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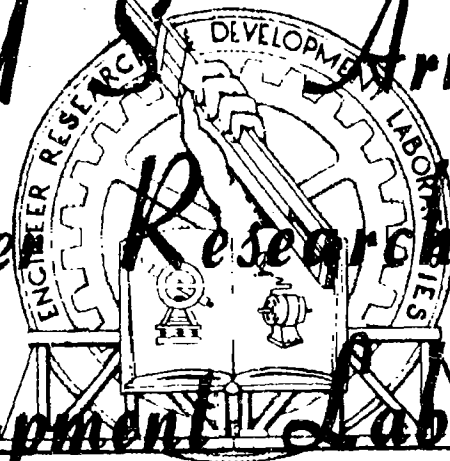
Research Report 1749-RR

REMOVAL OF CHEMICAL WARFARE AGENTS FROM
WATER BY VAPOR COMPRESSION DISTILLATION

Task 8M75-05-001-07

7 June 1963

U S Army
Engineer Research And
Development Laboratories



FORT BELVOIR, VIRGINIA

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AD U. S. Army Engineer Research and Development Laboratories, Fort Belvoir, Virginia - REMOVAL OF CHEMICAL WARFARE AGENTS FROM WATER BY VAPOR COMPRESSION DISTILLATION - Maurice Pressman, Robert P. Amman, and Virginia E. Bauer Report 1749-BB, 7 June 63, 20 pp, 26 illus, 33 tables DA Task 9675-05-001-07 Report covers a field study of the removal of seven chemical warfare (CW) agents from fresh and saline water by vapor compression distillation. Report concludes: (a) The Army's vapor compression distillation unit does not produce an acceptable distillate from fresh water contaminated with BZ, VX, A, GB, or TA; (b) the Army's vapor compression distillation unit does not produce an acceptable distillate from saline water contaminated with BZ, VX, or A; (c) mixed-bed ion exchange resin, containing a strongly acidic cation resin and a strongly basic anion resin, is effective for post-treatment of distillate containing all the CW agents studied; (d) granular activated carbon is effective for posttreatment of distillate containing all the CW agents studied, except A; (e) a small-scale batch coagulation process involving superphosphatation, activated carbon dechlorination, coagulation, and filtration is capable of effectively removing VX from a contaminated fresh water source; and (f) further study is required to determine operating characteristics of activated carbon and ion exchange resins in order to establish design criteria of auxiliary equipment for posttreatment of distillate from sources contaminated with CW agents.	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U. S. ARMY ENGINEER RESEARCH AND DEVELOPMENT LABORATORIES
FORT BELVOIR, VIRGINIA

(19) Research Report 1749-RR

(10) REMOVAL OF CHEMICAL WARFARE AGENTS FROM WATER

BY VAPOR-COMPRESSOR DISTILLATION

(11) Task 8M75-05-001-07

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BEEN APPROVED BY THE DEPARTMENT OF THE ARMY.

PREFACE

The investigation covered by this report was conducted by the Sanitary Sciences Branch, U. S. Army Engineer Research and Development Laboratories, Fort Belvoir, Virginia, under authority of Task 8M75-05-001-07, "Removal of CBR Contaminants from Water." A copy of the task card is included as Appendix A.

The period covered by this report is 12 September to 21 November 1962, during which time the basic subtask accomplished was the "Removal of Chemical Warfare Agents from Water by Vapor Compression Distillation." The following personnel were responsible for the acquisition and presentation of the data:

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o The Army Medical Service provided partial financial assistance
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SUMMARY

A field study of the removal of seven chemical warfare (CW) agents from fresh and saline water by vapor compression distillation was conducted at Graces Quarters, Maryland, during the period 12 September through 21 November 1962. The objectives of the study were: (1) To determine whether a U. S. Army vapor compression distillation unit could produce an acceptable distillate from fresh and saline water contaminated with seven CW agents (incapacitating agent, BZ; nerve gases, GA, GB, and VX; blood gas, AC; and vesicants, HD and HN-3); (2) in the event the unit did not produce a potable distillate, to develop auxiliary procedures, either pretreatment or posttreatment, capable of producing an acceptable water; (3) to study the vapor hazard in the operating area; and (4) to evaluate a small-scale batch coagulation process (not involving distillation) for removing VX from fresh water.

The results of this study indicate that:

- a. The Army's vapor compression distillation unit does not produce an acceptable distillate from fresh water contaminated with BZ, VX, AC, GB, or GA.
- b. The Army's vapor compression distillation unit does not produce an acceptable distillate from saline water contaminated with BZ, VX, or AC.
- c. Mixed-bed ion exchange resin, containing a strongly acidic cation resin and a strongly basic anion resin, is effective for posttreatment of distillate containing all the CW agents studied.
- d. Granular activated carbon is effective for posttreatment of distillate containing all the CW agents studied, except AC.
- e. A small-scale batch coagulation process involving superhypo-chlorination, activated carbon dehydrochlorination, coagulation, and filtration is capable of effectively removing VX from a contaminated fresh water source.
- f. Further study is required to determine operating characteristics of activated carbon and ion exchange resins in order to establish design criteria of auxiliary equipment for posttreatment of distillate from sources contaminated with CW agents.

REMOVAL OF CHEMICAL WARFARE AGENTS FROM WATER
BY VAPOR COMPRESSION DISTILLATION

I. INTRODUCTION

1. Subject. The objectives of this study were as follows:

- a. To determine the efficiency of the vapor compression distillation unit in the decontamination of water containing chemical warfare (CW) agents.
- b. To determine auxiliary water treatment procedures required to produce a potable water from a possibly unacceptable distillate.
- c. To study the vapor hazard in the operating area.
- d. To conduct related studies of a small-scale batch coagulation process not involving distillation.

2. Background and Previous Studies. Buswell, Sanchis, and Hudson (1) conducted the first study in the use of vapor compression distillation for the removal of CW agents from water. In their study, a 1,500-gallon-per-day distillation unit was evaluated with water contaminated with sulfur mustard (H), lewisite (L), and nitrogen mustard (HN-3). It was determined that the distillate produced was safe for 1-week consumption but was not palatable because of strong odors. Further investigation showed that dosages of up to 5 pounds of powdered activated carbon per 1,000 gallons of distillate followed by coagulation, sedimentation, and filtration produced a potable water.

Laboratory studies by several investigators pertaining to CW agent decomposition have provided valuable information which was used in predicting agent action during distillation in this study. Studies by Epstein and Bauer (2, 3), of the hydrolysis of Sarin (GB) in water, determined that the rate of hydrolysis is dependent on pH, temperature, and type and concentration of dissolved solids. The rate of hydrolysis of Sarin increased approximately 10 times per unit decrease of pH below 4, and at a given pH, the rate of hydrolysis doubled for each 10° C increase in temperature.

Additional work by Epstein (4) indicated that at a pH above 6.5, hydrolysis increased 10 times per unit increase in pH. It was also pointed out that hydrolysis is accelerated by anions,

with the exception of hydroxyl ions. The presence of calcium and magnesium ions in alkaline waters and cupric and manganous ions in acid waters was effective in increasing hydrolysis.

In considering the possible methods of additional distillate treatment, it was found that Epstein and Flannery (5); Epstein, Flannery, and Wessels (6); and Epstein (7) had removed GB from water with activated carbon and also with both cation and anion exchange resins. These studies indicated that activated carbon is effective in the removal of Tabun (GA) and Sarin (GB) and that pH, temperature, and hydrolysis products have little effect on the performance of the carbon. These studies also indicated that the resins act as catalysts in the decomposition of GB and that a strong-base anion resin is the most effective.

Lindsten and Bauer (8, 9) studied the removal of VX from water by use of cation exchange resin, in the hydrogen and sodium form, and a mixed-bed resin. The latter resin was the most effective of the three for removing VX from water contaminated to the level of 13.1 mg/l.

3. Maximum Permissible Concentration (MPC). In order to carry out an adequate evaluation of any item of water treatment equipment, it is necessary to know, first, what is to be removed and, second, what are the maximum amounts which can be permitted to remain in the water. With certain chemicals, the limits are clearly defined, based upon toxicological and other investigations. There are certain chemicals, however, for which the maximum limits cannot presently be completely defined. At the present time, The Surgeon General is making a critical review of toxicological information with the objective of determining more acceptable standards. Where such information is not adequate, further research will be necessary. Due to military expediency, however, it is often necessary to defined maximum limits based upon the best information available. Therefore, for the purpose of this study, the MPC values which are used and discussed are those which are presently being considered for adoption by the United States, United Kingdom, and Canadian Armies. Certain of these limits are still subjects of considerable discussion. For that reason, all analytical work carried out in this study was done quantitatively so that in the event new limits are established, the data contained in this report will still be usable. At the present time, the maximum limit for BZ is not well defined and is that which is presently being utilized by Chemical Corps Agencies dealing with this material. For the purpose of this study, the following 7-day maximum permissible concentrations were utilized:

Blood Gas (AC)	20.0 mg/l
Mustards (HD and HN-3)	2.0 mg/l
Nerve Gas (GA)	0.5 mg/l
Nerve Gas (GB)	0.1 mg/l
Nerve Gas (VX)	0.005 mg/l
BZ	0.01 mg/l

II. INVESTIGATION

4. General Layout and Test Equipment. The test site was located at Graces Quarters, Edgewood Arsenal, Maryland. Figure 1 is a diagram of the test site and equipment arrangement. Shifflett Pond at Graces Quarters was the source of fresh water. Figure 2 shows equipment set up to pump the water from the pond to a 2,000-gallon tank truck. Characteristics of this water were high color, low dissolved mineral content, pH 6.8 to 7.2, and a turbidity of approximately 50 units. The saline water was hauled to the site in 2,000-gallon quantities by tank truck (Fig. 3) from the lower Chesapeake Bay area. Analyses of this water indicated a dissolved mineral content of approximately one-third that of sea water and low turbidity and color. Standard military, collapsible, nylon-rubber tanks were used for storage of raw water and its subsequent contamination with the CW agents.

A prototype vapor compression distillation unit was used as the study unit. Figures 4 and 5 show the unit with associated equipment. Constructed of aluminum, it is a prototype of the proposed standard Army, trailer-mounted, 125-gph distillation unit. A schematic flow diagram is shown in Fig. 6. Feedwater is pumped through heat exchangers, where it picks up heat from the distillate and blowdown, and then through the engine water cooler, where it receives heat from the engine exhaust, before entering the evaporator. The water then passes up through the tubes of the evaporator and is partially vaporized. A concentrated brine (blowdown) is continuously drawn off through the heat exchanger and discharged to waste. The steam at approximately 212° F passes to the compressor, where compression raises its temperature to about 224° F. Upon being returned to the steam chamber surrounding the tubes of the evaporator, the steam condenses, gives up its latent heat of condensation to the incoming feedwater in the tubes, and passes out of the system as distillate through the heat exchanger. The temperature of the discharged distillate is about 10° F to 15° F above the incoming feedwater temperature.

The pressure on the suction side of the compressor is adjusted to approximately 6 inches of water above atmospheric pressure.

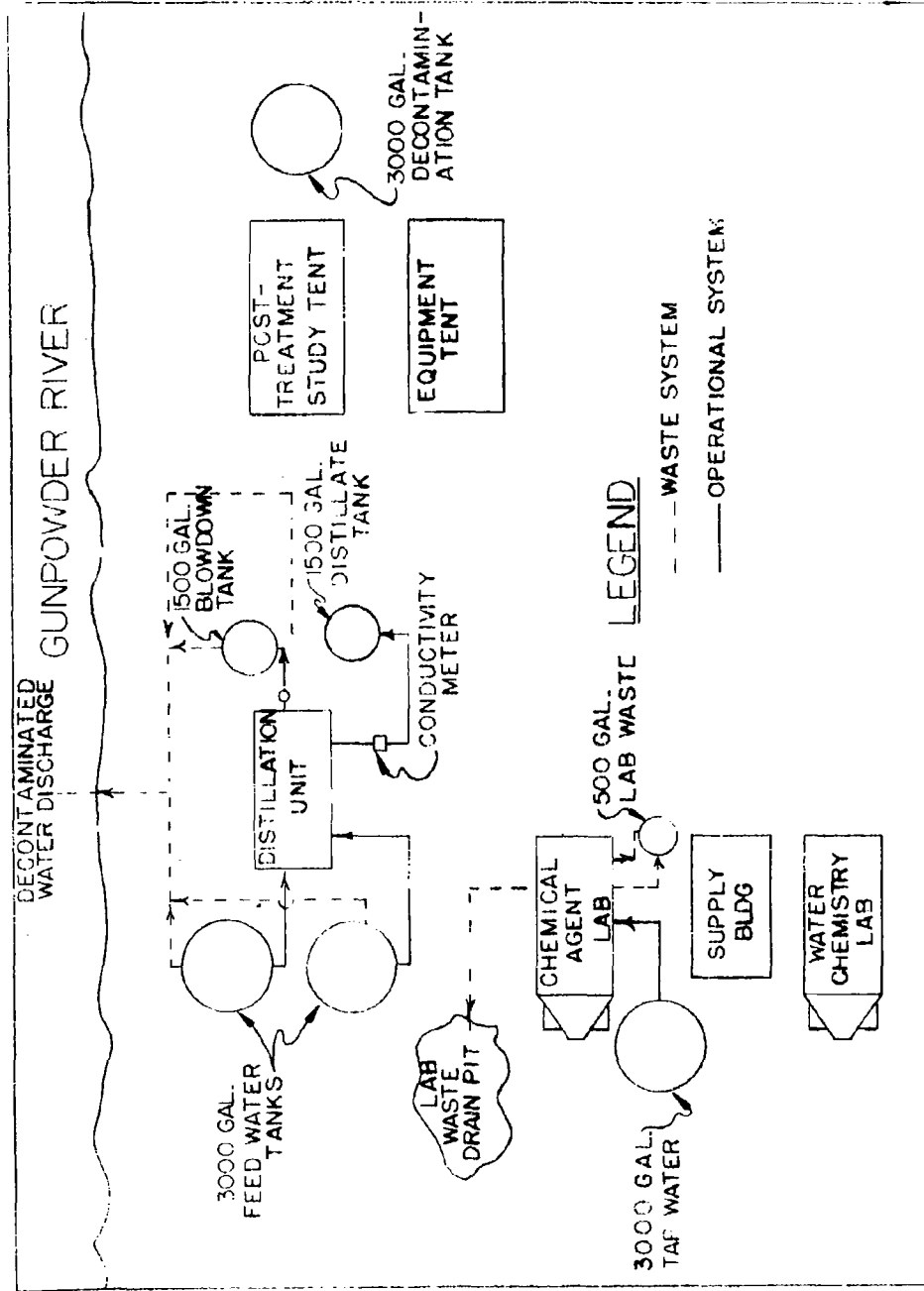
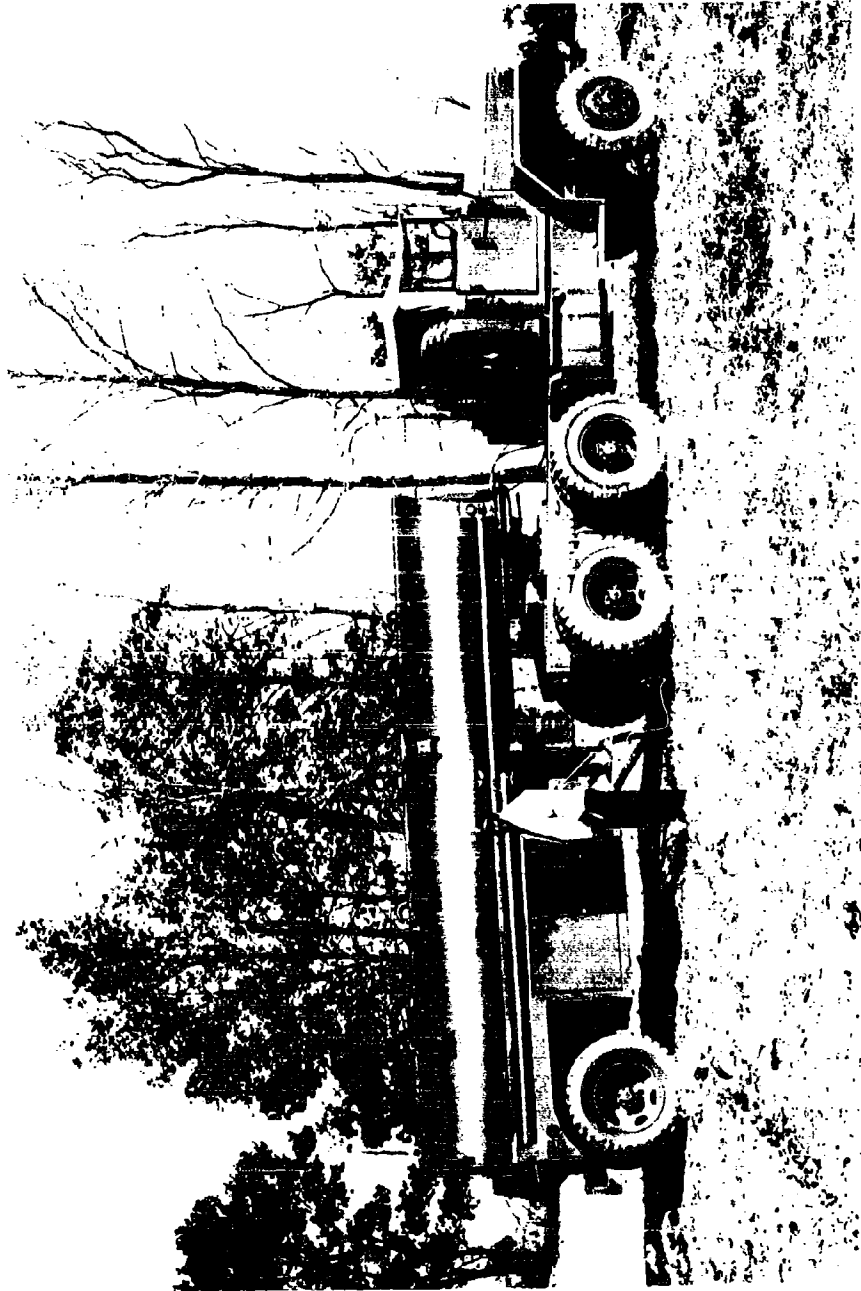


Fig. 1. Test site layout.



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Fig. 2. Shifflett Pond, Graces Quarters, Maryland, source of fresh water.



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Fig. 3. Tank truck used for hauling water.



Fig. 4. Distillation unit and associated equipment.

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On the discharge side, the pressure may vary from 2.5 to 5 psig, depending on scale formation in the evaporator. During operation, the volume of water held in the evaporator is 44 gallons and the average detention time is 22 minutes.

The two mobile laboratories shown in Fig. 7 were used for performing all analytical determinations at the test site.

5. Procedures. The distillation unit was operated typical of its intended use in the field. Seven CW agents were used. Five of these agents are described in Table I. Descriptions of two agents are omitted because of classification.

a. Distillation Study. The agents were delivered to the site by boat from Edgewood Arsenal (Fig. 8), usually in 100-gram quantities, on the day a run was made. In some cases, the agent had to be treated before it was added to the feedwater tank. The agent BZ was dissolved in a minimum quantity of hydrochloric acid. Because of low water temperatures and the limited solubilities of the mustards, the HD and HN-3 were dissolved in acetone before they were added to the feedwater tank. The agent AC (HCN) was produced in situ by the addition of solid sodium cyanide to the tank of water, followed by the addition of a stoichiometric amount of hydrochloric acid.

The other three agents used as contaminants were liquids which required no special preparation. While the agent was being poured into the tank, the water was stirred manually. After contamination was completed, the feedwater was recirculated constantly to maintain a uniform mixture. During all preparatory work, protective clothing was worn by operating personnel (Fig. 9). After preparation of the contaminated water, personnel involved were thoroughly decontaminated by use of a 200-ppm calcium hypochlorite solution (Fig. 10).

The unit was put into operation immediately after water decontamination. A warmup time of 1 hour was usually required before distillate was produced. An additional half hour was allowed for stabilization of distillate production. After the unit was stabilized, samples of raw water, blowdown, and distillate were collected hourly for chemical agent analysis; blowdown and distillate samples were also collected for other analytical determinations. Vapor samples were taken hourly at the blowdown sump (Fig. 11) and at the compressor. Random vapor samples were taken in the general environment and over the raw water tank during a run. A continuous-reading conductivity meter was also used to monitor the quality of distillate.

