

NRL Report 6185

# Analysis of Gas Identification Training Sets of the E17 Series

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## ABSTRACT

The Gas Identification Training Set (GITS), currently under Army development for all-service chemical warfare training uses, has been studied for performance and contents. Of the three major groups of modern CW agents, the agent representation of two groups, mustard gas and a G agent, were found to be insufficiently stable to hydrolysis. There is no representative of the very important V group of agents in the GITS. Most of the obsolete or obsolescent CW agents included in the GITS are adequately stable.

Two mechanisms are postulated to account for hydrolytic instability of the CW agents with the sorbents used by the GITS. One is cosorption of agent and water on sorbents of large specific area, e.g., activated carbon. The other is cosolution of agent and water in elastomers or plastics capable of dissolving appreciable amounts of water, e.g., crepe rubber. To defeat both instability mechanisms, mixing or dissolving CW agents in plastics which absorb very little water is recommended. Examples of these are silicon-containing or fluorine-containing organic polymers.

For maximum realism, the recommended agent contents of the GITS is one agent each of the G, V, and H groups, plus G/V, G/H, and H/V mixtures.

## PROBLEM STATUS

This is a final report on this phase of the problem; work on the problem is continuing.

## AUTHORIZATION

NRL Problem C08-22  
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## ANALYSIS OF GAS IDENTIFICATION TRAINING SETS OF THE E17 SERIES

### INTRODUCTION

A definite and present need exists throughout the U.S. armed services for a compact, simple, safe, and inexpensive training set containing samples of the principal chemical warfare (CW) agents.\* The purpose of such a set is to furnish small amounts of genuine CW agents in the vapor state for use in training personnel realistically in the detection or identification of those CW agents which are of major military importance.

It is believed that a Navy CW detection training set should possess the following characteristics to the maximum extent practicable:

1. An adequate degree of safety under all foreseeable circumstances.
2. Adaptability to use on all ships and with all units ashore.
3. A shelf life of at least five years, and a service life of at least one year.
4. Light weight.
5. Small size.
6. Low cost, thus assuring wide distribution.
7. No special stowage required.
8. Adaptability for the inclusion of additional CW agents when and if required.
9. Adaptability for the inclusion of a liquid or liquids with which to demonstrate the responses of liquid-detector papers.

The above characteristics are believed to be essentially in agreement with all statements of military requirements for CW agent identification training sets. Differences exist, however, between the Navy and the Army in the number and identity of agents to be included in such a set, and on the desirability or feasibility of adding agent mixtures. The background of the Navy requirements for a CW agent identification set is discussed in Appendix B.

The specific applications in which a Navy CW agent detection training set would play an essential or important role are:

1. Familiarization of all recruit and shipboard personnel with the general manipulation and responses of Navy CW detection devices (kits, alarms, papers, etc.).
2. Conduct of specialized training, drills, and Operational Readiness Inspections for damage controlmen and other CW defense personnel in all aspects of the use of Navy CW detection devices.

\*A glossary of CW agent symbols is given in Appendix A.

3. Periodic checking of detector kits for sensitivity of response and state of preservation of chemical reagents.

4. Periodic checking of automatic field alarms for sensitivity of response.

Development of the Gas Identification Training Set (GITS) E17 was begun by the Army Chemical Research and Development Laboratory (CRDL) at the request of the Bureau of Ships (BUSHIPS). The first E17 set to reach this Laboratory was forwarded by BUSHIPS in 1958. A preliminary report was made, the comments of which are largely still valid (1). In 1960, an additional twelve E17 sets were forwarded by BUSHIPS for distribution to various U.S. Navy activities and other interested U.S. and foreign agencies for observation, use, and comment. A BUSHIPS letter stated the type of information desired from the user agencies (2). The recipients of the twelve E17 sets are listed below:

- (a) Naval Schools Command, Treasure Island, San Francisco, California
- (b) Hdqtrs., Wright Air Development Center, WAFB, Ohio
- (c) Naval Unit, Chemical Corps School, Ft. McClellan, Ala.
- (d) NRL Tropical Exposure Site, Canal Zone, Republic of Panama
- (e) Naval Construction Battalion Center, Davisville, R.I. (Two sets)
- (f) Naval Construction Battalion Center, Port Hueneme, California (Two sets).
- (g) British Joint Services Mission, Washington, D.C.
- (h) Canadian Joint Staff, Washington, D.C.
- (i) Naval Research Laboratory, Washington, D.C. (Two sets).

