	8.5
Concentrate	10.0
9 percent suspension	8.8
6 percent suspension	8.5

Perhaps the simplest method of calculation is to base everything on the weight of CC-2 in pounds. In any given suspension the amount of CC-2 can be calculated if the total quantity of the suspension in gallons, the density of the suspension, and the percent CC-2 by analysis are known. It is not believed necessary to analyze concentrated suspensions. In calculations use the value of 28-percent CC-2 for the new standard low chlorinated paraffin concentrate. Therefore, each gallon of standard concentrate will contain 2.8 pounds of CC-2.

The fundamental equation to be remembered is that total volume times density times percent CC-2 equals pounds of CC-2:

$$\text{Volume} \times \text{density} \times \text{percent CC-2} = \text{pounds CC-2}$$

The following examples illustrate the method of calculation:

Example 1. A tank contains 135 gallons of impregnating suspension analyzing 7.8 percent CC-2, it is desired to make this up to 9 percent CC-2.

The pounds of CC-2 desired are

$$135 \times 8.8 \text{ (from table above)} \times .09 \text{ or } 107 \text{ pounds.}$$

The pounds of CC-2 present in the tank is

$$135 \times 8.8 \times .078 \text{ or } 92.5 \text{ pounds.}$$

The difference is 14.5 pounds of CC-2 that must be added. Since each gallon of concentrate contains 2.8 pounds of CC-2

$$14.5/2.8 = 5.2 \text{ gal of concentrate required.}$$

This method disregards the increased volume of the suspension due to the concentrate added but is sufficiently accurate for the purpose.

Example 2. In one of the suspension tanks there remains 39 gallons of suspension which analyzes 9.3 percent. It is desired to use this in making up 135 gallons of 6-percent suspension for impregnation of socks.

The pounds of CC-2 desired are

$$135 \times 8.5 \times .06 \text{ or } 69 \text{ pounds.}$$

The amount of CC-2 in the tank is

$$39.8.8 \times .09 \text{ or } 32 \text{ pounds.}$$

The difference (69-32) is 37 pounds of CC-2 which must be added as concentrate. Each gallon of concentrate contains 2.8 pounds of CC-2; hence $37/2.8$ or 13.2 gallons of concentrate must be added. The tank should then be filled up to 135 gallons with water. The actual amount of water to be added will be $135 - (39 + 13.2)$ or 82.8 gallons.

OPERATION OF THE PLANT

A. Procedure for Impregnating Clothing

After a dilute impregnating suspension has been prepared and analyzed, impregnation of suits, socks, or gloves may be started. One load for the impregnator constitutes 33 pounds of dry clothing. This is as follows:

11 two-piece suits	or
250 pairs of socks	or
100 pairs of gloves	

It is essential that the two-piece suits be opened up and shaken before being placed into the impregnator. They must not be thrown into the impregnator as they come, folded up. In the case of socks and gloves, all tags and staples must be removed before they are placed into the impregnator. Note: In some plants, the 33 pound load may have to be reduced if it is difficult to get the impregnator up to high speed for extraction or if belts on the dryers slip badly.

After the impregnator has been loaded, 25 gallons of the impregnating suspension should be pumped in. During this time and for five (5) minutes after the suspension has been pumped in, the impregnator should be kept tumbling. This is sufficient time to soak the clothes thoroughly. While continuing to tumble the clothes, pump the suspension out of the impregnator back into the suspension tank. When all the solution has been pumped out as indicated by the sight glass, stop the impregnator and allow it to come to rest. Shift the gear to the extraction position. If the gear does not want to go into the extraction position, keep pressing on it and give the "jog" button a quick punch. This will allow the gear to go all the way into the extraction position. Start the impregnator and continue the high-speed extraction for 20 seconds after top speed has been reached. After the machine has come to rest, as indicated by the

red light on the panel board, shift the gear back to the tumble position and tumble the clothes for two minutes. The suspension that is thrown out of the clothes by the extraction must of course be allowed to pump out. Keep pumping out of the impregnator during the entire extraction process.

After the two minutes of tumbling, the 20-second extraction is repeated. Follow this by another two-minute tumbling period. A final extraction of one minute is then made followed by 30 seconds of tumbling to break the clothes away from the sides of the impregnator. The clothing is now ready to be removed from the impregnator and transferred to one of the dryers. The entire impregnation cycle should be carried out in twenty minutes. The following time intervals are given as a summary.