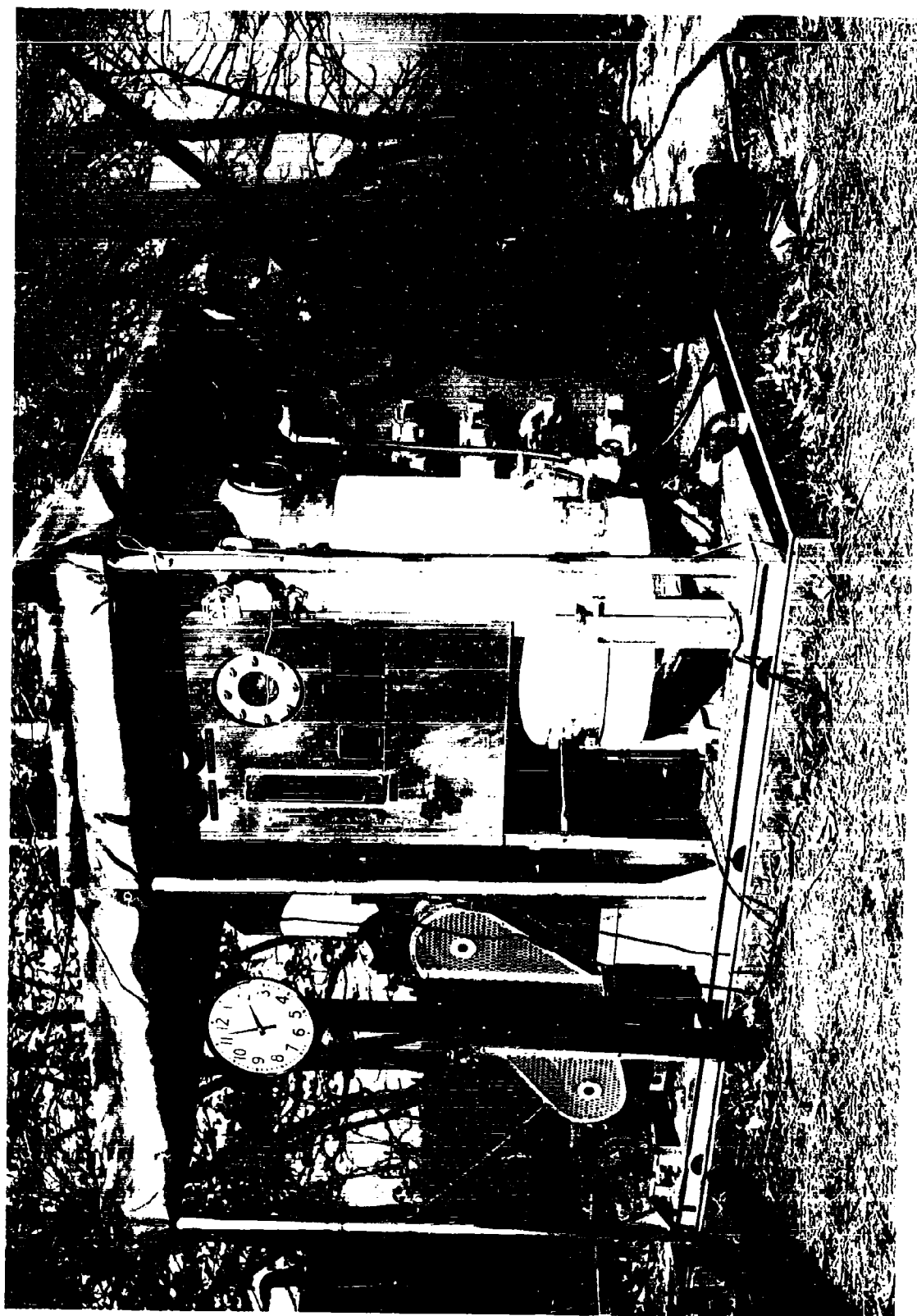


Fig. 5. Distillation unit.

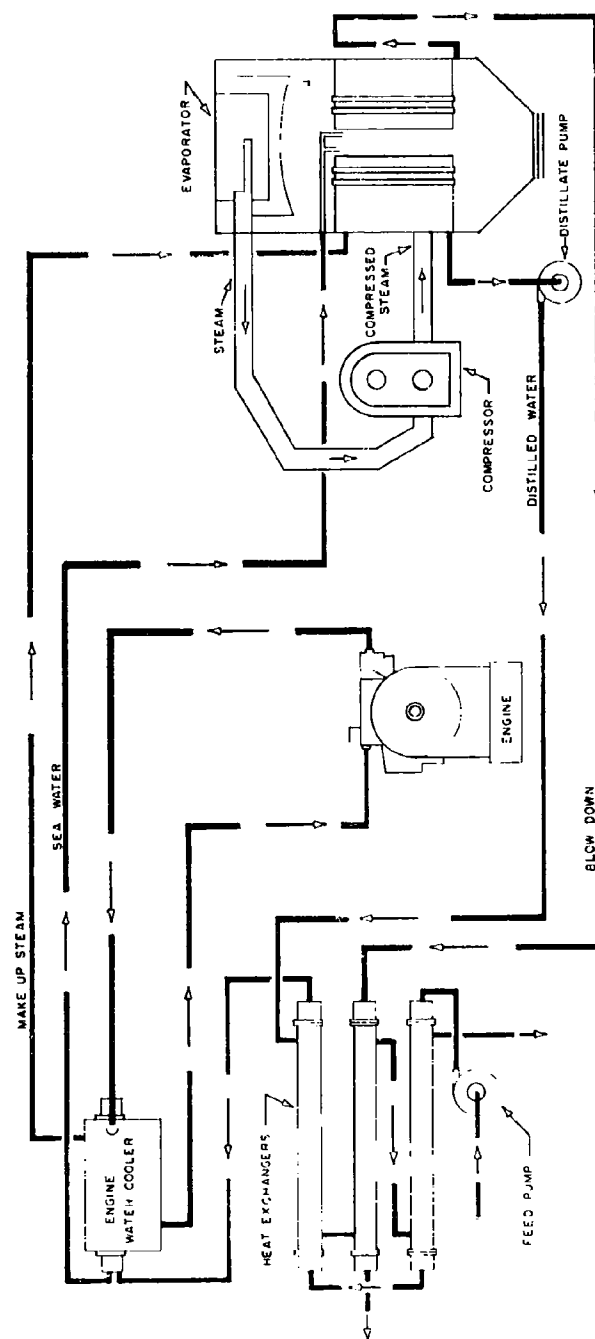


Fig. 6. Flow diagram of vapor compression distillation unit.



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Fig. 7. Mobile laboratories and storage shed.

Table I. Characteristics of Various CW Agents

Symbol	Agent	Formula	Freezing Point (°F)	Boiling Point (°F)	Decomposition Temperature (°F)	Hydrolysis Product
GA	Tabun	$\text{CN}(\text{CH}_3)_2\text{NP}(\text{O})(\text{OC}_2\text{H}_5)$	-57	159	264	NaCN and a substituted phosphonate (alkaline solution) $\text{HN}(\text{CH}_3)_2$ and $\text{C}_2\text{H}_5\text{O}(\text{OH})\text{P}(\text{O})(\text{CN})$ (acidic solution)
GB	Sarin	$\text{F}(\text{CH}_3)_2\text{CHOP}(\text{O})\text{CH}_3$	-36	297	Not available	HF in both alkaline and acidic solution
HD	Sulfur Mustard	$(\text{ClCH}_2\text{CH}_2)_2\text{S}$	58	142	300 to 350	Hydrogen chloride and thiodiglycol
HM-3	Nitrogen Mustard	$\text{N}(\text{CH}_2\text{CH}_2\text{Cl})_3$	39	Decomposes	Not known	Not identified
AC	Hydrogen Cyanide	HCN	7	79	151 Very stable	NH_3 , HCOOH

NOTE: BZ and VX are not included in this table because of security classifications. Further information is contained in TM 3-215 (10) and TM 5-700 (11).

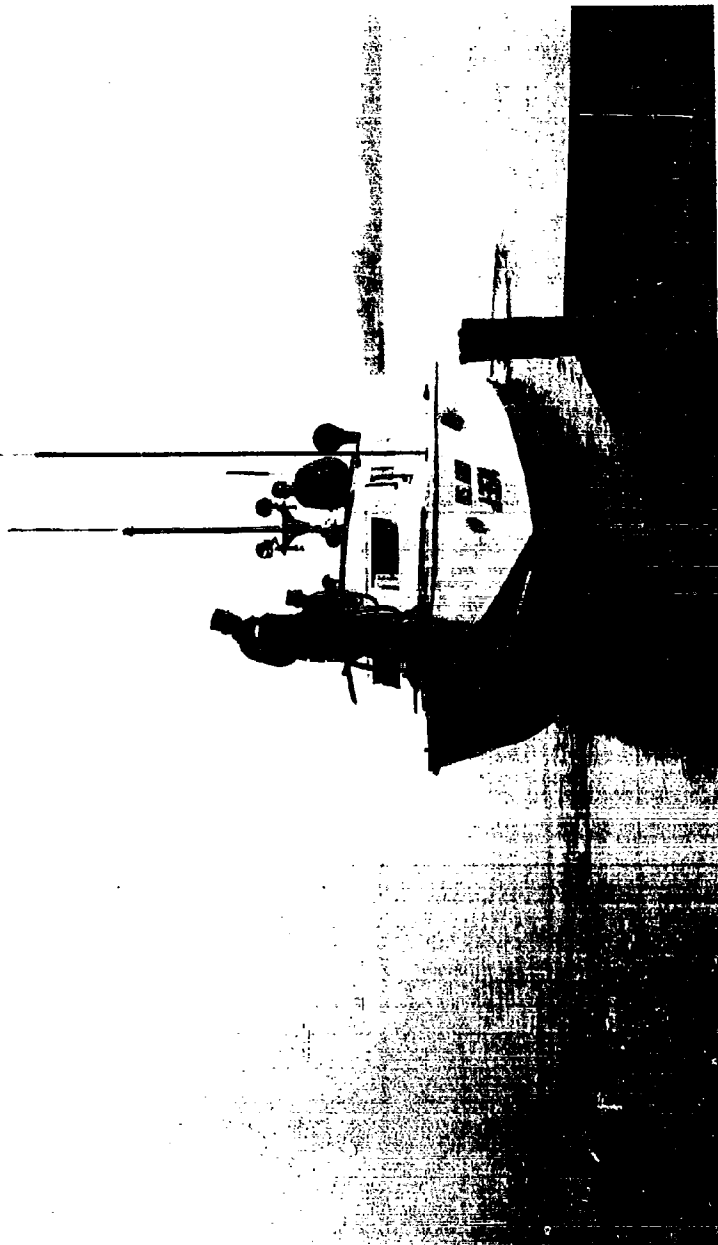


Fig. 8. Boat used to transport CW agents to test site.

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Fig. 9. Handling of agent prior to raw water contamination.



Fig. 10. Decontamination of personnel.

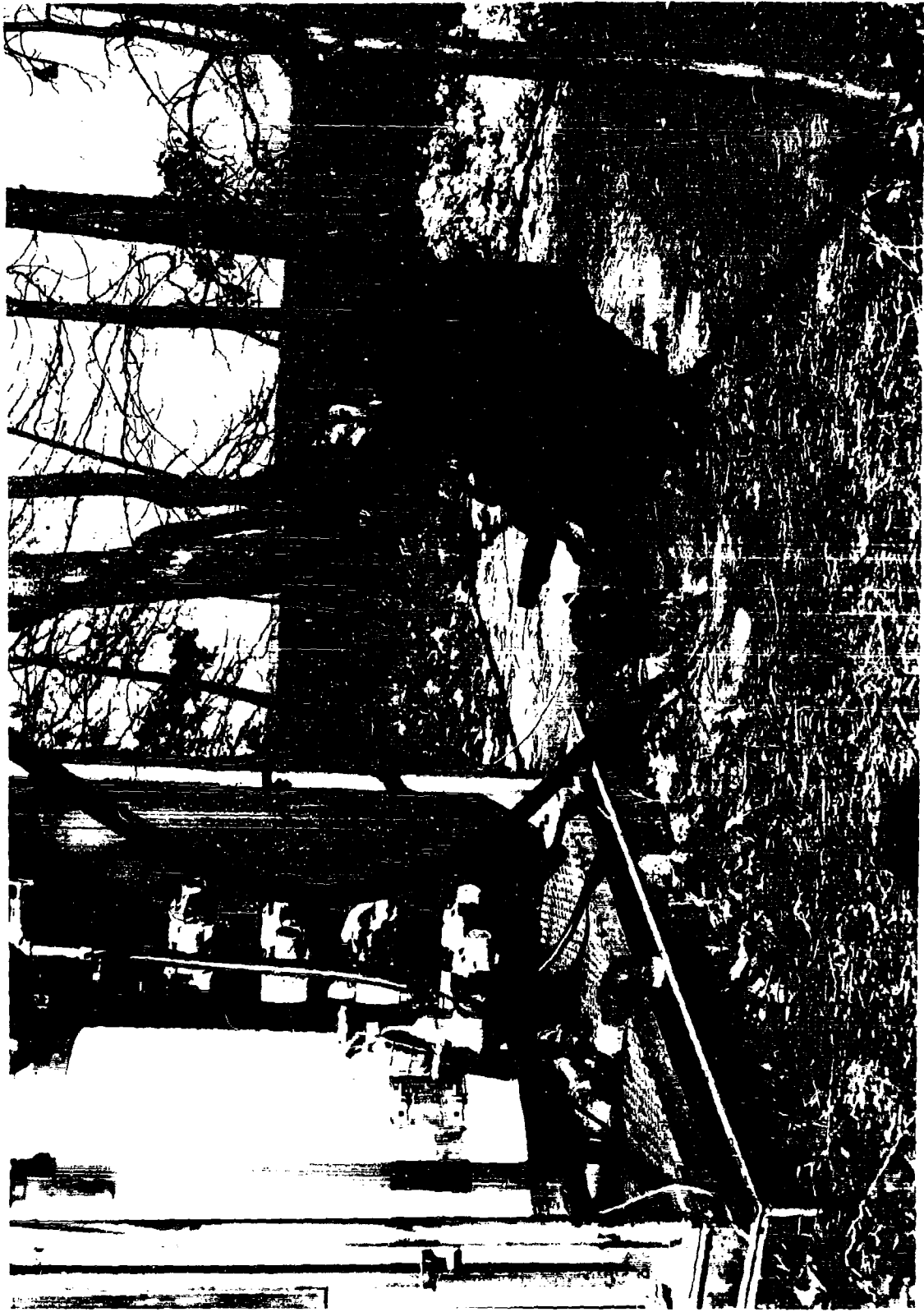


Fig. 11. Vapor sampling at blowdown sump.

During the course of a run, samples of feedwater, blowdown, and distillate were taken hourly for the various necessary analytical determinations. Agent concentration in each hourly sample was determined. Other determinations varied with the sample.

The water analyses performed on Shifflett Pond raw water were pH, color, turbidity, hardness, alkalinity, chlorides, and sulfates. The same analyses except those for hardness and sulfates were performed on saline raw water. The hourly blowdown sample was analyzed for pH, alkalinity, and chlorides. The distillate sample was analyzed for pH and chlorides. Laboratory work areas are shown in Figs. 12, 13, and 14. All procedures except those for agent detection were performed in accordance with Standard Methods for the Examination of Water and Wastewater, 11th Edition.

(1) Hydrogen Ion Concentration (pH). Portable Beckman pH meters, Models N and G, were used. Both units were standardized several times a day with standard buffer solutions.

(2) Color. The Hellige Aqua Tester with a standard color disc was used.

(3) Turbidity. For waters above 25 units, the Jackson candle turbidimeter was used. For water under 25 units, the Hach meter was used, but only to give an order of magnitude.

(4) Alkalinity (as CaCO_3). A standard solution of 0.020 N sulfuric acid solution, with a mixed indicator of methyl orange - xylene cyanol, was used to determine the total alkalinity.

(5) Hardness (as CaCO_3). Total hardness was determined by the EDTA (ethylenediaminetetraacetic acid) titration method, using an ammonium chloride - ammonium hydroxide buffer and Eriochrome Black T as the indicator.

(6) Chlorides (as Cl). Two methods of determining chlorides were used, depending upon the range of chlorides in the water. For water low in chlorides, the mercuric nitrate titration was used. This method used 0.0141 N mercuric nitrate solution and diphenylcarbazone-bromphenol blue mixed indicator. For the waters of high chloride content, a 0.10 N silver nitrate solution and a potassium chromate indicator were used.

(7) CW Agents. Analytical methods used in determining agent concentration are summarized in Table II. This table also shows the sensitivity of each method and the maximum limit



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Fig. 12. Colorimeter used in agent concentration analyses.

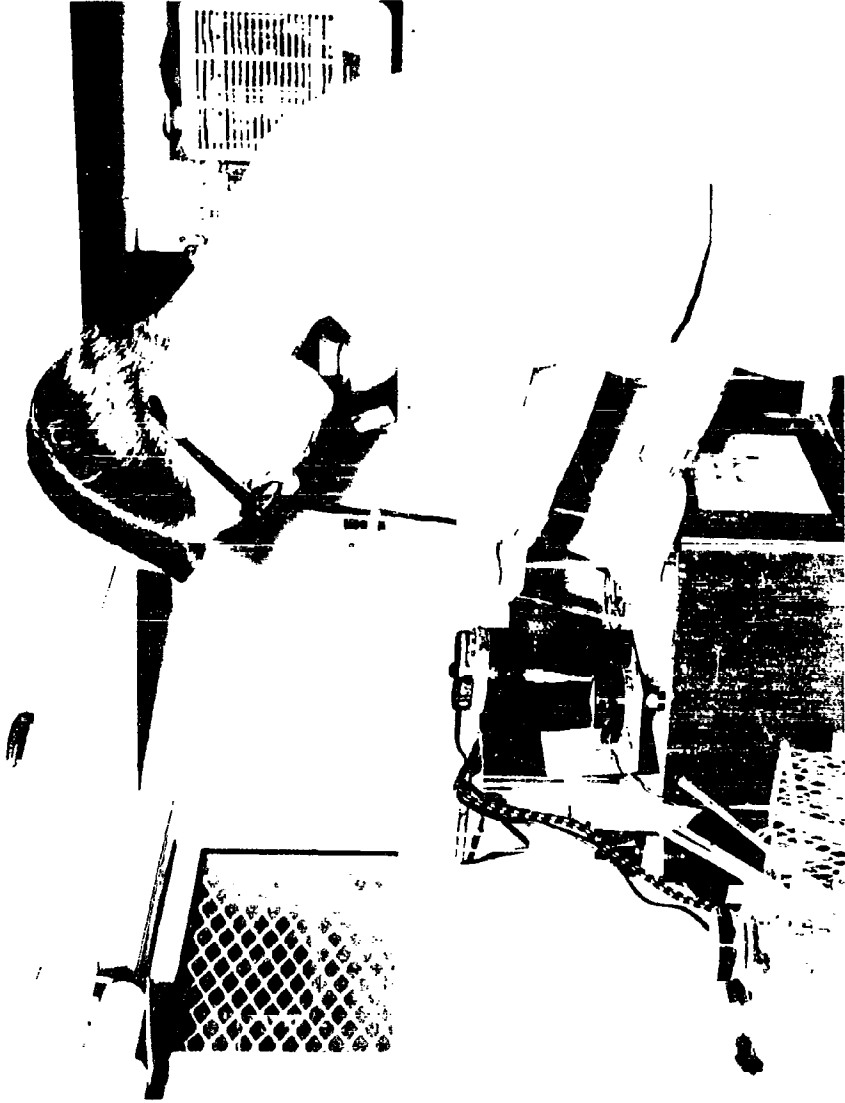


Fig. 13. Constant temperature bath used in agent concentration analyses.