The E17 set and its component parts are shown in Figs. 1-3 with the composition of the CW agent source in each bottle of the original 1954 model given in Table 1 (Ref. 3).

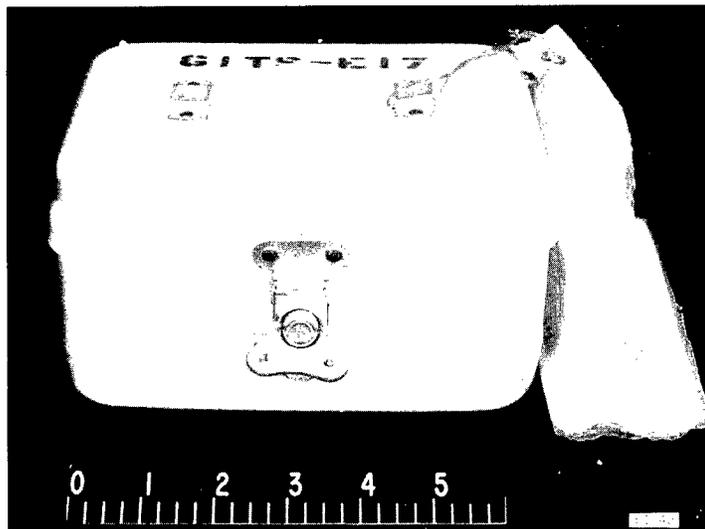


Fig. 1 - E17 exterior view

Fig. 2 - E17 interior view with two bottles inverted to show agent symbols on bottom

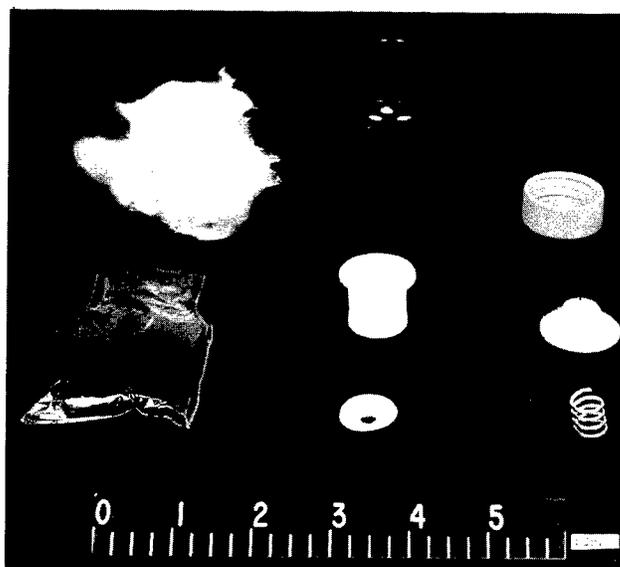
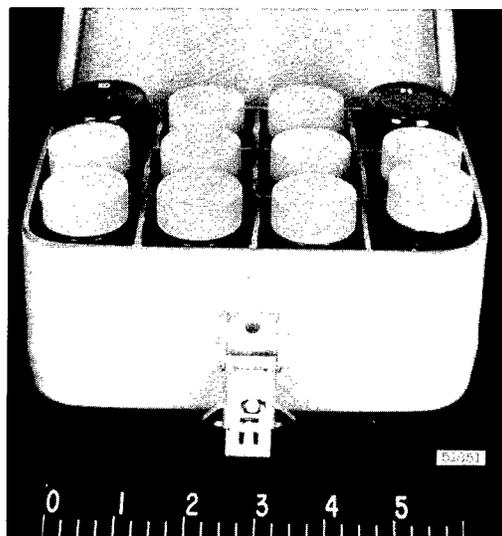