Loading clothes into the impregnator	1 minute
Pumping suspension in	2-1/2 minutes
Sauking	5 minutes
Pumping suspension out	2-1/2 minutes
20-second extraction	1/2 minute
Tumbling	2 minutes
20-second extraction	1/2 minute
Tumbling	2 minutes
Final extraction	1 minute
Tumbling	1/2 minute
Unloading	1 minute
Unused time	1-1/2 minutes
Total time	20 minutes

Many different impregnation cycles than the one above have been used. However, whenever an appreciable change is made in the cycle be sure to test the impregnated clothing for uniformity of impregnation. When socks and gloves are impregnated, shut off the machine while pumping solution in and out of the impregnator. Also reduce the tumbling periods between extractions to 1/2 minute. This will help to minimize shrinkage. The operation schedule given above is to be used with a 9-percent suspension when the high speed rpm of the impregnator is 300. The rpm of the impregnator should be checked regularly since a change in voltage will cause a change in impregnator speed. If the rpm of the impregnator cannot be maintained, an adjustment in the operation schedule or the suspension strength should be made to compensate.

To use as samples for analysis, it is customary to attach small swatches of cloth to suits in each batch. Pieces of Arzen cloth are furnished to be used as samples for the suits. Attach three 6 x 6 cloth samples to suits in each load. When socks and gloves are being impregnated it is necessary to cut up unimpregnated ones to use for analysis samples. The analysis samples can be stapled or tied on the clothing in each batch.

Plant tests have shown that a better agreement between CC-2 content of analysis samples and suits is obtained when the samples are two layers of cloth instead of one. It is therefore recommended that analysis samples consist of 12-in. by 3-in. tubes or sleeves. Only one end of the sleeve is attached to the garments.

B. Procedure for Laundering Clothes

When clothing is received for reimpregnation it will frequently be necessary to launder the clothing before carrying out the reimpregnation. The washing formula given below is a very mild one. It will not thoroughly clean very dirty clothes yet it permits removal of surface dirt without appreciable destruction of residual impregnite.

The detergent "Nacconol NR" is a synthetic wetting agent which works well in soft, hard, or even sea water. It is not alkaline and therefore causes less destruction of impregnite than soap. A further advantage of the synthetic detergent is that it can be used at lower concentrations than soap.

One load of dry clothing for laundering is 33 pounds. About 18 gallons of water at 90 F is run into the tank on top of the impregnator by means of the automatic valves. When the tank is filled with this amount of water, open the valve to the impregnator and let the water run in on the clothes. At the same time add 1-1/2 ounces of "Nacconol NR" through the slot on the upper left hand side of the impregnator. Start the clothes tumbling and continue for five minutes. Meanwhile, the tank should again be filled with 18 gallons of water at 90 F for the next wash. When the clothes have been washing for five minutes, open the drain and let the dirty water run out. When the water is drained out (about 1-1/2 minutes), close the drain valve and allow the 18 gallons of water in the tank to run into the impregnator. Add the same quantity of detergent as before and continue washing for another five minutes.

The machine should not be stopped during the draining and refilling. Once again the dirty water is drained and a third 18 gallons of water is added along with the detergent and washing continued for five minutes.

After the three five-minute "sudsings" the clothes should be rinsed three times with 18 gallons of water at 90 F. The clothes should be allowed to tumble for two minutes during each of the rinses. When the water has been drained out from the third rinse, stop the machine and shift the gear into the extraction position. Extract the clothes for three minutes at the high speed. After the extraction, let the clothes tumble for about 1/2 minute to break them away from the sides of the drum, take them out and put the load into one of the dryers. A summary of these operations is given below:

Loading	1 minute
1st wash	5 minutes
2nd wash	5 minutes
3rd wash	5 minutes
1st rinse	2 minutes
2nd rinse	2 minutes
3rd rinse	2 minutes
Extraction	3 minutes
Tumbling	1/2 minute
Unloading	1 minute

Total	26 minutes
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After some experience, an operator should be able to carry out two washing cycles in an hour. It may be possible with clothes that are not very dirty to use only two five-minute sudsings instead of three. In the event that very dirty clothes are received or unimpregnated clothing is to be laundered, it is recommended that the water temperature be raised to 140 F. For this purpose soap may be substituted for the synthetic detergent and four ounces of soap should be used for each 18 gallons of water.