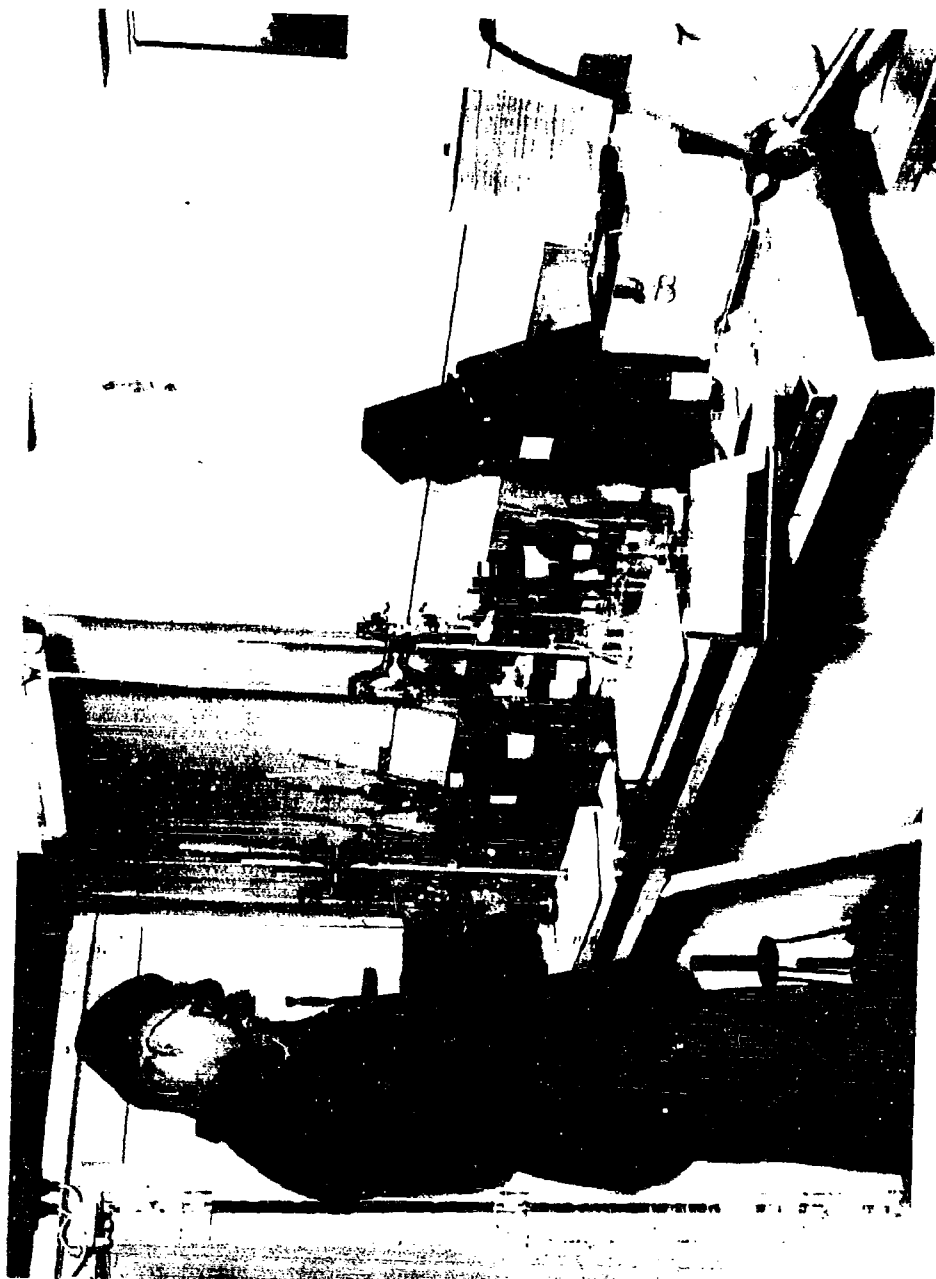


Fig. 14. Interior of water chemistry laboratory.

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Table II. Analytical Methods for Determining CW Agents in Water

Agent	Name	Method	Sensitivity		Remarks
			of Method (mg/l)	APC (a) (mg/l)	
BZ	(b)	(b) (c)	0.010	Recommended - 0.01	None
VX	(b)	(b) (d)	0.0005	0.005	None
GB	Sarin	Schoenemann (e)	0.10	0.10	Shifflett Pond water showed interference
		Chemical Enzyme	0.005	0.10	None
GA	Tabun	Schoenemann (e)	0.10	0.5	Shifflett Pond water showed interference
		Chemical Enzyme	0.005	0.5	None
		DB-3	2.0	0.5	Determination of cyanide (hydrolysis product of GA)
AC	Hydrogen Cyanide	DB-3	0.25	20	None
HN-3	Nitrogen Mustard	DB-3	1.25	2	None
HD	Sulfur Mustard	DB-3	3.0	2	None
	Chloramine T		4.0	2	Thiodiglycol (hydrolysis product of HD)

(a) Maximum permissible concentration (ref par 3).

(b) Available, but classified.

(c) See Reference 12.

(d) See Reference 9.

(e) See Reference 13.

as specified in paragraph 3 and notes interferences encountered in some procedures.

(8) Vapor Detection. Vapor sampling was done with the standard Army chemical agent test kit (M-18). This kit was supplemented with experimental enzyme tickets for the detection of V and G agents.

After each run, the holding tanks were decontaminated prior to release of the water into the Gunpowder River. Waters containing V agent were decontaminated by use of calcium hypochlorite and hydrochloric acid. The AC-contaminated and G-contaminated water was treated with calcium hypochlorite. The mustards were allowed to decay for 2 days before the water was released.

b. Carbon and Resin Column Studies. This portion of the study was designed to provide general information regarding the capability of granular activated carbon and ion exchange resin columns to remove CW agents from water and to indicate the direction of further study in the development of auxiliary equipment for the treatment of unacceptable distillate.

Carbons used were Nuchar WA (+30 mesh) and Nuchar C-190 (+30 mesh). Three ion exchange resins used were Duolite C-25, a strongly acidic cation resin; Duolite A-30B, an intermediate base anion resin; and a mixed resin obtained from laboratory demineralization units (Barnstead Red). The mixture contained a strongly acidic cation resin and a strongly basic anion resin. Table III gives some characteristics of these column materials.

Columns were prepared by using 5-cm-diameter glass cylinders, 60 cm in length. The column medium, 500 ml of dry granular carbon or 500 ml of wet resin added as a slurry, was supported by a one-hole rubber stopper covered with a fine nylon screen and a 5-cm layer of glass wool. The top of the column medium was covered with a glass wool plug about 5 cm thick. This prevented the feedwater stream from disturbing the medium and also acted as a filter for any incidental turbidity in the column feedwater. Before use, each column was washed with 3 or 4 liters of demineralized water. A typical column arrangement is shown in Fig. 15.

Contaminated water fed to the columns varied widely in composition. Straight distillate, spiked distillate, distillation unit feedwater, and mixtures of feedwater and distillate were used. In all cases except Test 1 (with BZ-contaminated feed), the flow rate through the column was adjusted to 150 ml/min (2.2 gpm/cu ft) by use of a Hoffman screw clamp. Samples of column feedwater

Table III. Characteristics of Granular Carbons and
Ion Exchange Resins Used in Column Studies

Characteristic	Carbons		Ion Exchange Resins			
	Trade Name		Type	Characteristic	Cation	Anion
	Nuchar WA	Nuchar C-190				
Odor	None	None				
Taste	None	None	Resin Composition	Polystyrene-sulfonic acid	Cross-linked polyalkylene-amine	
Mesh Range	4-30	4-30				
Apparent Density, Packed (lb/cu ft)	9-11	7-10	Active Groups	Sulfonic acid	Tertiary and quaternary amine	
Specific Surface Area (m^2/gm)	550-650	700-900	Divinylbenzene (%)	5.5	Not applicable	
Voids (packed column) (%)	75-80	75-80	Physical Form	Beads	Beads	
pH (distilled water extract)	8-10	8-10	Mesh Range	16-50	10-50	
Total Ash (max) (%)	6	6	Volume Change (%)	-4 (HR to NaR)	+3 (ROH to RCl)	
Water Solubles (max) (%)	3	3	Maximum Temp. ($^{\circ}C$)	120	80	
			Total Exchange Capacity (meq/ml)	1.6-1.7	2.6 (to HCl)	

and effluent were analyzed for pH and agent concentration. Waters containing six of the seven agents used in this study were fed to the columns. Agent HD was excluded since it was found that the distillation unit adequately removed both mustards from contaminated feedwater.

c. Batch Process Decontamination Study. This study was designed to determine the effectiveness of a batch coagulation process for removal of a typical CW agent from fresh water. The equipment used was a lyster bag, a filter (Water Purification Unit, Hand-Operated, Knapsack-Pack, Filter-Pad-Type, 1/4-GPM), and a mixed-bed ion exchange column (Fig. 16). Shiflett Pond water contaminated with 18.7 mg/l of VX was used for the test. The procedure involved was as follows: The lyster bag was filled with 36 gallons of contaminated water. Twenty grams of calcium hypochlorite (70 percent available chlorine) was added to provide a dosage of 100 mg/l available chlorine. This was followed by the addition of 82 grams of powdered carbon (600 mg/l), 7 grams of ferric chloride (50 mg/l), and 14 grams of limestone (100 mg/l). The coagulated water was pumped through the 1/4-gpm filter unit and then passed through a mixed-bed resin column, and the effluent was collected in a plastic drum. One-half-hour waiting periods were observed following hypochlorination, carbon addition, and coagulation.

6. Results. The results of each of the 20 distillation runs are given in Appendix B, Tables VI through XXV. Table VI shows the results of an agent-free run which was carried out to obtain control data. A summary of results of all the distillation runs is given in Table IV.

The results of the vapor sampling are given in Appendix C, Tables XXVI through XXIX.

Carbon and resin column study results are shown in Appendix D, Tables XXX through XXXVIII. A summary of results of the column studies is given in Table V.

The results of the batch coagulation decontamination study are presented in Appendix E, Table XXXIX.

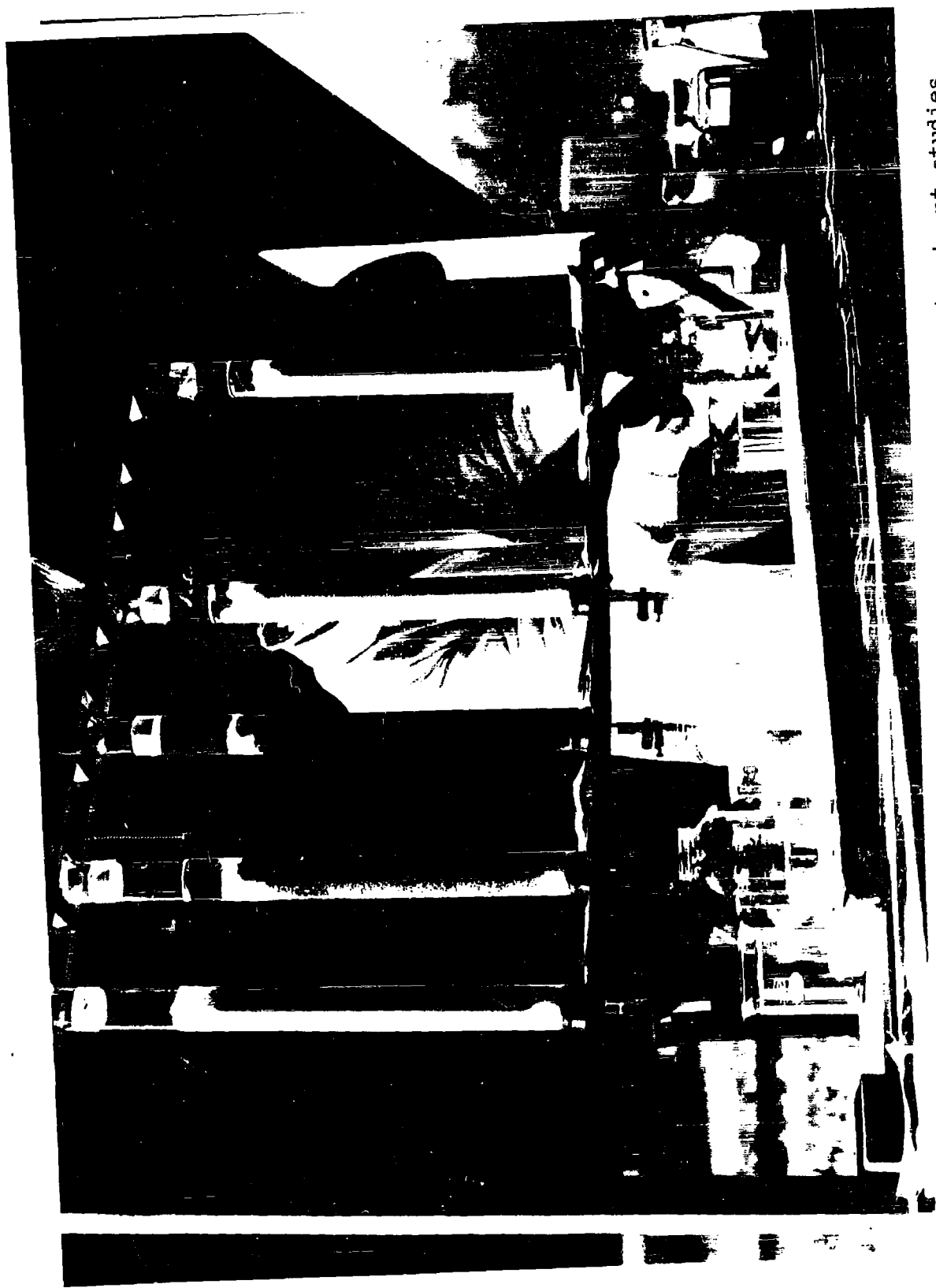


Fig. 15. Arrangement of carbon and resin columns for distillate treatment studies.



Fig. 16. Setup for small-scale batch decontamination study.

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Table IV. Summary of Results of Distillation Study

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Table No. Appendix A	Run No.	Type of Water	Agent	Feedwater Agent concentration (mg/l)	Distillate Agent concentration (mg/l)	Average Reduction (%)	MPC* (mg/l)	Posttreatment to Meet MPC
VI	1	Fresh	-	-	-	-	-	-
VII	2	Saline	BZ	11.0	0.023	99.8	0.010	Carbon or Resin
VIII	3	Fresh	BZ	16.5	0.036	99.8	0.010	" "
IX	4	Fresh	VX	16.4	1.25	92.4	0.005	Carbon or Mixed-Bed Resin
X	5	Saline	VX	19.3	1.34	92.9	0.005	" "
XI	14	Saline	VX	16.4	1.26	92.3	0.005	" "
XII	15	Saline	VX	19.8	1.26	93.6	0.005	" "
XIII	16	Saline	VX	20.6	1.34	92.5	0.005	" "
XIV	18	Fresh	VX	23.4	3.6	84.6	0.005	" "
XV	19	Distillate	VX	2.3	0.09	95.0	0.005	" "
XVI	6	Fresh	GB	15.8	0.24	93.5	0.1	" "
XVII	7	Saline	GB	5.9	0.04	99.4	0.1	None needed
XVIII	9	Saline	GB	9.4	0.1	99.9	0.1	" "
XIX	8	Fresh	GA	5.8	1.69	71.0	0.5	Carbon or Mixed-Bed Resin
XX	10	Saline	GA	7.5	0.03	99.6	0.5	None needed
XXI	17	Fresh	GA	12.8	0.16	98.8	0.5	Carbon or Mixed-Bed Resin
XXII	11	Fresh	AC	8.3	7.5	9.2	20.0	Mixed-Bed Resin
XXIII	12	Saline	AC	7.0	7.0	3.6	20.0	" "
XXIV	13	Fresh	HN-3	23.6	Trace	99.9	2.0	None needed
XXV	20	Fresh	HD	3.4	0.0	100.0	2.0	" "

* Maximum permissible concentration (ref par 3).

NOTE: Dashes indicate "not applicable."

Table V. Summary of Results of Carbon and Ion Exchange Resin Column Studies

Test No.	Agent	Composition	Feedwater		Volume Put Thru Column (liter)	Columns Used	MPC* (mg/l)	Columns Producing an Acceptable Effluent
			Agent Concentration (mg/l)					
1	BZ	Distillate	0.02 - 0.04		7.0	Nuchar WA; C-25(H)	0.01	Nuchar WA; C-25(H)
2	VX	Distillate	1.1 - 1.5		3.0	Nuchar WA; C-25(H); C-25(Ha)	0.005	Nuchar WA; C-25(H); C-25(Ha)
3	VX	Distillate and spiked distillate	1.3 - 1.5 115		64 6.4	Nuchar C-190; Mixed Resin**	0.005	Nuchar C-190; Mixed Resin**
4	VX	Spiked distillate	114		28	C-25(H); C-25(H) and A-30B(OH) in series	0.005	None
5	GB	Mixture of distillate and distillation unit feedwater	0.1 - 0.3		12.4	Nuchar WA; C-25(H); C-25(Ha); Mixed Resin**	0.1	Nuchar WA; Mixed Resin**
6	CA	Mixture of distillate and distillation unit feedwater	1.3		7.2	Nuchar WA; C-25(H) and A-30B(OH) in series; C-25(Ha); Mixed Resin**	0.5	Nuchar WA; C-25(H); A-30B(OH); Mixed Resin**
7	AC	Distillate	7.0		8.0	Nuchar WA; Mixed Resin**; Mixed Resin of C-25(H) and A-30B(OH)	20.0	Both Mixed Resin Columns
8	AC	Pond water	8.3		6.4	Nuchar WA; C-25(H) and A-30B(OH) in series; Mixed Resin**	20.0	C-25(H) and A-30B(OH) in series; Mixed Resin**
9	HN-3	Spiked distillate	5.2		8.0	Nuchar WA; Mixed Resin**	2.0	Nuchar WA; Mixed Resin**

* Maximum permissible concentration (ref par 3).

** Strongly acidic cation and strongly basic anion exchange resins.

III. DISCUSSION

7. Distillation Runs. Reference to the summary of results given in Table IV indicates less than 90-percent agent removal in only four cases. Although this high degree of reduction was obtained, only in the case of the mustards was the agent concentration in the distillate found to be below the maximum limits (paragraph 3). This indicates that the process of vapor compression distillation of water contaminated with CW agents would have to be supplemented with an auxiliary process in order to produce a potable distillate.

The details of the various runs are discussed in the order in which they are presented in the tables of Appendix B.

a. Run 1. This run was made with agent-free Shifflett Pond water to check on the distillation unit operation, sampling, analytical, and safety procedures. The results of the run indicated the unit was operating satisfactorily and that the scheduled sampling and analytical procedures could be properly performed by the test team during the run within the time periods allowed for these procedures.

b. BZ. Two runs were made, one with saline water and the other with fresh water. For the saline water run, the distillate required 2 hours before the agent concentration stabilized. In the pond water run, the agent concentration of the distillate reached a stable level immediately. Although distillation removed 99.8 percent of the agent during both runs, the agent concentration in the distillate was still twice the maximum limit specified in paragraph 3. A comparison of the graphs of the two runs (Appendix B, Figs. 17 and 18) shows little difference in the quality of the two distillates.

c. VX. Seven runs were made with VX as the contaminating agent. Four of the runs were made with saline water and two with Shifflett Pond water; one run was a redistillation of the distillate from Run 18 (Shifflett Pond water). In the six routine runs, with the agent concentration of the feedwater averaging 19.3 mg/l VX, the agent concentration in the distillate varied between 1.25 and 2.5 mg/l, which is 250 to 500 times greater than the MPC value. In the redistillation run, the VX concentration of the feedwater was 2.3 mg/l and the distillate averaged 0.09 mg/l VX. The average removal of VX accomplished in the six routine runs was 91 percent. In the redistillation run, a removal of 96 percent was obtained, indicating that the percent removal is independent of the agent concentration in the feedwater, within the range specified. No difference was apparent in the quality of the distillate in

either the saline or fresh water runs, and in all the runs, the blowdown and distillate had approximately the same agent concentration (Appendix B).

d. GB. Three runs were made with GB as the contaminating agent. The two saline water runs were $5\frac{1}{2}$ and $10\frac{2}{3}$ hours in duration, and one fresh water run was $5\frac{1}{2}$ hours in duration. In all of the runs, there was slight decomposition of the agent in the feedwater (Appendix B, Tables XVI-XVIII). In the saline water runs, the pH was such that the calcium, magnesium, and sulfates helped to accelerate decomposition of the agent. In the distillation unit evaporator, the agent went through seven half-lives in approximately 30 minutes. The high temperature and alkaline pH were the two major factors in producing this rapid decomposition. In the distillation run with saline water, the distillate was below the prescribed MPC value. The distillate from the Shifflett Pond water was above the tolerance level and required additional treatment.

e. GA. Two fresh water and one saline water runs were made with GA as the contaminant. In Run 8, Table XIX, the fresh water interfered with the Schoenemann test; consequently, all determinations were made by using the total cyanide procedure. This method of determination does not have the required accuracy or range to produce acceptable results. Therefore, the remaining two runs were evaluated by using the chemical enzyme test. Tabun (GA) had the same characteristic in the water as Sarin (GB) except that it decomposed more rapidly and produced cyanide as a hydrolysis product. The distillation unit did produce an acceptable distillate in Runs 10 and 17, made with saline and fresh water, respectively. This can be attributed partially to the high rate of decomposition of GA when exposed to high temperatures and alkaline pH.

f. AC. One fresh water run and one saline water run were made with hydrogen cyanide (AC) as the contaminating agent. Very little reduction of the AC resulted from distillation; the greatest reduction was 20 percent. The temperature and pH had little effect on decomposition.

g. HD and HN-3. One fresh water run was made with each mustard agent. The sulfur mustard (HD) and nitrogen mustard (HN-3) were readily decomposed to below the MPC by the distillation process.