Fig. 3 - Components of a single E17 container; Scotchpak inner bag, glass wool plug, glass bottle, anodized aluminum bottle cap, Teflon bottlenecks liner and retainer (below bottle), Teflon cap liner and cadmium-plated loading spring (below bottle cap)

Table 1  
Composition of CW-Agent Sources in E17 Gas  
Identification Training Set

Agent	Contents of Bottle
GA	3 ml GF + 1 drop ethyl malonate on 11 g crepe rubber + 4 g powdered iron
GB	3 ml GF on 11 g crepe rubber + 4 g powdered iron
HD	3 ml HD on 9 g 12-mesh charcoal
HN1	3 ml HN on 9 g 12-mesh charcoal
L	3 ml L on 11 g crepe rubber
ED	3 ml ED on 11 g crepe rubber
AC	20 g potassium cyanide + 1 ml water
CK	5 g cyanogen bromide + 17 g petrolatum
CN	20 g CN
CG	20 g triphosgene
PS	3 ml PS on 9 g 12-mesh charcoal
CX	3 g CX on 10 g wet 6/16-mesh silica gel

#### EXPERIMENTAL PROCEDURES

Information supplied by some of the recipients of the E17 sets, has been collated in Tables 2 and 3. Table 2 shows the loss in strength of the agent vapor sources in the sets during use by several activities. It is considered significant that substantial loss of strength occurred during even very limited use. The very precise and detailed data supplied by the NRL Tropical Exposure Site follow in Table 3. The agent sources were tested weekly or biweekly as shown in Table 3. Detector kits M18 and M15 supplied the materials for the vapor tests. Because of a GB bottle which had been broken in transit, no tests were made for that agent and the kits provide no means for detecting PS and CN. The HN source could not be made to give a test even when first opened. To simulate agent attrition by evaporation and hydrolysis, such as would occur in regular use of the set by a training activity, the bottles containing the agent sources were left open for a period of one hour on four or five days of each week. During the 165 days that the set was under test at the NRL Tropical Exposure Site, the bottles were open for 109 one-hour periods.

In order to determine whether the E17 set bottles were adequately protected against breakage, a drop test was used (Table 4). A closed set, containing its full complement of 12 bottles, was dropped from desk height (31 inches) onto an asphalt-tiled concrete floor. Any broken bottles resulting from a drop were replaced before the next drop. In a first series of drops, the set was dropped once on each of the six faces, once on each of the eight corners, and once on each of the twelve edges. In this series, only three bottles were broken in 26 drops. In a second series, ten drops were made on the front face and nine on a side face. Eight bottles were broken in 19 drops in this series. In this connection, two broken bottles were reported among the ten sets distributed for evaluation.

Table 2  
Report of Three Activities on Performance of Gas Identification Training Set E17

Activity Reporting	Temperature During Tests (°F)	Rel. Humidity During Tests (%)	Total Time in Use (hr)	Agent Vapor Source Strength*					
				Start of Tests			End of Tests		
				Strong	Weak	Nil	Strong	Weak	Nil
Naval Schools Command, Treasure Island	40-90	35-45	60-90	Not Tested			CX,ED,CK	AC,HD,HN, L	GA,GB
Naval Unit Army Chemical School, Fort McClellan	40	90	1/4-3/4	CG,AC,GA,ED,CN,PS	CX,L	CK,HD,HNI	CG,ED,CN,PS	AC,GA	CK,GB,HD,HNI CX,L
Naval Research Laboratory Washington, D.C.	68-72	20-70	10	CG,AC,GA,ED,CN,PS,GB,L,CK,CX	HD,HNI			CG,AC,GA,ED,CN,PS,GB,L,CK,CX	HD,HNI

\*Difficulty was commonly experienced with sticking or "freezing" of the CG and CS bottle caps.