C. Drying Proced

Drying of clothing that has been impregnated must be done carefully, since excessive heat will cause loss of impregnate and may also destroy the clothing. Drying curves obtained for the tumbler

dryers show that during the major part of the drying cycle the temperature remains between 70 and 80 C (160 - 175 F). Following this the temperature will gradually rise to 105 C (220 F).

When the drying is started be sure that the boiler is delivering the correct steam pressure (100 pounds), that steam is going into the coils of the dryer, and that both flaps are open over the steam coils. Continue the drying until the indicator registers 180 F. This will usually require about 50 minutes for a load of two-piece suits. When the temperature has reached 180 F, close the right-hand flap over the steam coils and continue drying for five more minutes. After this, close the other flap and allow the clothing to cool down to 140 F. The clothing should be taken out and taken to the folding table. Never leave a load of clothing in one of the dryers after it has finished drying. Also do not allow a load of hot clothing removed from one of the dryers to stay tightly packed in a cart or tub.

Drying of clothing that has been laundered should be carried out in the same manner since residual impregnite can break down suddenly and destroy the clothing if the temperature is allowed to go too high. Examine very carefully every load of clothing that has been laundered prior to reimpregnation to see if the drying has caused a deterioration of the fabric.

Socks and gloves are to be dried in the same manner as two-piece suits. It has been found that shrinkage of socks and gloves during impregnation can be minimized by drying at temperatures below 167 F. Therefore, use of minimum tumbling and use of drying temperatures below 167 F is recommended for impregnation of socks and gloves. A load of socks will not require much longer to dry than suits but the woolen gloves are very much slower to dry. Note: The indicating thermometers on the dryers must be checked periodically with a mercury thermometer. In one plant the thermometers were indicating 20 degrees too low and much impregnite was lost due to overheating.

LABORATORY TESTS AND ANALYSIS PROCEDURES

A. General Discussion

The successful impregnation of clothing depends in a large measure upon certain tests both on impregnating suspensions and impregnated clothing. The chemicals and equipment furnished with

the portable impregnating plant permit active chlorine or zinc oxide determinations to be made on suspensions and clothing.

Active chlorine determinations are most important since they give a measure of the protection to be expected from impregnated clothing. The method consists of extracting and dissolving the impregnate with a solvent. The active chlorine of the impregnate oxidizes iodide ion to free iodine which is titrated with thiosulfate.

Zinc oxide determinations consist of first dissolving the oxide in dilute sulfuric acid and then back-titrating the excess acid with sodium hydroxide solution.

B. Preparation and Standardization of Solutions

(1) Use of Fixinal Reagents

The primary standards furnished for standardizing the various reagents required in the chemical analysis are capsules containing accurately measured quantities of chemicals. The two types of fixinal capsules furnished are potassium dichromate N/10 and sodium hydroxide N/10. After removing one of the capsules from the cardboard container it should be washed carefully. Place the funnel part of the piercing apparatus in a one-liter volumetric flask making sure that the rubber seat protects the lip of the volumetric flask. Then place one of the sharp piercing rods down in the funnel so that it rests on the indentation with the point sticking up. Allow the indented end of the fixinal capsule to fall on the point and break. With the other piercing rod break the indentation in the side of the capsule. Use a wash bottle to wash all the materials out of the capsule into the flask. After this has been done and the capsule and funnel repeatedly washed to insure a quantitative transfer, make up the volume in the flask to the mark. After thorough mixing, this solution will then be one tenth normal. For making up a standard two-tenths normal solution it will be necessary to use two capsules or a 500-ml flask instead of a one-liter flask. The standard solution may now be transferred to a clean, dry bottle for storage. It is essential that all apparatus used in making up fixinal reagents be absolutely clean.

(2) Sodium Thiosulfate N/10

Weigh out 24.82 grams of thiosulfate crystals and dissolve in enough water to make one liter. After the crystals have all dissolved and the volume has been made up to one liter in a volumetric flask, the thiosulfate must be standardized against potassium dichromate. Prepare a N/10 potassium dichromate fixinal solution. From a burette run 20 ml of the standard dichromate into a 250-ml Erlenmeyer flask and add water to make a total volume of 100 ml. Add 5 ml of concentrated sulfuric acid and swirl the flask to mix the solution. Add a few crystals of potassium iodide and again swirl the flask to dissolve the crystals and aid in liberating the iodine. Titrate the iodine liberated with the thiosulfate solution to be standardized. When the solution is light yellow, indicating that most of the iodine is gone, add 5 ml of starch solution and continue titrating until the blue starch color is discharged. The color change will be from blue to green. Repeat the titration until check results are obtained within 0.1 ml.