8. Vapor Tests. The vapor tests were qualitative studies. The apparatus and equipment was of such a nature that any quantitative data concerning agent concentration would be unreliable. The main purpose of this study was to determine whether or not toxic vapors were released during the operation of the distillation unit.

It was found (Appendix C) that vapors of the agents were released at the blowdown sump and also at the compressor. The data are insufficient for a conclusion to be drawn as to whether these vapors represent a hazard over prolonged exposures, but the data do indicate that the wearing of protective clothing and gas masks is a sound practice.

9. Activated Carbon and Ion Exchange Resin Column Studies.

Table V indicates that activated carbon and ion exchange resins, within the limits of this study, show promise for the treatment of distillates produced by the vapor compression distillation unit. However, further studies will have to be made to determine the capacities of both carbon and resins, since in no cases were successfully operating columns loaded to exhaustion because of time limitations. In conjunction with this work, a study should be made to determine whether the action of the resins is a purely catalytic reaction or true ion exchange. In the work of Epstein, Flannery, and Wessels (6); Epstein (7); and Lindsten and Bauer (8), there is evidence that the resins also act as catalysts in the decomposition of agents GB and VX. To indicate the potential capacities of carbon or mixed resin columns, the results of the test with VX-contaminated distillate and column Nuchar C-190 and a mixed resin should be pointed out (Appendix D, Table XXXII, Test No. 3). With calculations based on a distillate feed of 1.4 mg/l and a column medium volume of 0.5 liter, the results of this test show that at least 1,100 column volumes of distillate could be successfully treated by both the carbon and mixed resin column.

a. Carbons. Nuchar WA (+30 mesh) used in the treatment of various column feedwaters containing PZ, VX, GB, GA, AC, and HN-3 produced effluents whose agent concentrations were well below acceptable levels, except in the case of AC. (It should be noted that AC can be oxidized by chlorination.) Nuchar C-190 was evaluated in one test with distillate contaminated with VX and successfully removed the agent from a spiked distillate feedwater which had been spiked to a concentration of 115 mg/l VX.

b. Resins. The results of the column studies summarized in Table V indicate that a contaminated distillate containing BZ, VX, GA, GB, AC, and HN-3 can be successfully treated with mixed resin composed of a strongly acidic cation resin and a strongly basic anion exchange resin. Separate columns of C-25(H) and A-30B(OH), in series, were somewhat less effective than the mixed resin in the removal of VX from spiked distillate. A column of C-25(H) was effective in removing BZ from a contaminated distillate. Resins C-25(H) and C-25(Na) were successful in removing VX from distillate containing 1.5 mg/l of agent. Resin C-25(Na) was ineffective in tests with distillates contaminated with GA and GB.

In an attempt to determine the fate of VX during the regeneration, with 4 N hydrochloric acid, of a Duolite C-25 (H) column which had removed 197 mg of VX from 12 liters of contaminated distillate, it was found that only 0.38 percent of the loaded amount of VX was recovered in the regenerant liquids. There are three possible answers to this low recovery: First, the destruction of agent due to hydrolysis of the VX by the resin prior to regeneration; second, hydrolysis of the agent by the strong acid used for regeneration; and third, the inability of the regenerating acid to remove completely the VX from the resin. This is an area where further study will be required.

10. Batch Decontamination Process Study. The process used in this study is a proposed, general, small-scale procedure for the removal of chemical, biological, and radiological agents from water. From various laboratory studies, it has been determined that strong hypochlorite solutions exert destructive effects on VX, G-agents, and AC. The mustards are removable by carbon adsorption. In this proposed procedure, the advantages of strong hypochlorite solution and carbon adsorption are utilized. Coagulation provides for clarification. Ion exchange resin treatment provides for the removal of soluble ionic contamination by demineralization.

The results of this study indicated that with the proposed procedure, about 35 gallons of potable water could be produced from a highly contaminated fresh water source containing 18.7 mg/l of VX, within approximately $3\frac{1}{2}$ hours.

IV. CONCLUSIONS

11. Conclusions. Based upon the results of this study, the following conclusions were made:

- a. The Army's vapor compression distillation unit does not produce an acceptable distillate from fresh water contaminated with BZ, VX, AC, GB, or GA.
- b. The Army's vapor compression distillation unit does not produce an acceptable distillate from saline water contaminated with BZ, VX, or AC.
- c. Mixed-bed ion exchange resin, containing a strongly acidic cation resin and a strongly basic anion resin, is effective for posttreatment of distillate containing all the CW agents studied.

d. Granular activated carbon is effective for post-treatment of distillate containing all the CW agents studied, except AC.

e. A small-scale batch coagulation process involving superhypo-chlorination, activated carbon de-hypo-chlorination, coagulation, and filtration is capable of effectively removing VX from a contaminated fresh water source.

f. Further study is required to determine operating characteristics of activated carbon and ion exchange resins in order to establish design criteria of auxiliary equipment for posttreatment of distillate from sources contaminated with CW agents.

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APPENDICES

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APPENDIX A

AUTHORITY

RDT & E PROJECT CARD		1. TYPE OF REPORT ☐ NEW ☐ FINAL ☐ REPLACES (No. & Date) 8-75-07-460, 31 Dec 59		REPORT CONTROL SYMBOL CSCRD-1 (R2)	
2. PROJECT TITLE Task		3. SECURITY OF PROJECT U		4. PROJECT NO. 8875-05-001	
Removal of CDR Contaminants from Water		5. TASK NO. 8875-05-001-07		6. REPORT DATE 31 March 1961	
7. BASIC FIELD OR SUBJECT Water Supply and Sanitation		8. SUB FIELD OR SUBJECT SUB GROUP Water Filtration, Purification and Distillation		9. CATEGORY SO	
10. COGNIZANT AGENCY Corps of Engineers		11a. CONTRACTOR AND/OR GOVERNMENT LABORATORY USA Engr Res & Dev Labs Ft. Belvoir, Va.		11b. CONTRACT NUMBER	
11. DIRECTING AGENCY Mil Engr Div, RMD, OCM		12. PARTICIPATION BY OTHER MILITARY DEPTS. AND OTHER GOVT. AGENCIES Chem Corps (P)(Facilities) (Technical Personnel) (Equipment) (Project No. 4-75-05-001)		13. EST. COMPLETION DATES DEV. Cont HNGR TEST. USMR TEST OPERATIONAL	
14. REQUESTING AGENCY Office, Chief of Engineers		15. DATE APPROVED 4 March 1960		16. EST. SUPPORT LEVEL ☒ UNDER \$50,000 ☐ \$50,000 - \$100,000 ☐ \$100,000 - \$250,000 ☐ \$250,000 - \$500,000 ☐ \$500,000 - \$1,000,000 ☐ OVER \$1,000,000	
17. COORDINATION ACTIONS W/ OTHER MILITARY DEPTS. & OTHER GOVT. AGENCIES Army Med Serv (C) OCDM (Funds, C)		18. PRIORITY 1-B		19. GUSSET CODE 5600	
20. CIXG par 1439c(s)		21. SPECIAL CODES			
22. REQUIREMENT AND/OR JUSTIFICATION There is a requirement for determination of the effectiveness of Army field water purification equipment and processes now standard and under development for removing chemical, biological, and radioactive agents from water to be used for drinking and other purposes. This requirement has been established by the threat of new and more efficient agents and weapons of war.					
23. BRIEF OF PROJECT AND OBJECTIVE					
a. <u>Brief</u> See Annual Resubmittal Card dated 31 December 1959.					
b. <u>Approach</u> See Annual Resubmittal Card dated 31 December 1959.					
c. <u>Tasks</u> Not applicable.					
DD FORM 613		PREVIOUS EDITIONS ARE OBSOLETE.		PAGE 1 OF 2 PAGES	

7DT & E PROJECT CARD CONTINUATION		REPORT DATE	TASK PROJECT NO.
		31 March 1961	8M75-05-001-07
d. <u>Other Information</u>			
Agencies interested in this project, in addition to the Corps of Engineers, with which liaison will be maintained and which will be furnished copies of the reports on the project are the Department of the Navy, Department of the Air Force, Chemical Corps, Army Medical Service, and CONARC.			
DD FORM 613c FEB 1960			
REPLACES DD FORM 613-1, WHICH IS OBSOLETE.			
PAGE 2 OF 2 PAGES			

APPENDIX B

REMOVAL OF CHEMICAL WARFARE AGENTS FROM
WATER BY VAPOR COMPRESSION DISTILLATION
(Tables VI through XXV)

Table VI. Run 1 - Distillation of Shifflett Pond Water
Without Agent

DATE: 26 September 1962

OBJECTIVE: To check the distillation unit operation, sampling,
analytical, and safety procedures by distillation of
agent-free water

FEEDWATER ANALYSIS: pH 7.2, turbidity 45 units, color 70 units,
hardness 40 mg/l (CaCO_3), alkalinity 20 mg/l
(CaCO_3), chloride 4 mg/l (Cl), sulfate 5 mg/l
(SO_4)

CHEMICAL DATA

Sample Time (hr)	Distillate		Blowdown		
	pH	Chloride (mg/l Cl)	pH	Chloride (mg/l Cl)	Alkalinity (mg/l CaCO_3)
1000	7.2	0.2	8.9	3.4	49
1100	6.2	0.1	9.1	6.5	56
1200	7.8	1.8	9.1	6.6	48
1300	7.9	0.25	9.1	8.8	53
1400	6.8	0.45	9.1	8.2	51
Average	7.2	0.6	9.1	6.7	51

AVERAGE OPERATIONAL DATA

Length of Run: 6 hr

Rates (gph): Distillate 104, Blowdown 70, Feedwater 174

Temperatures: Feedwater 65° F, Blowdown 140° F, Distillate 72° F

Ambient Temperature: 68° F

Distillate Conductivity: Not determined

Table VII. Run 2 - Removal of BZ from Saline Water by Vapor Compression Distillation

F

DATE: 27 September 1962

FEEDWATER ANALYSIS: pH 7.7, alkalinity 60 mg/l (CaCO₃), chloride 9160 mg/l (Cl)

Sample Time (hr)	Feedwater BZ (mg/l)	Distillate		Blowdown		
		pH	Chloride (mg/l Cl)	BZ (mg/l)	pH	Chloride (mg/l Cl) Alkalinity (mg/l CaCO ₃) BZ (mg/l)
1030	11.69	-	-	0.0079	-	- - -
1045	-	-	-	0.0044	-	- - -
1100	-	7.3	24.5*	0.0077	8.4	13,200 39 -
1115	-	-	-	0.0258	-	- - -
1130	-	-	-	0.0223	-	- - -
1200	8.97	6.3	1.6	0.0219	6.0	19,200 36 2.85
1300	11.09	6.1	0.1	0.0350	8.1	20,800 60 3.19
1400	12.70	5.9	0.3	0.0341	8.1	20,500 54 3.00
1500	10.77	6.3	0.2	0.0359	8.0	20,400 62 2.14
1600	10.86	5.7	0.9	0.0359	8.2	20,000 73 2.71
Average	11.0	6.3	0.6	0.0231	8.1	19,000 54 2.78

AVERAGE OPERATIONAL DATA

Length of Run: 5.5 hr
 Rates (gph): Distillate 108, Blowdown 60, Feedwater 168
 Temperatures: Feedwater 62° F, Blowdown 142° F, Distillate 75° F
 Distillate Conductivity: 0.222 micromho/cm
 Ambient Temperature: 60° F
 Percent Removal of BZ: Distillate 99.8, Blowdown 74.7

* Not included in calculation of average.

NOTE: Dashes indicate no determinations were made.

Plot of data of Run 2 is shown on Fig. 17.

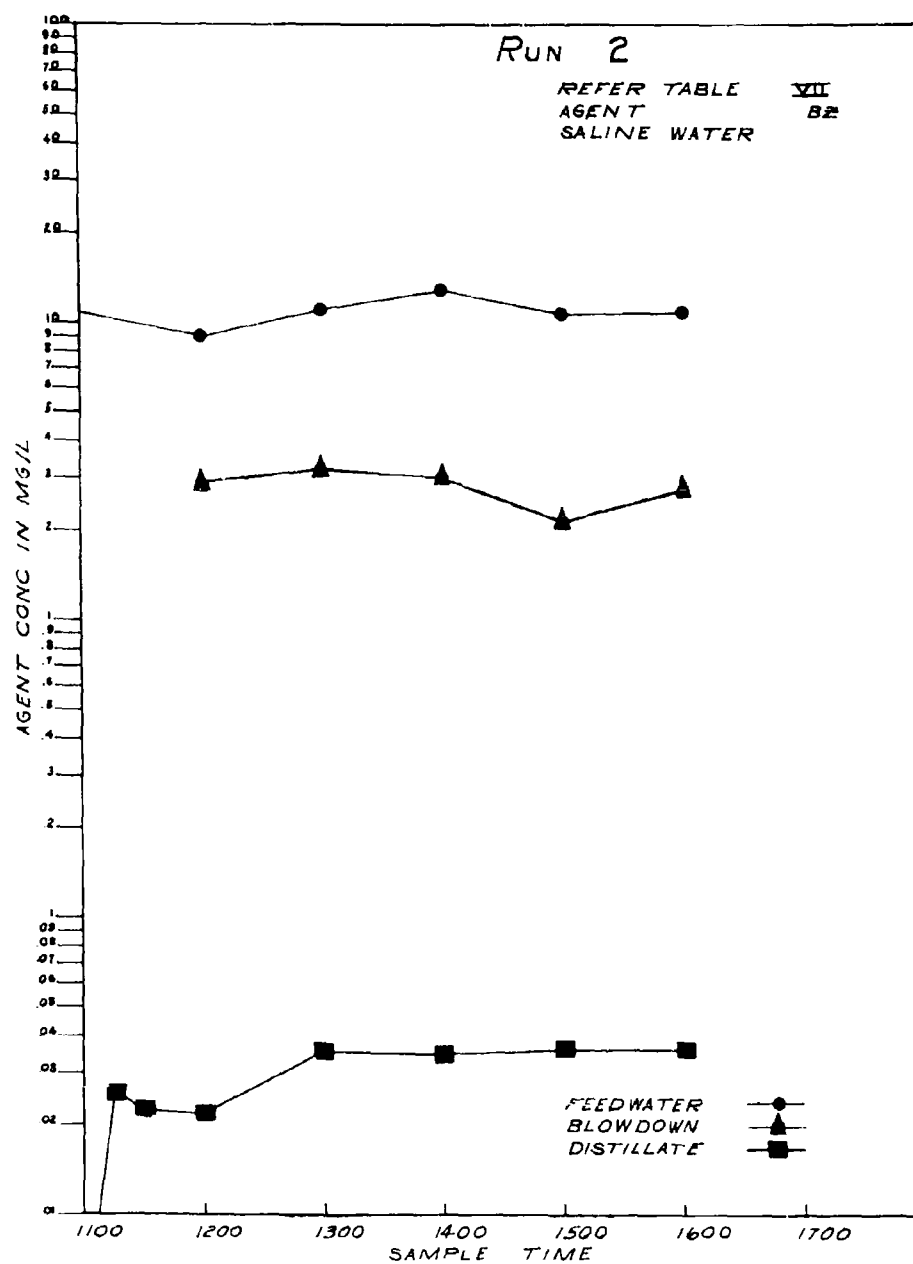


Fig. 17. Plot of data of Run 2.

Table VIII. Run 3 - Removal of BZ from Shirlett Ford Water by Vapor Compression Distillation

DATE: 1 October 1962

FEEDWATER ANALYSIS: pH 6.7, turbidity 46 units, color 70 units, hardness 47 mg/l (CaCO_3), alkalinity 19 mg/l (CaCO_3), chloride +8 mg/l (Cl), sulfate 11 mg/l (SO_4)

Sample Time (hr)	Feedwater BZ (mg/l)	Distillate		Blowdown		
		pH	Chloride (mg/l Cl)	BZ (mg/l)	pH	Chloride (mg/l Cl) Alkalinity (mg/l CaCO_3) BZ (mg/l)
1245	16.39	-	-	0.040	8.6	383 33 4.53
1300	16.73	7.2	0.5	0.033	-	- - -
1315	-	-	-	0.034	-	- - -
1330	-	-	-	0.036	-	- - -
1345	-	-	-	0.032	-	- - -
1400	16.72	5.8	0.5	0.039	8.2	125 26 3.89
1500	15.83	5.6	1.0	0.034	8.6	68 44 3.42
1600	16.73	5.7	0.4	0.039	8.4	78 51 3.88
1700	16.37	-	0.2	0.031	-	43 47 4.24
Average	16.46	5.9	0.5	0.036	8.4	139 40 3.99

AVERAGE OPERATIONAL DATA

Length of Run: 4.25 hr
 Rates (gph): Distillate 108, Blowdown 60, Feedwater 163
 Temperatures: Feedwater 66° F, Blowdown 145° F, Distillate 73° F
 Distillate Conductivity: 0.125 micromho/cm
 Ambient Temperature: 74° F
 Percent Removal of BZ: Distillate 99.8, Blowdown 75.8

NOTE: Three samples of distillate, unchlorinated and chlorinated to a residual of

1.4 mg/l Cl_2 , gave no taste or odor.
 Dashes indicate no determinations were made.
 Plot of data of Run 3 is shown on Fig. 18.