Table 3  
Report of NRL Tropical Exposure Site on Performance of Gas Identification Training Set E17

Agent	Bulb Compressions*													
	1960									1961				
	9/20	9/28	10/5	10/12	10/19	11/2	11/18	12/5	12/19	1/3	1/16	1/30	2/14	2/27
GA	15	15	15	15	50	75	100	120VF	150	>150†	>175	>175	>175	>175
HD	25	25	25	30	30	45	60	75	75	75	100	125	125	125
L	12	12	36	60	100	120	120F	140	150	150	150F	150F	150F	150F
ED	5	5	5	5	5	8	10	15	20	20	20	20	20	15
AC	1	1	1	6	9	9	12	13	13	13	13	13	13	13
CK	2	25	36	55	55	65	100	120F	>140	>140	>140	>150	>150	>175
CG	1	1	2	4	7	8	50	>120	>120	>120	>120	>120	>120	§
CX	12	20	85	>100	>120	>120	†							
Temperature avg of daily avg (°F)	78	79	79	79	78	78	78	79	78	81	81	80	80	81
RH avg for periods when bottles open (percent)	85	95	83	96	87	90	91	79	91	82	75	73	74	83

\*In all cases the number of bulb compressions was that required to match a standard color, unless an "F" (fainter than the standard color) or a "VF" (much fainter than the standard color) appears. In the case of HD, CG, and CX, the standard color is that shown in the M18 kit instructions. In the case of the other agents, it was necessary to make special color standards. The approximate colors of these standards are: L, pale blue; ED, blue-black; AC, deep blue; CK, dark brick red; GA, light yellow-orange.

†A > sign indicates no color was obtained after this number of bulb compressions.

‡Although deposits were brushed from the bottle threads each time it was opened, at this point the cap became permanently attached to the bottle.

§Phosgene tubes of M18 kit became discolored.

Table 4  
Fragility Test of Bottles in Gas Identification Training Set E17

Number of Drops	Impact Area of Set	Number of Broken Bottles	Location of Broken Bottles in Set
6	Faces	2	Corner near face impacted
8	Corners	0	-
12	Edges	1	Corner near edge impacted
10	Front Face	2	Corner near face impacted
9	Side Face	6	Corners near face impacted: 2 Center of face impacted: 1 Center of front or rear face: 3

A further test was conducted to provide an indication of the effectiveness of the seal provided by the spring-loaded Teflon tapered plug in the bottle-cap seating against the Teflon bottleneck insert. The need for this test was indicated by the occasional odor of CW agents noticed when the lid of a set was opened. Moreover, the frequent sticking of the caps on CG and CX bottles showed that these agents or their hydrolysis product (hydrogen chloride) were escaping past the seal and attacking the aluminum caps. To provide access to the interior of a capped bottle, the bottom of a bottle was cut off smoothly. The contents were removed, and a one-hole rubber stopper substituted for the base of the bottle. Laboratory compressed air at 1.0 psi was admitted to the bottle via the rubber stopper. When the bottle was placed under water, bubbles of air escaped from the cap-bottle junction at a rate of about two per second. The same leak occurred when the cap was loosened and retightened. This amount of leakage suggests that the cap could not be considered leakproof, and that both loss of agent vapor from the bottle and penetration of water vapor into the bottle could occur at an appreciable rate. Common commercial phenolic plastic caps, with conventional liners, did not allow any detectable leakage when substituted for the aluminum/Teflon cap and subjected to the same pressure test.

## DISCUSSION

### Stability of Agent Sources

Tables 2 and 3 show that the individual vapor sources of the E17 set have a much shorter service life than the one-year military characteristic figure set in 1955 (1). There are also strong indications in the tables that the five-year shelf life expected has not been attained (1). For example, Table 2 lists several vapor sources for which the source strength was weak or nil when the set was first opened. Thus, within a few months of manufacture, several vapor sources in unopened bottles deteriorated from initial values, presumed to be very strong, to weak, or zero-strength sources. These observations are confirmed by the preliminary CRDL report (1). Table 5 presents summary stability data extracted from both the report (1) and the data of the NRL Tropical Exposure Site.