Since the dichromate is N/10, 20 ml should require 20 ml of thiosulfate. Calculate the normality of the thiosulfate from the equation:

$$\text{ml} \times \text{normality} = \text{ml} \times \text{normality}$$

When the thiosulfate has been standardized label it with the normality as actually determined. It is not necessary that the thiosulfate be exactly N/10 but it should be within the limits 0.090-0.110.

(3) Sulfuric Acid N/5

Measure out 6 ml of concentrated acid in a 10-ml graduate and carefully pour it into about 50 ml of water. Allow the solution to cool, transfer it to a volumetric flask and make up to one liter. Standardize the acid by titrating against two-tenths normal sodium hydroxide fixinal solution. Pipette 25 ml of acid into an Erlenmeyer flask, add an equal volume of water and three drops of indicator. (see (6)). Titrate with sodium hydroxide from a burette (with a pinch-cock) until the color changes from purple to green. Calculate the normality of the acid after check titrations have been obtained.

(4) Sodium Hydroxide N/5

Weigh out 9 grams of sodium hydroxide pellets and by stirring, dissolve in 100 ml of water in a beaker. After all the hydroxide has dissolved and the solution cooled, transfer to a one-liter volumetric flask and make up to one liter. Standardize this base against sulfuric acid which has been recently standardized against the fixinal sodium hydroxide. Do not keep sodium hydroxide in the volumetric flask but transfer to a Pyrex bottle with a rubber stopper. Glass stoppers are liable to freeze and soft glass is gradually attacked by sodium hydroxide.

(5) Starch Solution

To make up a starch solution for use as an indicator in the thiosulfate-iodine reaction, weigh out 10 grams and dissolve in 100 ml of cold water. Add this to 900 ml of hot water and stir. When the solution has cooled it is ready for use. A few drops of chloroform will prevent mold formation.

(6) Methyl Red-Methylene Blue Indicator

This indicator is used in the acid-base titrations since its color change occurs at the proper pH for the zinc oxide titration. Weigh out 0.20 grams of methyl red and dissolve in 200 ml of methanol. Weigh out 0.10 grams of methylene blue and dissolve in 100 ml of water. Mix the two solutions. This indicator then consists of two parts of 0.1-percent methyl red solution and one part of 0.1-percent methylene blue solution.

(7) Titrating Solvent for Impregnite

The solvent used to dissolve the impregnite for titrating either suspension or cloth samples consists of a mixture of three parts of chloroform and seven parts of glacial acetic acid. Measure out 300 ml of chloroform and mix in a bottle with 700 ml of acetic acid. Be very careful in handling acetic acid and the mixture. Wash with water if any is spilled on the skin, since glacial acetic acid will cause blisters.

C. Analysis for Active Chlorine

(1) Impregnating Suspension

In analyzing a suspension for determining the CC-2 content it is absolutely essential that the suspension be shaken or stirred just before it is sampled. Weigh out between 2.0 and 2.5 grams of the suspension into a weighing bottle. After the exact weight has been taken to 0.01 gram, transfer the sample into an Erlenmeyer flask being careful to wash out all the sample into the flask with a wash bottle. Add 50 ml of chloroform-acetic acid mixture, stopper and shake. Remove stopper and wash it down with water, and add water if necessary so that a total of about 50 ml water is in the flask. Add 0.5 gram of potassium iodide, swirl the contents of the flask and titrate with thiosulfate. When the color has changed to a light yellow add 5 ml of starch solution and continue titrating until the blue color is just discharged. Some difficulty may be encountered at first with suspensions containing blue dye. With some experience, however, the end-point will be easy to detect.

The analysis of concentrated suspension should not be necessary. In the event that it is necessary as a check on how much XXCC-3 was added, the method of analysis will be essentially the same as outlined for a suspension of impregnating strength.

(2) Cloth Samples

Cloth samples are cut out by using a tool steel die to cut a disc of 1.5 inches in diameter. Place the cloth to be cut over the flat lead sheet taking care that it is not wrinkled or stretched. Place the die on the spot to be cut out and pound it with a hammer. Experience will show how hard it is necessary to pound. More than one layer of cloth may be cut at once.