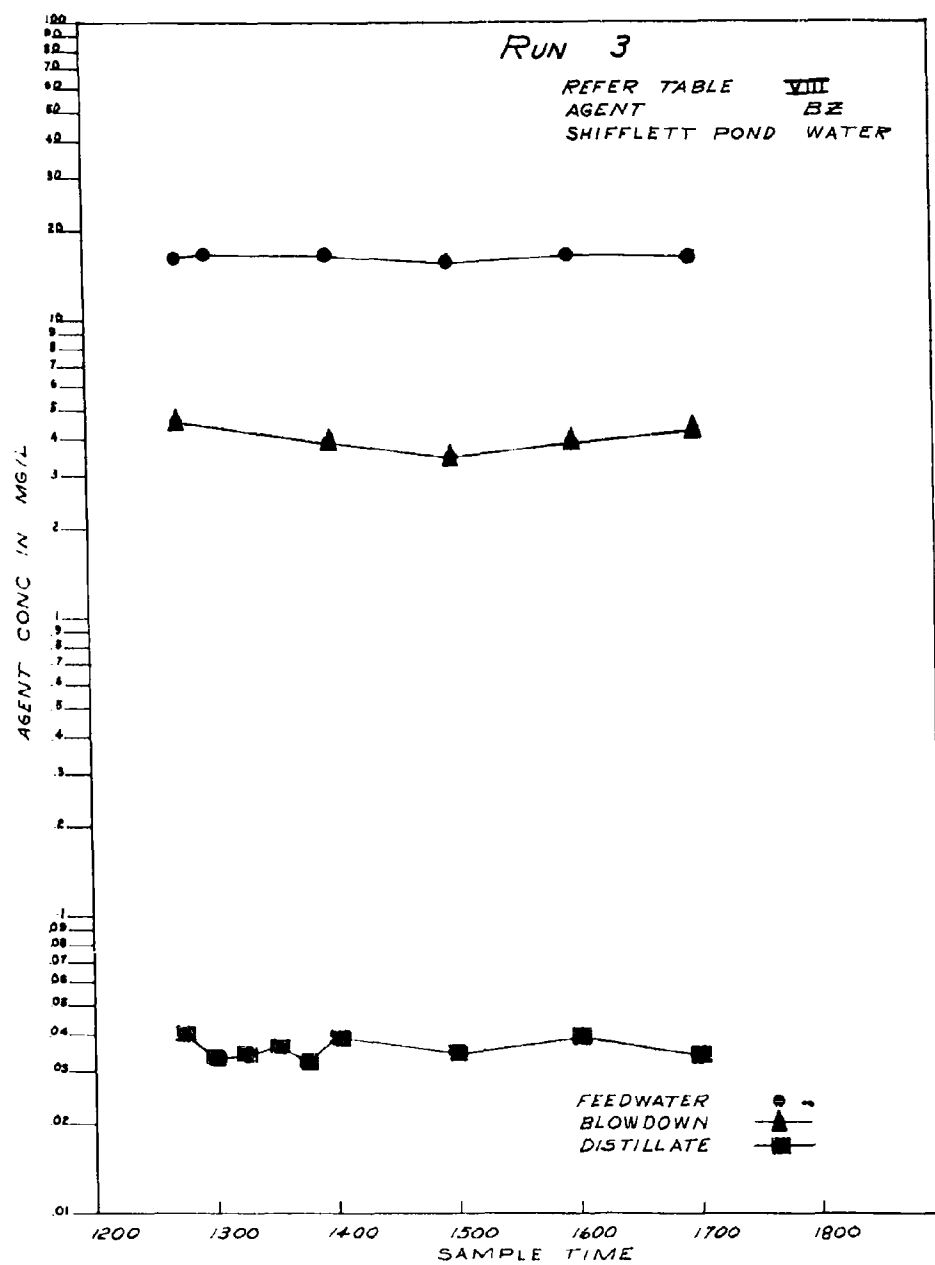


Fig. 18. Plot of data of Run 3.

Table IX. Run 4 - Removal of VX from Shirllett Pond Water by Vapor Compression Distillation

DATE: 3 October 1962

FEEDWATER ANALYSIS: pH 6.7, turbidity 120 units, color 70 units, hardness 21 mg/l (CaCO_3), alkalinity 29 mg/l (CaCO_3), chloride 23 mg/l (Cl)

CHEMICAL DATA

Sample Time (hr)	Feedwater		Distillate		Blowdown	
	pH	VX (mg/l)	pH	Chloride (mg/l Cl)	VX (mg/l)	Alkalinity (mg/l CaCO_3)
1100	6.65	15.67	6.7	1.0	1.12	5.3
1200	6.6	14.33	6.9	0.2	1.40	3.2
1300	6.8	17.72	7.0	0.5	1.52	3.5
1400	-	-	6.6	0.2	1.24	8.3
1500	6.7	15.00	6.8	0.5	1.02	8.2
1600	6.6	18.35	6.4	36.2*	1.22	8.3
Average	6.7	16.37	6.7	0.5	1.25	8.3

AVERAGE OPERATIONAL DATA

Length of Run: 5.2 hr
 Rates (gph): Distillate 109, Blowdown 105, Feedwater 21
 Temperatures: Feedwater 68° F, Blowdown 147° F, Distillate 91° F
 Distillate Conductivity: 0.435 micromho/cm
 Ambient Temperature: 72° F
 Percent Removal of VX: Distillate 92.4, Blowdown 91.2

* Not included in calculation of average.

NOTE: Dashes indicate no determinations were made.
 Plot of data of Run 4 is shown on Fig. 19.

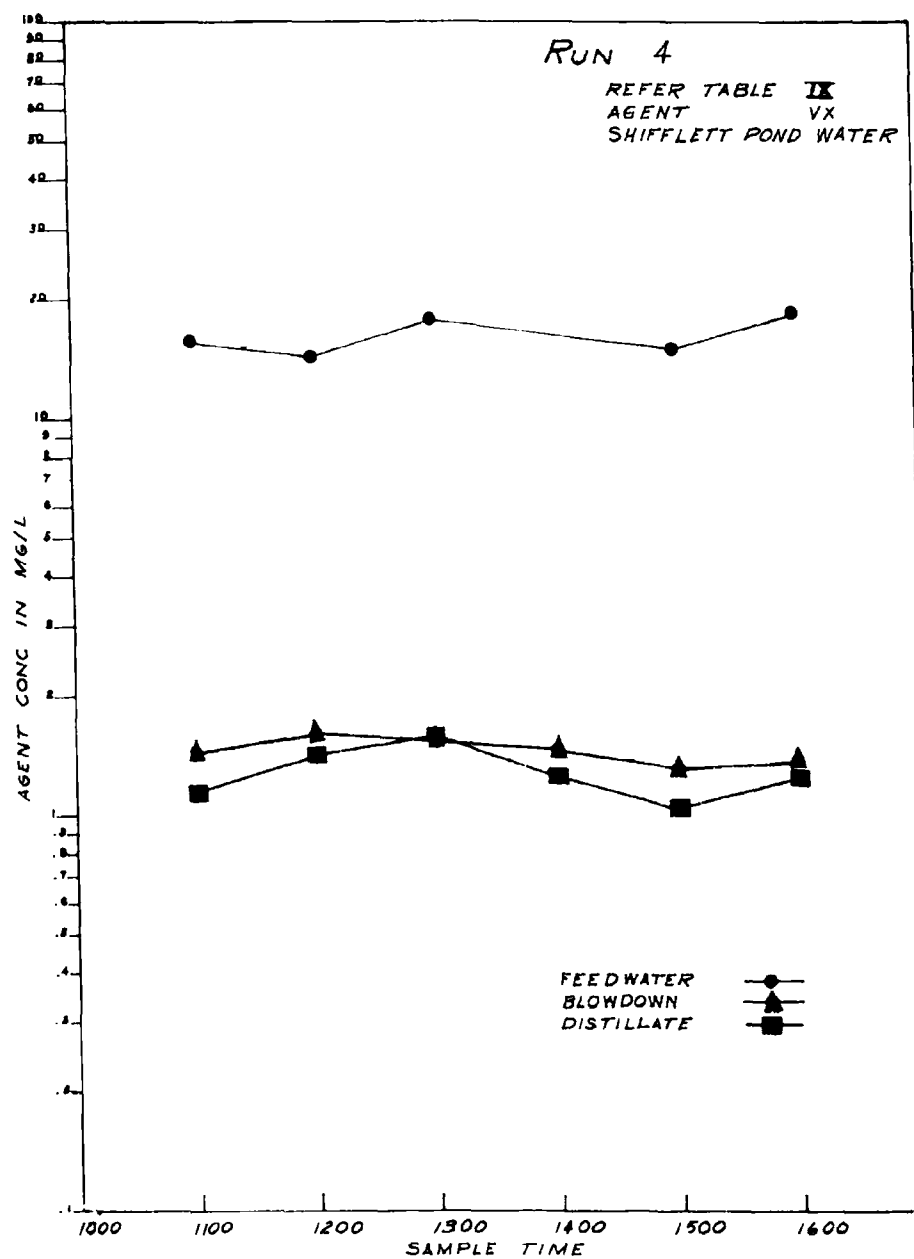


Fig. 19. Plot of data of Run 4.

Table X. Run 5 - Removal of VX from Saline Water by Vapor Compression Distillation

DATE: 8 October 1962

FEEDWATER ANALYSIS: pH 8.2, turbidity 5 units, color 30 units, alkalinity 91 mg/l (CaCO₃),
chloride 5300 mg/l (Cl⁻)

CHEMICAL DATA

Sample Time (hr)	Feedwater		Distillate		pH	Blowdown	
	pH	VX (mg/l)	Chloride (mg/l Cl)	VX (mg/l)		Chloride (mg/l Cl)	Alkalinity (mg/l CaCO ₃) VX (mg/l)
1130	8.2	16.67*	29.0*	0.43*	5.4	7,600*	47* 1.07*
1230	8.2	18.60	0.3	1.32	5.3	13,600	63 1.50
1330	8.2	20.25	1.2	1.05	5.7	15,800	66 1.06
1430	8.0	19.11	0.5	1.35	5.3	17,900	86 1.33
1530	-	19.25	0.5	1.83	-	17,000	117 1.79
1630	-	19.33	0.5	1.16	-	17,200	87 1.39
Average	8.2	19.31	0.6	1.34	5.6	16,300	84 1.41

AVERAGE OPERATIONAL DATA

Length of Run: 5 hr
 Rates (gph): Distillate 101, Blowdown 93, Feedwater 124
 Temperatures: Feedwater 69° F, Blowdown 144° F, Distillate 30° F
 Distillate Conductivity: 0.435 micromho/cm
 Ambient Temperature: 72° F
 Percent Removal of VX: Distillate 92.9, Blowdown 92.5

* Not included in calculation of average.

NOTE: Dashes indicate no determinations were made.

Plot of data of Run 5 is shown on Fig. 20.

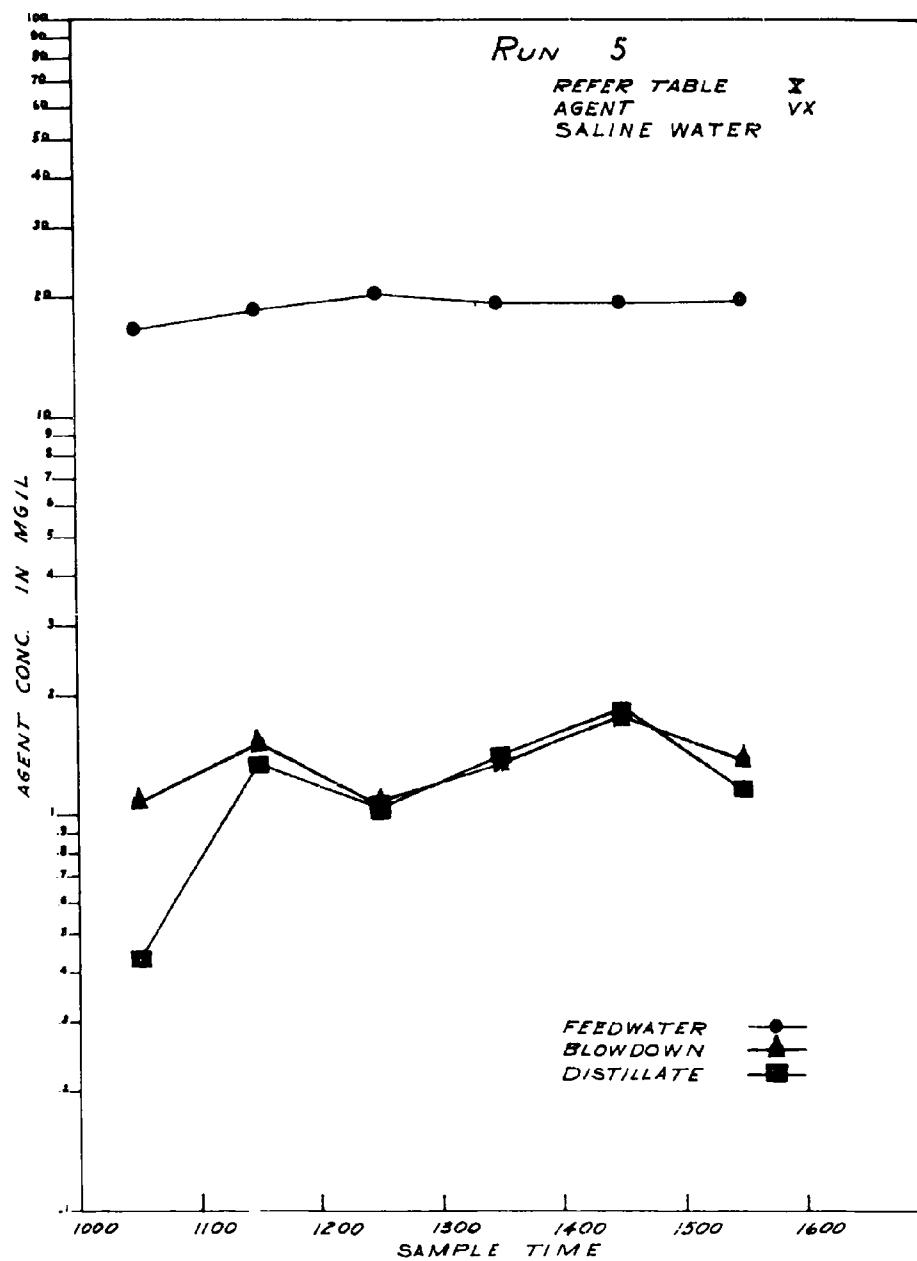


Fig. 20. Plot of data of Run 5.

Table XI. Run 14 - Removal of VX from Saline Water by Vapor Compression Distillation

DATE: 2 November 1962

FEEDWATER ANALYSIS: pH 7.7, turbidity 5 units, color 15 units, alkalinity 59 mg/l (CaCO_3),
chloride 7240 mg/l (Cl)

CHEMICAL DATA						
Sample Time (hr)	Feedwater		Distillate		Blowdown	
	pH	VX (mg/l)	pH	Chloride (mg/l Cl)	VX (mg/l)	VX (mg/l)
0300	5.2	14.91	7.1	4.0	1.36	1.06
0900	8.0	17.90	6.3	1.0	1.28	1.06
1000	7.1	19.16	6.6	0.9	1.12	0.97
1100	7.6	13.58	7.2	1.1	1.30	1.14
1200	7.5	14.83	7.2	1.0	1.26	0.92
1300	7.7	18.08	7.6	1.1	1.22	1.24
Average	7.7	16.41	7.0	1.5	1.26	1.06

AVERAGE OPERATIONAL DATA

Length of Run: 5.3 hr
 Rates (gph): Distillate 104, Blowdown 58, Feedwater 162
 Temperatures: Feedwater 48° F, Blowdown 129° F, Distillate 67° F
 Distillate Conductivity: 0.435 micromho/cm
 Ambient Temperature: 47° F
 Percent Removal of VX: Distillate 92.3, Blowdown 93.5

Table XII. Run 15 - Removal of VX from Saline Water by Vapor Compression Distillation

DATE: 5 November 1962

FEEDWATER ANALYSIS: pH 7.8, color 35 units, alkalinity 63 mg/l (CaCO_3), chloride 6550 mg/l (Cl)

CHEMICAL DATA

Sample Time (hr)	Feedwater		Distillate		Blowdown	
	pH	VX (mg/l)	pH	Chloride (mg/l Cl)	VX (mg/l)	Alkalinity (mg/l CaCO_3)
1200	7.9	20.25	6.8	3.6	0.56*	7.3
1300	7.9	21.08	6.9	6.2	1.36	8.6
1400	7.8	21.83	7.0	1.4	1.42	8.6
1500	7.9	18.66	7.4	1.0	1.45	8.4
1600	7.6	17.87	7.0	0.9	1.26	8.6
1700	7.6	19.04	6.8	1.0	1.51	9.0
Average	7.8	19.79	7.0	2.4	1.26	8.4
					16,100	62

AVERAGE OPERATIONAL DATA

Length of Run: 5 hr
 Rates (gph): Distillate 101, Blowdown 54, Feedwater 155
 Temperatures: Feedwater 45° F, Blowdown 131° F, Distillate 65° F
 Distillate Conductivity: 0.555 micromho/cm
 Ambient Temperature: 43° F
 Percent Removal of VX: Distillate 93.6, Blowdown 91.7

* Not included in calculation of average.

DATE: 7 November 1962

DATE: 1 NOVEMBER 1958

FEEDWATER ANALYSIS: pH 7.2, color 20 units, turbidity 75 units, alkalinity 59 mg/l (CaCO_3), chloride 7100 mg/l (Cl)

CHEMICAL DATA						
Sample Time (hr)	Feedwater		Distillate		Blowdown	
	pH	VX (mg/l)	pH	Chloride (mg/l Cl)	VX (mg/l)	Alkalinity (mg/l CaCO ₃)
1130	6.9	19.1	7.2	0.5	0.31	29*
1230	7.3	18.65	7.0	1.5	1.17	95
1330	7.3	23.53	7.1	1.5	1.50	106
1430	7.0	20.67	6.9	1.0	2.05	117
1530	7.2	21.70	6.7	3.5	2.12	113
1630	7.1	20.53	7.0	1.0	1.50	112
Average	7.2	20.65	7.0	1.2	1.51	109

Length of Run: 5 hr

Length of Run: 5 hr

<u>Rates (gph):</u>	Distillate 101, Blowdown 49, Feedwater 150
<u>Temperatures:</u>	Feedwater 42° F, Blowdown 130° F, Distillate 62° F

Temperatures: Feedwater 42° F, Blowdown 130° F, Distillate 62° F

Distillate Conductivity: 0.715 microhm/cm

Distillate concn: 52°

Ambient Temperature: 72.0
Percent Removal of VX: Distillate 92.5, Blowdown 92.0

* Not included in calculation of average.

Table XIV. Run 18 - Removal of VX from Shifflett Pond Water by Vapor Compression Distillation

DATE: 13 November 1962

FEEDWATER ANALYSIS: pH 8.1, turbidity 65 units, color 60 units, hardness 22 mg/l (CaCO_3), alkalinity 20 mg/l (CaCO_3), chloride 16 mg/l (Cl), sulfate 35 mg/l (SO_4)

CHEMICAL DATA

Sample Time (hr)	Feedwater		Distillate		pH	Blowdown			
	pH	VX (mg/l)	Chloride (mg/l Cl)	VX (mg/l)		Chloride (mg/l Cl)	Alkalinity (mg/l CaCO ₃)	VX (mg/l)	
1100	9.0	23.9	7.8	0.8	7.8	1.5	7.1	20	2.2
1200	8.1	25.2	7.8	0.5	7.8	1.6	32.0	42	2.4
1300	8.2	25.5	8.4	1.0	8.4	2.8	3.6	28	5.2
1400	8.1	18.9	7.6	0.6	7.6	11.4	7.1	28	4.0
1500	7.8	23.7	7.1	1.0	7.1	2.6	52.3	66	3.0
1600	8.1	23.0	7.8	1.0	7.8	1.5	1.7	68	2.8
Average	8.1	23.4	7.8	0.9	7.8	3.6	17.3	42	3.3

AVERAGE OPERATIONAL DATA

Length of Run: 7 hr
 Rates (gph): Distillate 101, Blowdown 49, Feedwater 150
 Temperatures: Feedwater 48° F, Blowdown 131° F, Distillate 71° F
 Distillate Conductivity: 0.417 micromho/cm
 Ambient Temperature: 46° F
 Percent Removal of VX: Distillate 84.6, Blowdown: 85.9

Table XV. Run 19 - Removal of VX from Distillate of Run 18 by Vapor Compression Distillation

DATE: 14 November 1962

FEEDWATER ANALYSIS: pH 7.1, color 2 units, turbidity 8 units, sulfate 3 mg/l (SO_4), chloride 2 mg/l (Cl), alkalinity 8 mg/l (CaCO_3).

CHEMICAL DATA

Sample Time (hr)	Feedwater		Distillate		Blowdown	
	pH	VX (mg/l)	pH	Chloride (mg/l Cl)	VX (mg/l)	Alkalinity (mg/l CaCO_3)
1000	7.1	2.46	-	-	-	-
1030	-	-	8.0	0.5	0.08	16
1130	-	-	8.0	0.8	0.09	11
1230	-	-	8.5	1.2	0.03	10
1330	-	-	8.4	3.2	0.09	11
1430	-	2.08	8.5	0.5	0.16	16
Average	7.1	2.27	8.3	1.2	0.09	13

AVERAGE OPERATIONAL DATA

Length of Run: 4.25 hr
 Rates (gph): Distillate 101, Blowdown 48, Feedwater 149
 Temperatures: Feedwater 55° F, Blowdown 138° F, Distillate 74° F
 Distillate Conductivity: 0.476 micromho/cm
 Ambient Temperature: 55° F
 Percent Removal of VX: Distillate 96.0, Blowdown 88.1

NOTE: Dashes indicate no determinations were made.

Table XVI. Run 6 - Removal of GB from Shifflett Pond Water by Vapor Compression Distillation

DATE: 10 October 1962

FEEDWATER ANALYSIS: pH 7.1, turbidity 14 units, color 60 units, hardness 30 mg/l (CaCO₃), alkalinity 22 mg/l (CaCO₃), chloride 20 mg/l (Cl), sulfate 27 mg/l (SO₄)

CHEMICAL DATA

Sample Time (hr)	Feedwater		Distillate		Blowdown	
	pH	GB (mg/l)	pH	Chloride (mg/l Cl)	GB (mg/l)	Alkalinity (mg/l CaCO ₃)
1100	7.3	17.78	7.0	0.25	0.125	32
1200	7.3	16.16	6.0	0.70	0.168	27
1300	7.1	15.25	6.3	0.25	0.201	40
1400	7.4	15.96	6.3	0.25	0.177	46
1500	7.8	14.89	6.6	0.75	0.429	48
1600	7.5	14.96	6.9	0.25	0.356	43
Average	7.4	15.83	6.5	0.40	0.243	39

AVERAGE OPERATIONAL DATA

Length of Run: 5.5 hr
 Rates (gph): Distillate 114, Blowdown 86, Feedwater 200
 Temperatures: Feedwater 66° F, Blowdown 139° F, Distillate 79° F
 Distillate Conductivity: 0.111 micromho/cm
 Ambient Temperature: 67° F
 Percent Removal of GB: Distillate 98.5, Blowdown 99.0

* Not included in calculation of average.

NOTE: Four distillate samples (1100 through 1400 hr), unchlorinated or chlorinated to 2 mg/l Cl₂ residual, gave no taste or odor.
 Plot of data of Run 6 is shown on Fig. 21.

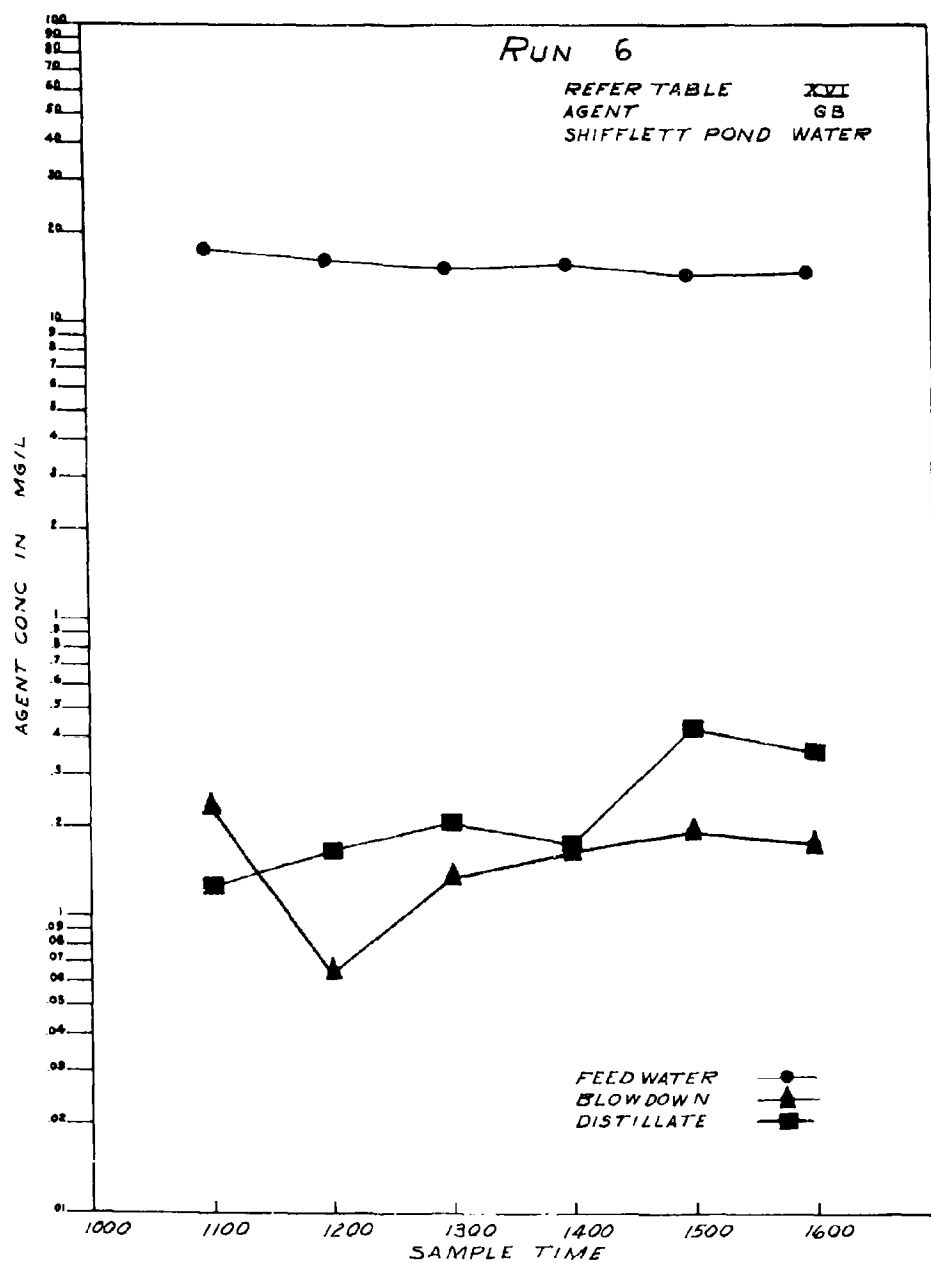


Fig. 21. Plot of data of Run 6.

Table XVII. Run 7 - Removal of GB from Saline Water by Vapor Compression Distillation

DATE: 12 October 1962

FEEDWATER ANALYSIS: pH 7.4, turbidity 1 unit, color 20 units, alkalinity 62 mg/l (CaCO_3),
chloride 7050 mg/l (Cl)

CHEMICAL DATA

Sample Time (hr)	Feedwater		Distillate		Blowdown		
	pH	GB (mg/l)	pH	Chloride (mg/l Cl)	GB (mg/l)	pH	Chloride (mg/l Cl) Alkalinity (mg/l CaCO_3) GB (mg/l)
1100	7.3	9.14	6.1	1.4	0.094	8.7	12,100 52 0.199
1200	7.3	6.65	5.9	0.75	0.033	8.4	16,300 65 0.186
1300	7.1	-	6.4	0.50	0.020	8.5	17,100 70 0.186
1400	7.4	4.92	6.4	1.6	0.0	8.6	17,400 66 0.124
1500	7.8	4.78	6.4	0.25	0.049	8.9	17,200 71 0.31
1600	7.5	4.06	6.4	0.50	0.045	8.8	16,100 85 0.65
Average	7.4	5.91	6.3	0.84	0.04	8.6	16,000 68 0.28

AVERAGE OPERATIONAL DATA

Length of Run: 5.5 hr
 Rates (gph): Distillate 108, Blowdown 96, Feedwater 204
 Temperatures: Feedwater 71° F, Blowdown 136° F, Distillate 84° F
 Distillate Conductivity: 0.166 micromho/cm
 Ambient Temperature: 87° F
 Percent Removal of GB: Distillate 99.5, Blowdown 95.3

NOTE: Dash indicates no determination was made.
 Plot of data of Run 7 is shown on Fig. 22.

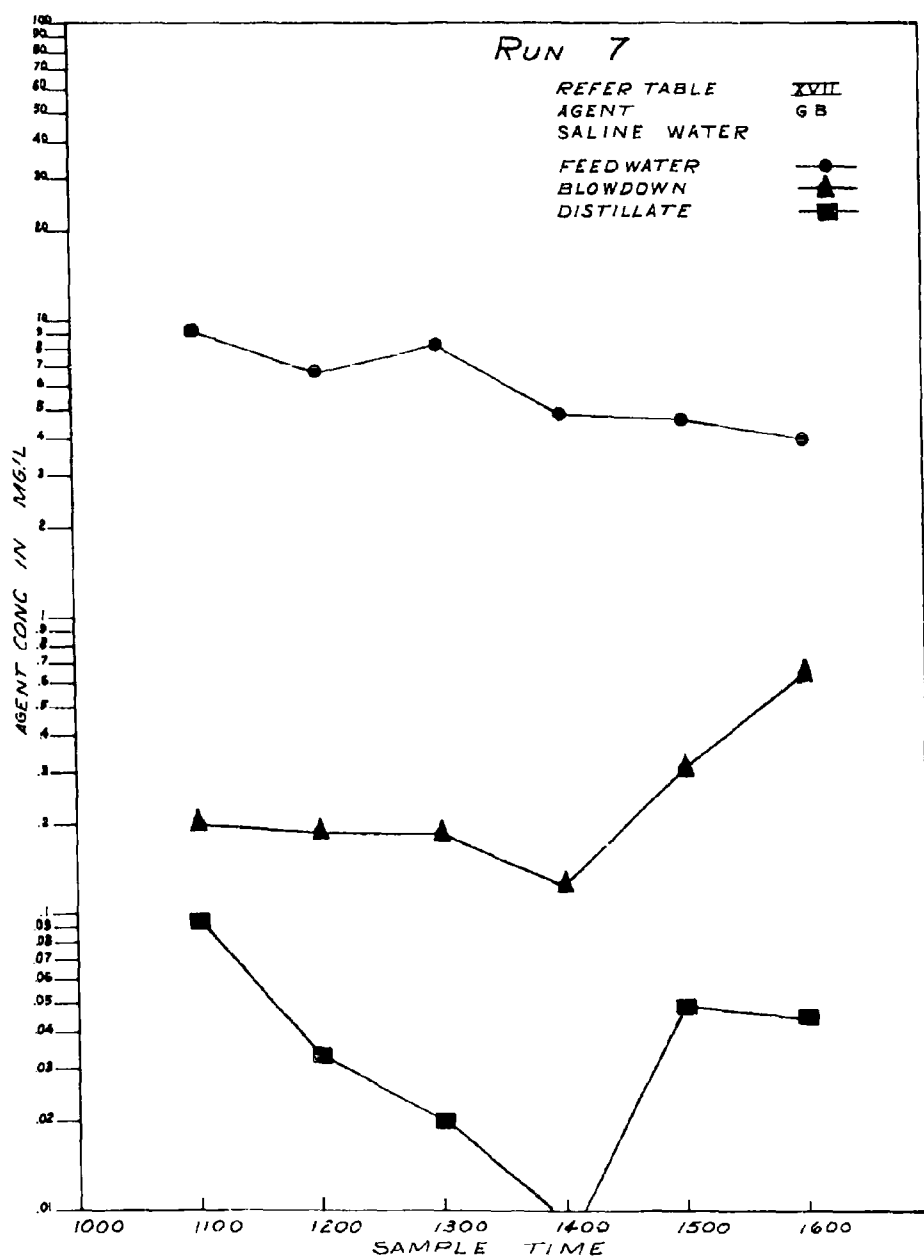


Fig. 22. Plot of data of Run 7.

Table XVIII. Run 9 - Removal of GB from Saline Water by Vapor Compression Distillation

DATE: 18 October 1962									
FEEDWATER ANALYSIS: pH 7.2, turbidity 2 units, color 5 units, hardness 2200 mg/l (CaCO ₃), alkalinity 58 mg/l (CaCO ₃), chloride 6620 mg/l (Cl)									
CHEMICAL DATA									
Sample Time (hr)	Feedwater		Distillate		Blowdown				
	pH	GB (mg/l)	pH	Chloride (mg/l Cl)	GB (mg/l)	pH	Chloride (mg/l Cl)	Alkalinity (mg/l CaCO ₃)	GB (mg/l)
0900	7.3	13.02	6.2	1.0	0	6.8	1,755*	58	0
1000	7.5	12.36	5.8	0.5	0.1	8.8	19,150	55	Trace
1100	7.3	11.78	6.4	0.5	0.1	8.6	20,500	71	Trace
1200	7.2	10.85	6.5	0.2	0.1	8.3	20,400	67	Trace
1300	7.2	9.88	5.8	0.2	0.1	8.2	17,900	83	Trace
1400	7.2	9.80	6.0	0.5	0.17	8.7	17,700	68	0.80
1500	7.2	9.35	5.7	0.5	0.1	8.4	19,000	66	Trace
1600	7.2	8.41	6.4	1.4	0	8.6	19,500	66	Trace
1700	-	-	-	-	-	-	-	-	-
1800	7.2	6.45	6.2	1.0	0.1	8.8	18,000	66	Trace
1900	7.0	6.72	6.2	0.6	0.1	8.2	18,200	66	Trace
2000	6.5	5.65	5.3	2.0	0.1	8.0	19,400	56	Trace
Average	7.2	9.39	6.1	0.8	0.1	8.3	19,000	66	Trace
AVERAGE OPERATIONAL DATA									

Length of Run: 10.7 hr
 Rates (gph): Distillate 108, Blowdown 91, Feedwater 199
 Temperatures: Feedwater 67° F, Blowdown 145° F, Distillate 78° F
 Distillate Conductivity: 0.200 micromho/cm
 Ambient Temperature: 62° F
 Percent Removal of GB: Distillate 99, Blowdown 99

* Not included in calculation of average.

NOTE: The 0900 hr sample of distillate, unchlorinated and chlorinated to a 2.0 mg/l Cl₂ residual, gave no taste or odor.

Dashes indicate no determinations were made (unit inoperative between 1635 hr and 1705 hr).

DATE: 16 October 1962

DATE: 16 October 1962

FEEDWATER ANALYSIS: pH 7.1, turbidity 25 units, color 50 units, hardness 32 mg/l (CaCO₃), alkalinity 27 mg/l (CaCO₃), chloride 9 mg/l (Cl), sulfate 18 mg/l (SO₄)

CHEMICAL DATA							
Sample Time (hr)	Feedwater		Distillate		Blowdown		
	pH	GA (mg/l)	pH	Chloride (mg/l Cl)	GA (mg/l)	pH	Chloride (mg/l Cl)
					Alkalinity (mg/l CaCO ₃)		GA (mg/l)
0930	7.0	9.59	-	-	-	-	-
1100	7.0	7.37	3.7	2.0	5.35	3.1	320
1200	6.9	6.59	4.3	2.2	1.12	7.7	226
1300	7.3	4.91	4.6	2.2	0.14	7.9	94
1400	7.2	4.23	6.0	2.8	-	8.2	70
1500	7.6	4.02	5.1	2.5	-	8.6	57
1600	6.9	4.04	5.5	2.2	0.14	8.7	97
Average	7.1	5.82	4.9	2.4	1.69	8.2	144
AVERAGE OPERATIONAL DATA							

Length of Run: 5.5 hr
Rates (gph): Distillate 108, Blowdown 81, Feedwater 139
Temperatures: Feedwater 66° F, Blowdown 158° F, Distillate 36° F
Distillate Conductivity: 0.286 micromho/cm
Ambient Temperature: 78° F
Percent Removal of GA: Distillate 71.0

* (Cl + CN) as mg/l Cl.

NOTE: Agent concentration data of this run not of required in the analytical procedures by the pond water.

Dashes indicate no determinations were made.

Table XX. Run 10 - Removal of GA from Saline Water by Vapor Compression Distillation

DATE: 23 October 1962

FEEDWATER ANALYSIS: pH 7.1, turbidity 7 units, color 5 units, hardness 2300 mg/l (CaCO_3), alkalinity 54 mg/l (CaCO_3), chloride 67.0 mg/l (Cl)

CHEMICAL DATA

Sample Time (hr)	Feedwater		Distillate			Blowdown		
	pH	GA (mg/l)	pH	Chloride* (mg/l Cl)	GA (mg/l)	Chloride* (mg/l Cl)	Alkalinity (mg/l CaCO_3)	GA (mg/l)
0910	7.3	13.4	-	-	-	-	-	-
1100	7.4	8.14	6.1	2.3	0.022	1.32	17,750	0.03
1200	7.3	7.93	6.3	2.8	0.032	1.47	17,750	0.0062
1300	7.4	6.90	6.3	2.6	0.031	1.80	18,350	0.008
1400	7.7	6.45	6.5	6.1	0.029	1.72	19,200	0.008
1500	7.3	4.72	5.4	87.5**	0.026	1.68	18,300	0.009
1600	7.4	4.78	6.6	5.3	0.021	1.53	19,200	0.009
Average	7.4	7.47	6.2	4.2	0.027	1.59	18,800	0.012

AVERAGE OPERATIONAL DATA

Length of Run: 5.7 hr
 Rates (gph): Distillate 106, Blowdown 75, Feedwater 131
 Temperatures: Feedwater 62° F, Blowdown 142° F, Distillate 77° F
 Distillate Conductivity: 0.333 micromho/cm
 Ambient Temperature: 68° F
 Percent Removal of GA: Distillate 99.6, Blowdown 99.9

* (Cl + CN) as mg/l Cl.

** Not included in calculation of average.

NOTE: Two samples of distillate (1330 hr and 1530 hr), unchlorinated and chlorinated to a 2.0 mg/l Cl_2 residual, gave no taste or odor.

Dashes indicate no determinations were made.

Plot of data of Run 10 is shown on Fig. 23.

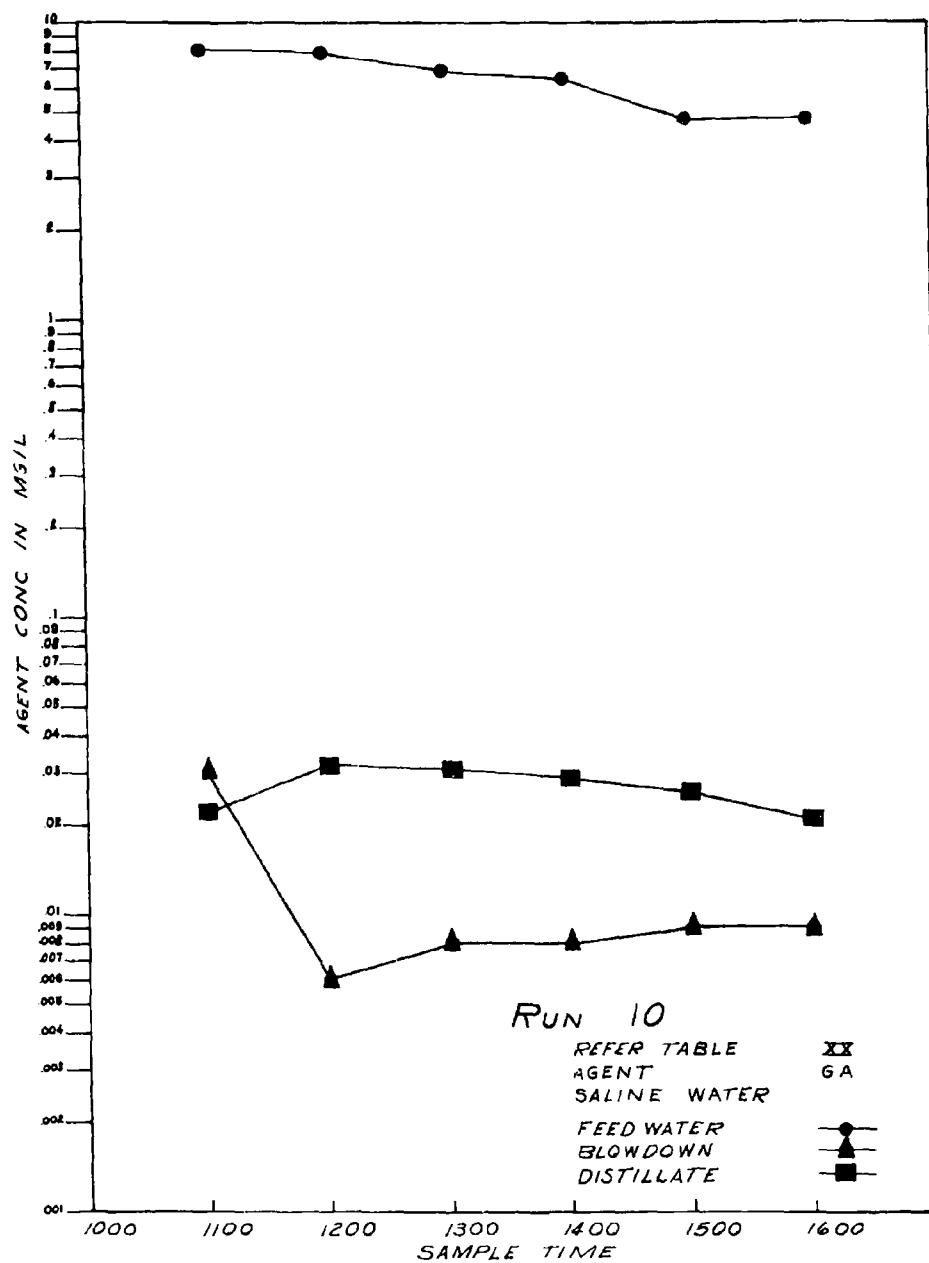


Fig. 23. Plot of data of Run 10.