Place from three to six discs in an Erlenmeyer flask and add 50 ml of chloroform-acetic acid mixture. Shake the flask vigorously and then allow it to stand with occasional shaking for ten minutes. Add about 50 ml of water washing down the stopper and sides. Add 0.5 gram of potassium iodide crystals and titrate with thiosulfate in the same manner as for analyzing suspension. Do not waste potassium iodide as it is expensive. It is suggested that the contents of the flask be kept swirling while the thiosulfate is being added. When

the end-point is reached, stopper the flask and shake vigorously. If the blue starch color does not return, the end-point has been reached; otherwise continue titrating until the blue color does not return on shaking.

D. Analysis for Zinc Oxide

(1) Suspension Samples

Weigh out above five grams of suspension and transfer to an Erlenmeyer flask as directed for active chlorine analyses. Add from a pipette 25 ml of 0.2N sulfuric acid. The wash water used in transferring the suspension sample from the weighing bottle should be about 25 ml. Shake the contents of the flask vigorously to make sure that the acid dissolves all the zinc oxide. Add three drops of methyl red-methylene blue indicator and titrate with 0.2N sodium hydroxide.

(2) Cloth Samples

Use from three to six cloth discs cut out with the die for analysis. Add 25 ml of 0.2N sulfuric acid from a pipette to the samples in the Erlenmeyer flask. Add an equal volume of water and shake the flask thoroughly. Shake the flask frequently over a period of ten minutes in order to extract all the zinc oxide from the fabric. Add three drops of indicator and titrate with sodium hydroxide. It is harder to get the zinc oxide out of cloth with the dilute acid than it is to dissolve out impregnate with solvent. Therefore it will be necessary to shake harder and longer for zinc oxide determinations. Do not attempt to determine the zinc oxide content of woolen gloves. Test only the Arnsen suits and the socks.

E. Methods of Calculating and Expressing Results

There are four or five methods of expressing the results obtained from titrating cloth samples for active chlorine. The method in use at the large impregnation plants is to express results in percent by weight of CC-2. This requires weighing out the cloth samples to be analyzed. It will be necessary to know this value for purposes of marking the clothing with the marking machine.

Another method of expressing the titration results is in units of milligrams of CC-2 per square centimeter. A third method is to express the results in units of milligrams of active chlorine per square centimeter.

Since the latter values range from 0.0 to 0.8 the fourth method is to express the results in units of tenths of a milligram of active chlorine per square centimeter.

The die used to cut out cloth discs has an area of 11.4 square centimeters. Therefore any of the units based on area can be used. To avoid weighing the cloth as well as cutting out a given area, the following factors are given to permit the conversion since percent CC-2 must be calculated for marking clothes. To convert milligrams of active chlorine per square centimeter ($\text{mg Cl} + / \text{cm}^2$) to percent CC-2 multiply by:

for Arzen suits	20
for socks	20
for gloves	8

In calculating the active chlorine value for cloth samples, it is necessary to know the number of die cuts used, the milligrams of thiosulfate required, and the normality of the thiosulfate. The formula for calculating the milligrams of active chlorine per square centimeter is:

$$\frac{\text{ml} \times \text{normality} \times 17.75}{\text{no. of discs} \times 11.4} \quad \text{or} \quad \frac{\text{ml} \times \text{normality} \times 1.555}{\text{no. of discs}}$$

The figure 17.75 is the number of milligrams of active chlorine equivalent to 1 ml of N thiosulfate. There are 11.4 square centimeters in one disc of cloth cut by the die. To change to units of mgCC-2/cm^2 divide by .145. Example: Three die cuts of Arzen cloth required 10.8 ml of 0.0963 N thiosulfate

$$\frac{10.8 \times 0.0963 \times 1.555}{3} = \begin{array}{l} .54 \text{ mg Cl} + / \text{cm}^2 \\ \text{or } 3.7 \text{ mg CC-2/cm}^2 \\ \text{or } 10.8 \text{ percent CC-2} \end{array}$$

In analyzing suspension samples the percent by weight of CC-2 in the suspension is desired. It is necessary to know the weight of the sample, the milligrams of thiosulfate required and the normality of thiosulfate. The formula is:

$$\frac{\text{ml} \times \text{normality} \times 0.01775 \times 100}{\text{wt of sample} \times 0.145} \quad \text{or} \quad \frac{\text{ml} \times \text{normality} \times 12.2}{\text{wt of sample}}$$

The figure 0.01775 is the grams of active chlorine equivalent to one ml of N thiosulfate, 0.145 changes weight of active chlorine to weight of CC-2, and 100 gives the answer in percent. Example: A 2.18-gram sample of suspension required 17.6 ml of 0.1071 N thiosulfate.

$$\frac{17.6 \times 0.1071 \times 12.2}{2.18} = 10.5\text{-percent CC-2}$$

The methods of calculating zinc oxide are similar. However, zinc oxide content is usually expressed in terms of percent by weight of the CC-2. This necessitates having an active chlorine determination for every sample that is given a zinc oxide test.