Table XXI. Run 17 - Removal of GA from Shifflett Pond Water by Vapor Compression Distillation

DATE: 8 November 1962

FEEDWATER ANALYSIS: pH 6.9, turbidity 65 units, color 60 units, hardness 22 mg/l (CaCO₃), alkalinity 20 mg/l (CaCO₃), chloride 13 mg/l (Cl)

CHEMICAL DATA

Sample Time (hr)	Feedwater		Distillate			Blowdown		
	pH	GA (mg/l)	pH	Chloride* (mg/l Cl)	GA (mg/l)	pH	Chloride* (mg/l Cl)	Alkalinity (mg/l CaCO ₃) GA Cyanide (mg/l CN)
1030	6.7	14.6	6.9	3.0	0.08	7.9	212	49 0.5
1130	6.6	12.0	6.2	3.3	0.16	8.7	340	75 0.4
1230	6.8	12.8	6.0	2.8	0.24	8.6	230	61 0.2
1330	6.9	13.2	6.3	2.6	0.17	8.9	158	88 0.6
1430	7.1	10.2	6.2	2.8	0.11	8.6	120	82 0.6
1530	7.2	13.8	6.3	3.0	0.19	8.6	156	78 0.3
Average	6.9	12.8	6.3	2.9	0.16	8.6	203	72 0.4

AVERAGE OPERATIONAL DATA

Length of Run: 5.25 hr
 Rates (gph): Distillate 101, Blowdown 53, Feedwater 154
 Distillate Conductivity: 0.250 micromho/cm
 Temperatures: Feedwater 45° F, Blowdown 130° F, Distillate 71° F
 Ambient Temperature: 56° F
 Percent Removal of GA: Distillate 98.6, Blowdown 98.8

* (Cl + CN) as mg/l Cl.

NOTE: Dash indicates no determination was made.

Plot of data of Run 17 is shown on Fig. 24.

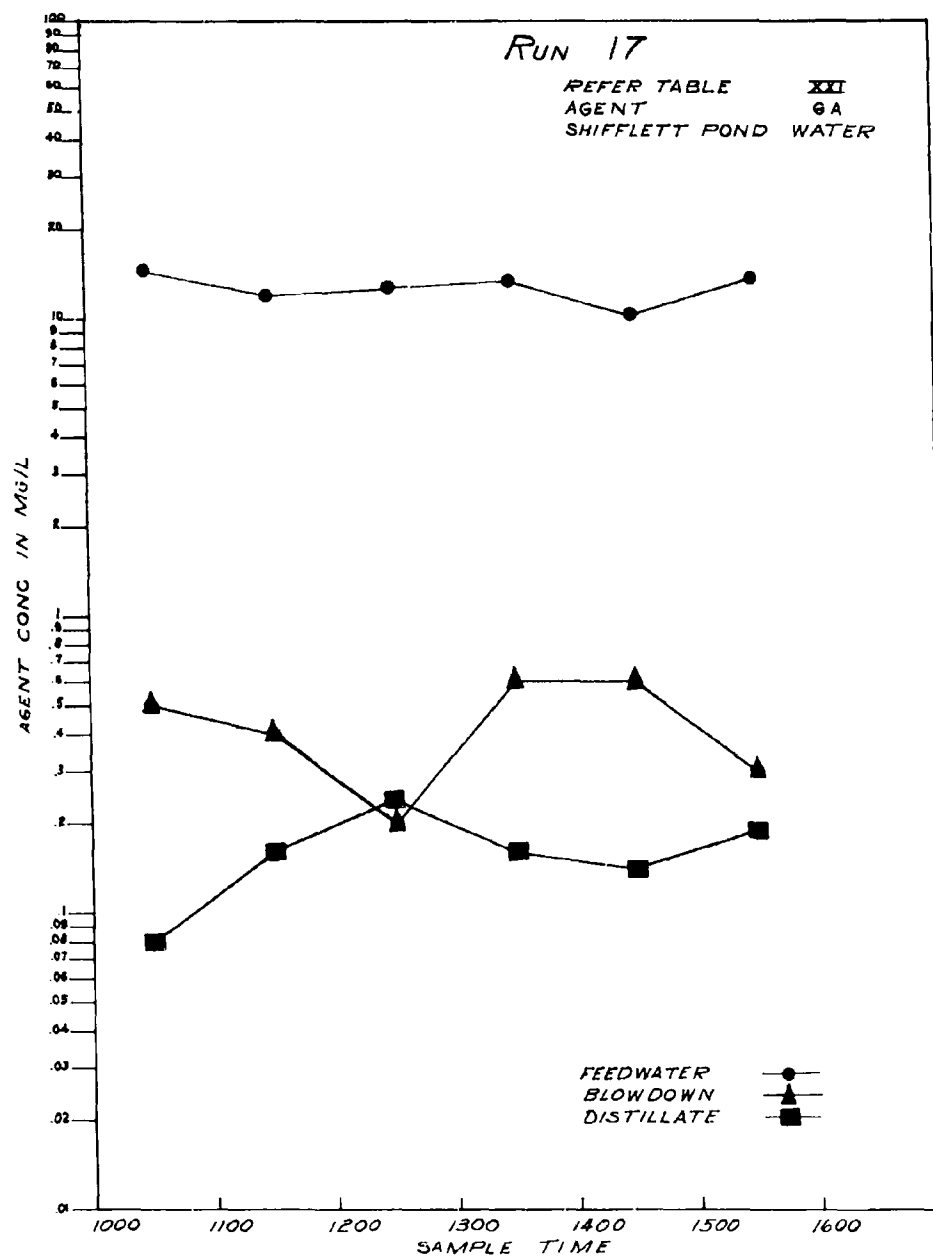


Fig. 24. Plot of data of Run 17.

Table XXII. Run 11 - Removal of AC from Shifflett Pond Water by Vapor Compression Distillation

DATE: 25 October 1962

FEEDWATER ANALYSIS: pH 6.9, turbidity 1 units, color 70 units, hardness 25 mg/l (CaCO_3), alkalinity 20 mg/l (CaCO_3), chloride 6 mg/l (Cl), sulfate 15 mg/l (SO_4)

CHEMICAL DATA							
Sample Time (hr)	Feedwater		Distillate		Blowdown		
	pH	AC (mg/l)	pH	Chloride* (mg/l Cl)	AC (mg/l)	pH	Chloride* (mg/l Cl) Alkalinity (mg/l CaCO_3) AC (mg/l)
1030	6.7	8.09	6.1	5.6	3.60	5.7	332 40 1.20
1130	6.5	8.18	6.0	10.7	7.93	5.9	213 56 1.16
1230	7.0	8.12	6.3	9.6	8.36	5.3	152 44 2.41
1330	7.0	8.57	6.4	16.2	8.83	9.2	130 47 1.27
1430	7.0	8.56	6.2	9.9	7.95	5.7	139 60 1.49
1530	7.1	7.68	6.6	10.1	6.09	5.5	113 62 2.78
Average	6.9	8.25	6.3	10.4	7.49	5.7	181 52 1.72

AVERAGE OPERATIONAL DATA

Length of Run: 5.25 hr
 Rates (gph): Distillate 101, Blowdown 50, Feedwater 151
 Temperatures: Feedwater 50° F, Blowdown 127° F, Distillate 59° F
 Distillate Conductivity: 0.333 micromho/cm
 Ambient Temperature: 41° F
 Percent Removal of AC: Distillate 9.2, Blowdown 79.2

* (Cl + CN) as mg/l Cl.

NOTE: The 1400 hr sample of distillate, unchlorinated and chlorinated to a 2.0 mg/l Cl₂ residual, gave no taste or odor.
 Plot of data of Run 11 is shown on Fig. 25.

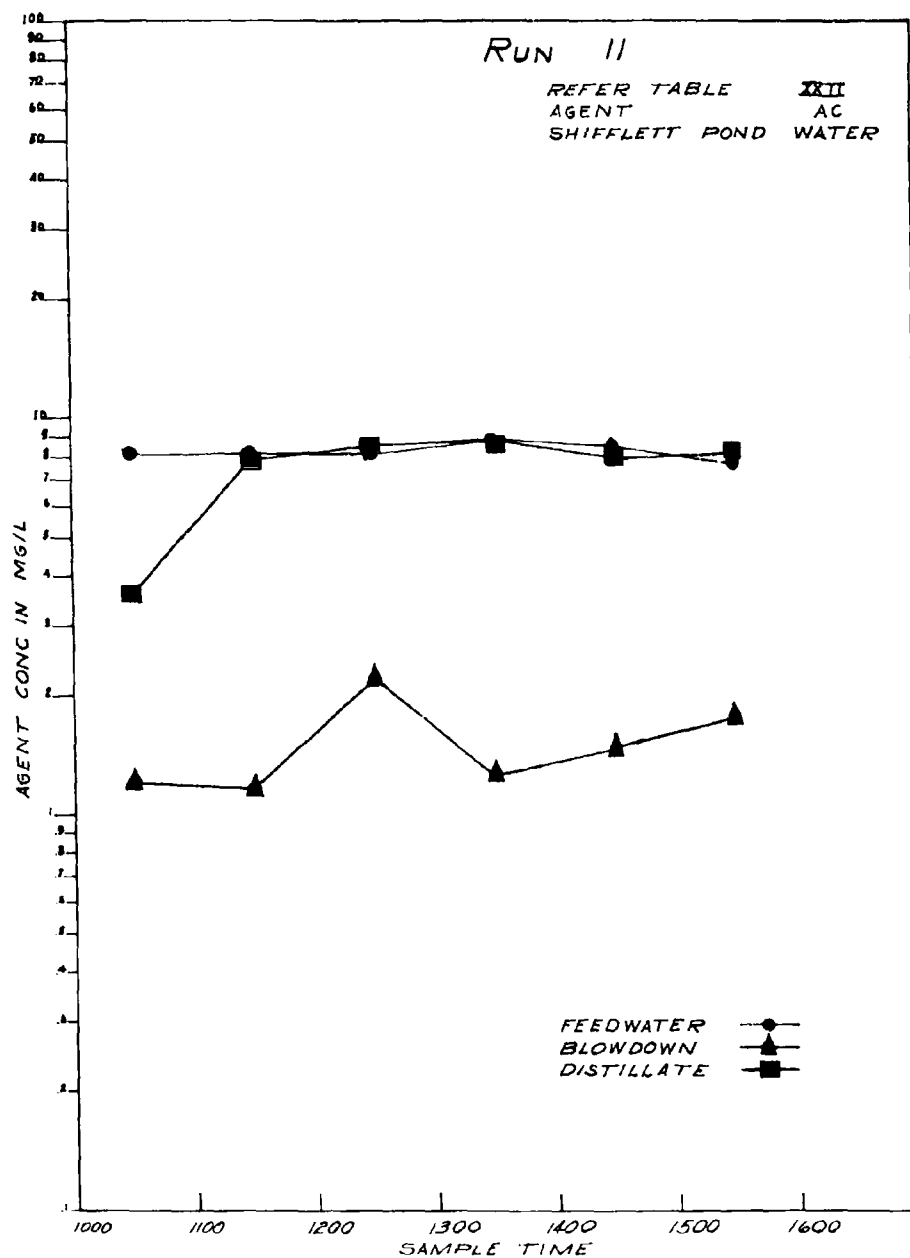


Fig. 25. Plot of data of Run 11.

Table XXIII. Run 12 - Removal of AC from Saline Water by Vapor Compression Distillation

DATE: 26 October 1962

FEEDWATER ANALYSIS: pH 7.3, turbidity 40 units, color 20 units, alkalinity 56 mg/l (CaCO₃), chloride 6400 mg/l (Cl)

CHEMICAL DATA

Sample Time (hr)	Feedwater		Distillate		Blowdown		
	pH	AC (mg/l)	pH	Chloride* (mg/l Cl)	AC (mg/l)	pH	Chloride* (mg/l Cl) Alkalinity (mg/l CaCO ₃) AC (mg/l)
1000	7.5	7.42	6.2	12.0	6.81	3.2	5,600 48 3.41
1100	7.3	7.48	6.6	8.0	6.90	3.1	17,400 48 2.45
1200	7.2	7.10	6.3	9.0	7.33	3.4	18,750 58 0.93
1300	7.4	7.30	6.3	9.6	7.06	3.2	19,350 56 1.33
1400	7.4	6.77	6.3	7.6	6.65	3.2	16,200 52 1.75
1500	7.2	6.13	6.2	9.2	7.19	3.1	19,800 62 1.12
Average	7.3	7.03	6.3	9.1	6.99	3.2	16,200 54 1.83

AVERAGE OPERATIONAL DATA

Length of Run: 5.2 hr
 Rates (gph): Distillate 101, Blowdown 57, Feedwater 153
 Temperatures: Feedwater 48° F, Blowdown 129° F, Distillate 59° F
 Distillate Conductivity: 0.285 micromho/cm
 Ambient Temperature: 43° F
 Percent Removal of AC: Distillate 0.6, Blowdown 74.0

* (Cl + CN) as mg/l Cl.

NOTE: Plot of data of Run 12 is shown on Fig. 26.

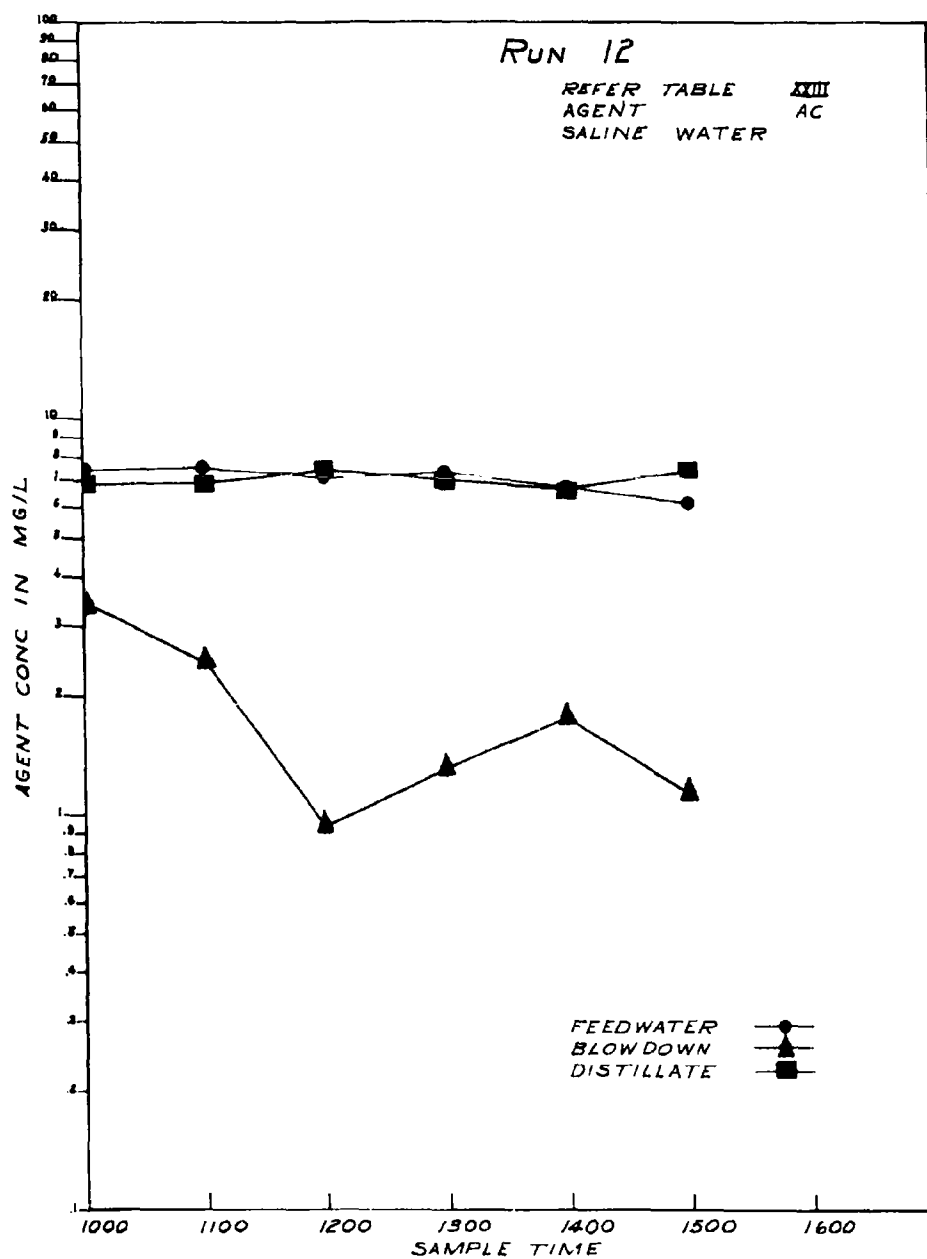


Fig. 2C. Plot of data of Run 12.

Table XXIV. Run 13 - Removal of HN-3 from Shifflott Pond Water by Vapor Compression Distillation

DATE: 31 October 1962

FEEDWATER ANALYSIS: pH 6.9, turbidity 60 units, color 70 units, hardness 21 mg/l (CaCO₃), alkalinity 22 mg/l (CaCO₃), chloride 15 mg/l (Cl), sulfate 22 mg/l (SO₄)

CHEMICAL DATA

Sample Time (hr)	Feedwater		Distillate		Blowdown		
	pH	HN-3 (mg/l)	pH	Chloride (mg/l Cl)	HN-3 (mg/l)	pH	Chloride (mg/l Cl) Alkalinity (mg/l CaCO ₃) HN-3 (mg/l)
1000	6.5	27.04	-	-	-	-	-
1100	6.7	25.86	5.8	3.4	Trace	8.4	202 36 0.0
1200	6.6	23.95	6.2	3.2	Trace	8.6	74 44 0.0
1300	7.1	22.60	6.4	6.0	0.0	8.9	78 51 Trace
1400	7.1	23.38	6.5	41.0*	Trace	8.6	85 54 Trace
1500	7.2	21.30	6.4	4.4	Trace	8.7	84 50 0.0
1600	7.3	21.05	6.6	5.0	0.0	8.1	85 42 0.0
Average	6.9	23.60	6.3	4.4	Trace	8.6	101 46 0.0

AVERAGE OPERATIONAL DATA

Length of Run: 5.4 hr
Rate (gph): Distillate 101, Blowdown 71, Feedwater 172
Temperatures: Feedwater 50° F, Blowdown 134° F, Distillate 68° F
Distillate Conductivity: 0.125 micromho/cm
Ambient Temperature: 48° F
Percent Removal of HN-3: Distillate > 99, Blowdown > 99

* Not included in the calculation of the average.
NOTE: Eight samples of distillate, unchlorinated, and one sample chlorinated to a 2.0 mg/l Cl₂ residual, gave no taste or odor.
Dashes indicate no determinations were made.

Table XXV. Run 20 - Removal of HD from Shifflett Pond Water by Vapor Compression Distillation

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DATE: 16 November 1962

FEEDWATER ANALYSIS: pH 6.2, turbidity 46 units, color < 70 units, hardness 220 mg/l (CaCO_3), alkalinity 32 mg/l (CaCO_3), chloride 54 mg/l (Cl).
(Note: Feedwater was put into tank previously used for saline water)

CHEMICAL DATA

Sample Time (hr)	Feedwater			Distillate			Blowdown					
	pH	HD (mg/l)	TDG* (mg/l)	pH	Chloride (mg/l Cl)	HD (mg/l)	TDG (mg/l)	pH	Chloride (mg/l Cl)	Alkalinity (mg/l CaCO ₃)	HD (mg/l)	TDG (mg/l)
1030	6.2	5.0	14.4	-	-	-	-	-	-	-	-	-
1100	6.1	8.3	16.8	6.6	0.2	0.0	3.2	3.3	1390	42	0.0	0.0
1200	6.1	5.3	15.0	7.0	0.5	0.0	2.6	3.0	2280	49	-	14.0
1300	5.9	3.0	14.7	6.3	0.5	-	5.6	7.3	1570	35	-	24.6
1400	6.1	0.3	18.7	6.6	1.0	-	4.7	7.7	2310	52	-	59.2
1500	6.7	1	15.1	3.2	0.5	-	2.5	7.4	7200	53	-	39.2
1600	6.3	1	13.3	6.4	5.0	-	2.5	7.5	4620	49	-	53.8
Average	6.2	3.4	15.4	6.6	1.3	0.0	3.5	7.7	3230	47	0.0	31.8

AVERAGE OPERATIONAL DATA

Length of Run: 5.5 hr
 Rates (gph): Distillate 101, Blowdown 49, Feedwater 150
 Temperatures: Feedwater 42° F, Blowdown 139° F, Distillate 70° F
 Distillate Conductivity: 0.111 micromho/cm
 Ambient Temperature: 50° F
 Percent Removal of HD: Distillate 100, Blowdown 100

* Thiodiglycol, hydrolysis product of HD.

NOTE: Two samples of distillate (1100 hr and 1430 hr), unchlorinated and chlorinated to 2 mg/l Cl_2 residual, gave no taste or odor.

Dashes indicate no determinations were made.

APPENDIX C

VAPOR TESTS ON VAPOR COMPRESSION DISTILLATION UNIT
(Tables XXVI through XXIX)

Table XXVI. Vapor Tests During VX Runs

TYPE OF TEST: Enzyme inhibition ticket
 EQUIPMENT: Squeeze bulb, 50 ml; buffer; indicator
 PROCEDURE: 50 bulb depressions in vapor area, hourly
 SENSITIVITY: 0.004 - 0.008 mg/l

Run No.	Date (1962)	Weather Conditions			Sampling Period (hr)	Sampling Location	Total Samples Taken	Reactions	
		General	Air Temp (°F)	Relative Humidity (%)	Wind Speed (fpm)			Negative	Positive
4	3 Oct	Clear, sunny	70-73	-	-	Compressor Blowdown sump	6 6	6 6	0 0
5	3 Oct	Overcast, cloudy, scattered showers	70-74	-	-	Compressor Blowdown sump	5 5	5 5	0 0
14	1 Nov	Clear, sunny	40-52	56-72	400-4100	Compressor* Blowdown sump	6 6	5 6	1 0
15	3 Nov	Rain, clearing	42-45	51-100	300-400	Compressor Blowdown sump	5 5	5 5	0 0

* Test tickets, exposed at compressor and over water in feed tank for 15 minutes, showed positive reactions.

NOTE: Dashes indicate no determinations were made.

Table XXVII. Vapor Tests During GB Runs

TYPE OF TEST: Enzyme inhibition ticket EQUIPMENT: Squeeze bulb, 50 ml; buffer; indicator PROCEDURE: 50 bulb depressions in vapor area, hourly SENSITIVITY: 0.004 - 0.008 mg/l											
Run No.	Date (1952)	General	Weather Conditions			Sampling Period (hr)	Sampling Location	Total Samples Taken	Reactions		
			Air Temp (°F)	Relative Humidity (%)	Wind Speed (fpm)				Nega- tive	Posi- tive	
6	10 Oct	Clear, sunny	68-80	48-71	350-420	0930-1500	Compressor Blowdown sump	7 6	7 6	0 0	
7	12 Oct	Clear, sunny	80-91	50-58	600-800	1100-1600	Compressor Blowdown sump Over feedwater	6 6 1	6 6 0	0 0 1	
9	18 Oct	Cloudy, overcast	50-70	60-75	<100	1000-2000	Compressor Blowdown sump Over feedwater Environment	11 11 1 1	10 10 0 1	1 1 1 0	

NOTE: Distillation unit inoperative 1635 hr to 1705 hr during Run 9.

Table XXVIII. Vapor Tests During GA Runs

TYPE OF TEST: Enzyme inhibition ticket EQUIPMENT: Squeeze bulb, 50 ml; buffer; indicator PROCEDURE: 50 bulb depressions in vapor area, hourly SENSITIVITY: 0.004 - 0.008 mg/l									
Run No.	Date (1962)	Weather Conditions			Sampling Period (hr)	Sampling Location	Total Samples Taken	Reactions	
		General	Air Temp (°F)	Relative Humidity (%)	Wind Speed (fpm)			Nega- tive	Posi- tive
8	16 Oct	Cloudy, overcast	70-78	70-95	280-900	Compressor Blowdown sump Environment	7 6 2	0 3 2	7 3 0
10	23 Oct	Clear, changing to overcast	64-72	-1-68	400-1000	Compressor Blowdown sump Over feedwater*	6 6 1	6 6 1	0 0 0

* One ticket, dipped into feedwater tank, gave positive reaction.

Table XXIX. Vapor Tests During AC Runs

TYPE OF TEST: Standard detection tube
 EQUIPMENT: Standard AC detection tube and bulb
 PROCEDURE: 50 bulb depressions
 SENSITIVITY: 10 mg/l

Run No.	Date (1962)	Weather Conditions			Sampling Period (hr)	Sampling Location	Total Samples Taken	Reactions	
		General	Air Temp (°F)	Relative Humidity (%)				Negative	Positive
11	25 Oct	Cloudy, overcast	42-49	39-59	1130-1530	Compressor Blowdown sump Environment	5	5	0
							5	1	4
							1	1	0
12	26 Oct	Clear	50-53	50-53	1100-1500	Compressor Blowdown sump	5	5	0
							5	0	5

APPENDIX D

RESULTS OF ACTIVATED CARBON AND
ION EXCHANGE RESIN COLUMN STUDIES
(Tables XXX through XXXVIII)

Table XXX. Test 1 - Removal of BZ from Distillate by
Passage Through Carbon and Ion Exchange Resin Columns

DATES: 27 September and 1 October 1962

COLUMN DATA: Each column - 500 ml volume and approximately
30 cm high

Carbon - Nuchar WA (+30 mesh)
Resin - Duolite C-25(H)

FEEDWATER: Distillate from Run 2 and Run 3

FLOW RATE: 50 ml/min (0.7 gpm/cu ft of resin)

SAMPLE VOLUME: 1000 ml

Run No.	Sample Time (hr)	Feedwater		Effluents			
				Nuchar WA		C-25(H)	
		pH	BZ (mg/l)	pH	BZ (mg/l)	pH	BZ (mg/l)
2	1200	6.3	0.022	-	-	-	-
	1300	6.1	0.035	-	0.0	-	-
	1400	5.9	0.034	-	0.007	-	-
	1500	6.3	0.036	-	0.0	-	-
3	1300	7.2	0.033	9.9	0.0	8.5	0.0
	1400	5.8	0.039	9.9	0.0	7.0	0.0
	1500	5.6	0.034	-	0.0	-	0.0

NOTE: One liter of Shifflett Pond water, contaminated with 16 mg/l BZ, showed complete removal of BZ after passing through the carbon column.

Dashes indicate no determinations were made.

Table XXXI. Test 2 - Removal of VX from Distillate by
Passage Through Carbon and Ion Exchange Resin Columns

DATE: 3 October 1962

COLUMN DATA: Each column - 500 ml volume and approximately
30 cm high

Carbon - Nuchar WA (+30 mesh)

Resins - Duolite C-25(H)

Duolite C-25(Na)

FEEDWATER: Distillate from Run 4

FLOW RATE: 150 ml/min (2.2 gpm/cu ft of resin)

SAMPLE VOLUME: 1000 ml

Sample Time (hr)	Feedwater		Effluents					
	pH	VX (mg/l)	Nuchar WA		C-25(H)		C-25(Na)	
			pH	VX (mg/l)	pH	VX (mg/l)	pH	VX (mg/l)
1100	6.7	1.12	9.3	0.0	6.3	0.00019	9.0	0.00031
1200	6.9	1.40	9.1	0.0	6.2	0.0	9.0	0.0
1300	7.0	1.52	9.6	0.00021	6.1	0.0	9.4	0.00024

Table XXXII. Test 3 - Removal of VX from Distillate
and Spiked Distillate by Passage Through
Carbon and Ion Exchange Resin Columns

DATES: 2, 5, 7, 8, 15 November 1962

COLUMN DATA: Each column - 500 ml volume and approximately
30 cm high

Carbon - Nuchar C-190 (+30 mesh)

Resin - Mixed-Bed (Barnstead Red)

FEEDWATER: Distillate of Runs 14, 15, 16, and spiked distillate of
Run 18

FLOW RATE: 150 ml/min (2.2 gpm/cu ft of resin)

EFFLUENT FRACTION VOLUME: 800 ml

Date (1962)	Feedwater VX (mg/l)	Column Effluents				
		Fraction Number	Nuchar C-190		Mixed-Bed Resin	
			pH	VX (mg/l)	pH	VX (mg/l)
2 Nov (Run 14)	1.3	1	10.2	Trace	8.6	Trace
		5	10.0	0.0018	6.8	"
		10	-	Trace	-	0.0
		15	9.6	0.0	-	0.0
		20	9.7	0.0	7.2	0.0
5 Nov (Run 15)	1.3	21	9.5	0.0	7.5	0.0
		25	9.4	0.0	7.0	0.0
		30	8.3	0.0	8.0	Trace
		35	8.3	0.0	7.9	0.0
		40	7.9	0.0	8.1	0.0
7 Nov (Run 16)	1.5	41	8.4	Trace	7.9	0.0
		45	7.7	"	7.8	0.0
		50	8.0	"	7.4	0.0
		55	7.4	"	8.0	0.0
		60	7.4	0.0003	7.4	Trace
8 Nov (Run 16 cont'd)	1.5	61	-	Trace	-	0.0
		65	-	0.0	-	0.0
		70	-	0.0	-	0.0
		75	-	0.0	-	0.0
		80	-	0.0	-	0.0
15 Nov (Run 18)	115	81	8.0	Trace	7.9	0.0009
		85	7.0	"	6.5	0.0015
		88	7.6	"	7.4	0.0011

NOTE: Dashes indicate no determinations were made.

Table XXXIII. Test 4 - Removal of VX from Spiked Distillate
by Passage Through Cation and Anion Exchange Resin Columns

DATES: 13, 14, 15 November 1962

COLUMN DATA: Each column - 500 ml volume and approximately
30 cm high

Resins - Duolite C-25(H)
Duolite A-30B(OH)

FEEDWATER: Distillate from Run 18 spiked to 114 mg/l VX
concentration

FLOW RATE: 150 ml/min (2.2 gpm/cu ft of resin)

EFFLUENT FRACTION VOLUME: 800 ml (Beginning with fraction number 21,
400 ml of C-25(H) effluent was fed to
A-30B(OH) column)

Date (1962)	Fraction Number	Column Effluents			
		C-25(H)		A-30B(OH)	
		pH	VX (mg/l)	pH	VX (mg/l)
13 Nov	1	3.4	0.061	-	-
	2	4.4	0.122	-	-
	3	4.5	0.139	-	-
	4	4.5	0.151	-	-
	5	4.8	0.156	-	-
	8	4.5	0.162	-	-
	10	4.7	0.084	-	-
	13	4.5	0.112	-	-
	15	4.6	0.105	-	-
	17	4.5	0.104	-	-
	20	4.4	0.090	-	-
14 Nov	21	-	-	-	-
	23	4.3	0.111	8.6	0.006
	30	4.4	0.095	7.3	0.021
15 Nov	31	-	-	-	-
	35	4.4	0.104	7.0	0.037

NOTE: Dashes indicate no determinations were made.

Table XXXIV. Test 5 - Removal of GE from Spiked Distillate by Passage Through Carbon and Ion Exchange Resin Columns

DATE: 13 October 1962

DATE: 12 October 1962
COLUMN DATA: Each column - 500 ml volume and approximately 30 cm high

Carbon - Nuchar WA (+30 mesh)

Carbon - Norlon 6015
Resins - Duolite C-25(H)

Dioptite $\text{Ca}_2\text{Si}_2\text{O}_7(\text{Na})$

Mixed-Bed (Barnstead Red)

fixed-bed (Pall-SBCA 100)

FEEDWATER: Mixture of Run 2 distillate and dis-

FEED RATE: 150 ml/min (2.2 gpm/cu ft of resin)

FLOW RATE: 150 ml/min (2.2 gpc/c)

Feedwater			Effluents									
Composition	Distilla- tion Unit	GB (mg/l)	Fraction No.	Nuclear WA		C-25(H)		C-25(Na)		Mixed-Bed		
				pH	GB (mg/l)	pH	GB (mg/l)	pH	GB (mg/l)	pH	GB (mg/l)	
Distillate	Feedwater	(ml)										
19,600	400	0.14	1	10.0	<0.1	2.6	0.10	8.8	0.07	7.3	<0.1	
"	"	"	2	-	-	2.7	0.26	9.0	0.13	-	-	
"	"	"	3	-	-	2.6	0.14	8.9	0.15	-	-	
"	"	"	4	9.5	<0.1	3.1	0.29	8.5	0.29	7.0	<0.1	
"	"	"	5	9.3	<0.1	-	-	-	-	8.8	<0.1	
19,600	400	0.12	9	-	-	-	-	-	-	-	<0.1	
"	"	-	13	9.4	<0.1	-	-	-	-	8.4	<0.1	
"	"	-	16	-	-	-	-	-	-	-	-	
19,000	1000	0.29	17	-	-	-	-	-	-	-	-	
"	"	"	23	8.8	<0.1	-	-	-	-	8.5	<0.1	

NOTE: Dashes indicate no determinations were made.

1

COLUMN DATA: Each column - 500 ml volume and approximately 30 cm high

FEED/WATER: Mixture of approximately 5 volumes distillate (Rm 10) and 1 volume distillation unit feedwater (saline); GA concentration 1.3 mg/l

EFFLUENT FRACTION VOLUME: 800 ml (400 ml of effluent from C-25(H) column fed to A-30B(OH) column)

NOTE: Dash indicates no determination was made.

Table XXXVI. Test 7 - Removal of AC from Distillate by Passage
Through Carbon and Ion Exchange Resin Columns

DATE: 26 October 1962

COLUMN DATA: Each column - 500 ml volume and approximately 30 cm
high

Carbon - Nuchar WA (+30 mesh)

Resins - Mixed-Bed (Barnstead Red)

Mixed-Bed (Prepared) (1 vol Duolite

C-25(H) and 2 vol Duolite A-30B(OH))

FEEDWATER: Distillate from Run 12 containing 7.0 mg/l AC

FLOW RATE: 150 ml/min (2.2 gpm/cu ft of resin)

EFFLUENT FRACTION VOLUME: 800 ml

Effluent Fraction No.	Effluents					
	Nuchar WA		Mixed-Bed (Barnstead)		Mixed-Bed (Prepared)	
	pH	AC (mg/l)	pH	AC (mg/l)	pH	AC (mg/l)
1	10.2	1.2	8.2	0	7.3	0
2	10.1	3.3	-	-	-	-
3	10.2	2.5	-	-	-	-
5	-	-	6.6	0	6.6	0
10	-	-	6.8	0	6.4	0

NOTE: Dashes indicate no determinations were made.

Table XXXVII. Test 8 - Removal of AC from Shifflett Pond Water
by Passage Through Carbon and Ion Exchange Resin Columns

DATE: 25 October 1962

COLUMN DATA: Each column - 500 ml volume and approximately 30 cm
high

Carbon - Nuchar WA (+30 mesh)

Resins - Duolite C-25(H)

Duolite A-30B(OH)

Mixed-Bed (Barnstead Red)

FEEDWATER: Shifflett Pond water contaminated with 8.3 mg/l AC

FLOW RATE: 150 ml/min (2.2 gpm/cu ft of resin)

EFFLUENT FRACTION VOLUME: 300 ml (400 ml of effluent from C-25(H)
column fed to A-30B(OH) column)

Effluent Fraction No.	Effluents							
	Nuchar WA		C-25(H)		A-30B(OH)		Mixed-Bed	
	pH	AC (mg/l)	pH	AC (mg/l)	pH	AC (mg/l)	pH	AC (mg/l)
1	9.9	0	4.0	2.53	8.9	0	8.0	0
3	9.4	1.68	-	-	-	-	-	-
5	9.2	3.91	4.4	6.87	9.2	0	6.6	0.1
7	9.0	3.48	-	-	-	-	-	-
8	-	-	-	-	9.1	0	5.0	0

NOTE: Dashes indicate no determinations were made.

Table XXXVIII. Test 9 - Removal of HN-3 from Spiked Distillate
by Passage Through Carbon and Ion Exchange Resin Columns

DATE: 31 October 1962

COLUMN DATA: Each column - 500 ml volume and approximately
30 cm high

Carbon - Nuchar WA (+30 mesh)

Resin - Mixed-Bed (Barnstead Red)

FEEDWATER: Mixture of approximately 4 volumes distillate (Run 13)
and 1 volume of distillation unit feedwater (fresh);
HN-3 concentration 5.2 mg/l

FLOW RATE: 150 ml/min (2.2 gpm/cu ft of resin)

EFFLUENT FRACTION VOLUME: 800 ml

Effluent Fraction No.	Effluents			
	Nuchar WA		Mixed-Bed	
	pH	HN-3 (mg/l)	pH	HN-3 (mg/l)
1	9.9	0.0	7.8	0.0
5	9.8	0.0	8.4	0.0
10	8.9	0.8	8.1	0.0

APPENDIX E

SPECIAL STUDY OF BATCH DECONTAMINATION

(Table XXXIX)

Table XXXIX. Special Study - Decontamination of Fresh Water Contaminated with VX
by a General Batch Treatment Procedure

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DATE: 19 November 1962

- PROCEDURE: 1. Fill lyster bag with 36 gal. VX-contaminated Shiflett Pond water - Sample #1.
2. Add 20 gm 70% $\text{Ca}(\text{OCl})_2$ (100 mg/l available chlorine). Stir and let settle 1/2 hr - Sample #2.
3. Add 82 gm activated carbon (Muchar 3-115) (600 mg/l). Stir occasionally 1/2 hr - Sample #3.
4. Add 7 gm FeCl_3 (50 mg/l) and 14 gm limestone (100 mg/l). Stir rapidly 2 min, stir slowly 4 min, and let settle 1/2 hr - Sample #4.
5. Pump supernatant through Arm/ 1/4-GPM Knapsack Filter (Set No. 1) - Sample #5.
6. Pump Set No. 1 effluent through mixed-bed ion exchange resin column - Sample #6.
7. Chlorinate final composite effluent to 2 mg/l residual chlorine - Sample #7.

Sample Number	Contaminated Water Analysis	Analysis After					
		1	2	3	4	5	6 7
VX (mg/l)	18.72	2.24	0.0017*	0.0026*	0.0029	0.0004	0.0003
pH	8.3	10.2	6.8	5.5	5.6	7.3	8.3
Turbidity (units)	55	-	-	20	1	1	-
Color (units)	>70	-	-	-	0	0	-
Hardness (mg/l CaCO_3)	24	-	-	1+3	144	-	-
Alkalinity (mg/l CaCO_3)	22	-	26	6	6	-	-
Chloride (mg/l Cl)	5.5	-	-	115	115	-	-
Sulfate (mg/l SO_4)	23	-	-	-	-	-	-
Conductivity (mg/l NaCl)	-	-	-	-	-	1.0	-
Chlorine Residual (mg/l Cl_2)	-	-	-	-	-	-	2.0

* Sample filtered before analysis.

NOTE: Dashes indicate no determinations were made.

Category 16 - Water Supply and Sanitation

DISTRIBUTION FOR USAERDL REPORT 1749-RR

TITLE Removal of Chemical Warfare Agents from Water by Vapor
Compression Distillation

DATE OF REPORT 7 June 63 TASK 8M75-05-001-07 CLASSIFICATION Uncl.

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