The formula for calculating milligrams per square centimeter of zinc oxide is:

$$\frac{\text{ml} \times \text{normality} \times 40.7}{\text{no. of discs} \times 11.4} \quad \alpha \quad \frac{\text{ml} \times \text{normality} \times 3.56}{\text{no. of discs}}$$

The milligram and normality refer to the amount of acid actually used by the zinc oxide. The burette reading of the sodium hydroxide is used to calculate the equivalent amount of acid. This value is subtracted from the total amount of acid and the difference represents acid neutralized by the zinc oxide. Example: Six discs of Arzen cloth required 18.1 ml of sodium hydroxide normality 0.1975. The acid used was 0.2046N. It is first necessary to convert the milligrams of base at its normality to milligrams of acid at the acid normality.

$$\text{ml } 0.2046 \text{ N acid} = \frac{18.1 \times 0.1975}{0.2046} \quad \text{or } 17.5 \text{ ml}$$

Therefore, 25-17.5 or 7.5 ml of acid was used by the zinc oxide.

$$\frac{7.5 \times 0.2046 \times 3.56}{6} = .908 \text{ mg ZnO/cm}^2$$

Assuming that the cloth analyzed for 3.8 mg CC-2/cm², then the percent ZnO would be .908/3.8 or 25 percent.

Analysis of suspensions for zinc oxide also requires that the burette reading of the base be converted to its acid equivalent at the normality of the acid. Knowing the amount of acid actually used by the zinc oxide and its normality the percent zinc oxide is calculated by the formula:

$$\frac{\text{ml} \times \text{normality} \times 4.07}{\text{wt of sample}} = \text{percent ZnO in sample}$$

The figure 4.07 is one hundred times the weight of zinc oxide, equivalent to one ml of N acid.

INSPECTION, HANDLING, AND MARKING OF CLOTHES

A. Inspection and Surveying Clothing

Under certain conditions the reimpregnation of clothing might become a major factor for the portable impregnating plants. It is necessary, therefore, to be able to examine impregnated clothing carefully with regard to strength. First examine all garments for tears, rips or other imperfections which might permit passage of gas. If any defects are found, the clothing should be discarded or repaired. While the clothing is being examined for defects it should be tested for strength. There is no quantitative method of doing this in a simple manner and much will depend on the experience of the man in charge. Grab a single layer of cloth in the pants or jacket with both hands and give it a quick hard tug. If it gives way the cloth has rotted and must be discarded. Another test is to try to poke a thumb or pencil through the cloth. If this can be done easily the clothing should be discarded. Another simple test is to pull on some of the loose strands at the end of the drawstring around the bottom of the jacket. Until experience is gained in testing for strength, an unimpregnated garment should be used for reference.

Socks and gloves should also be tested for deterioration in a similar manner. However, since the socks are part wool and the gloves are all wool, less deterioration is to be expected from the standpoint of loss of strength.

If the clothing has been found satisfactory from the standpoint of defects and strength, it should be tested next for active chlorine content. However, if the clothing is dirty and requires laundering it is best to wait until after laundering for making active chlorine tests. In the event that the garments appear to have an adequate active chlorine loading and may not require reimpregnation, check the results on representative samples. If this shows that the clothing is adequately impregnated, it may be reissued without being reimpregnated.

A value of 6-percent CC-2 has been established as the lower limit for clothing which is to be reissued from any impregnating plant. In case of doubt, it is always safer to reimpregnate clothing.

Clothing that has been worn, especially if exposed to gas, must be examined thoroughly and completely.

The area of suit to be analyzed for impregnite content is determined by its past history. If the clothing has always been in storage, use the pockets or pocket flaps for analysis in order to save the garment. Suits that have been worn or exposed to mustard gas must be examined in areas most likely to have lost impregnite. For the jumper this is the top of the shoulder, the elbow, and the hood. The crotch, seat, and knee should be tested on the trousers. Since sunlight causes impregnated clothing to deteriorate more rapidly, carefully examine those areas of suits that have been unduly exposed to light.

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