The above figures, obtained from two entirely independent sources under widely differing conditions, are regarded as a reliable summary of the stability of the CW agent sources in the 1954-model of the E17 set. The reasons for the varying stabilities of the

Table 5  
Performance of Gas Identification Training Set E17

	Agent Vapor Source Strength		
	Strong	Weak	Nil
Chemical Corps (Obtained After Two Months' Storage of Set at 125° F)	ED AC CG	HD HN1 GA GB	CK L CX
NRL Tropical Exposure Site (Obtained After One Month's Daily Simulated Use)	ED AC CG	HD GA CK L	HN1 (nil when received) CX

different agents are believed to be related in part to the nature of the sorbents, where such are used, and in part to the varying degrees of chemical stability of the agents themselves. The AC and CG sources (potassium cyanide and triphosgene, respectively), which use no sorbent, are about 20 grams each of pure agent, and probably remain strong sources for long periods simply because of the very large quantity of agent available. An additional factor making for stability of the CG source is that there is present no substrate or sorbent of large specific surface, such as activated charcoal, to enlarge the area of contact with adsorbed water and thereby promote hydrolysis. The persistent strength of the ED source may be attributed to stability toward hydrolysis plus a possible large solubility in the rubber substrate used for this agent, although there are no specific data available to support this inference. The low stability of GF (the actual G agent used in both the GA and GB sources) is probably related to the well-known autocatalytic decomposition reaction of the G family, which is promoted by water and acid decomposition products. A good moisture adsorbent or reactant, plus an acid-accepting stabilizer, would probably extend substantially the life of the GF sources. The short life of HD and HN on charcoal, under the conditions of use of the E17 set, is not surprising. Both of these agents hydrolyze by a similar mechanism at a rapid rate (the hydrolysis half-life of HD at room temperature is only about five minutes). The activated charcoal substrate used for HN1 and HD in the 1954 model of the E17 set can serve as an efficient medium for the hydrolysis reaction because (a) it adsorbs both CW agents and water strongly, and (b) its very large specific area (over a thousand square meters per gram) allows intimate molecular contact of the sorbed agents and water. This and related mechanisms involved in the decomposition and stabilization of CW agent vapor sources are discussed in Appendix B.

It should be pointed out, however, that HD is quite stable on activated charcoal which has been extremely well dried. Brown has shown in this Laboratory that an activated carbon/HD mixture, in which the carbon is acid-washed and dried in vacuo, still functioned as a useful source of HD vapor after 18 months' storage in which it was sealed from contact with atmospheric moisture (4).

This apparatus was designed primarily as an exceptionally rugged, large-capacity portable source of GB agent vapor for the testing of E21 automatic alarms in special shipboard operations. In addition to the use of very dry activated carbon as agent substrate, it provides for the drying of the air drawn through the carbon bed. By these means the hydrolysis of CW agents on activated carbon can be reduced to a low level.

#### Packaging

The packaging of the E17 sets has been studied particularly with regard to the bottles, the bottle closures, and the plastic case. The only deficiency of the case is in the shock

protection given the glass bottles. The necessary improvement should be easy to achieve by the addition of small amounts of semirigid foamed plastic padding, although this alteration will presumably necessitate a concurrent enlargement of the case.

The bottles and the bottle closures present several problems. The noticeable increase in friction between the bottles and bottle caps, which develops after a short period of use, seems to be associated with deposits of metallic aluminum on load-bearing sections of the bottle threads. These deposits, which are readily detectable with a hand lens, suggest that the anodizing on the cap threads is incomplete or nonadherent, and that the friction is characteristic of the galling action of aluminum on aluminum. Although the initial increase in friction between bottles and caps is not particularly objectionable, it does appear to increase with progressive failure of the anodizing on the cap threads. More important, loss of the anodizing coating allows rapid attack on the cap threads by the more active CW agents in combination with atmospheric moisture, or possibly by atmospheric moisture alone. Treatment of the bottle cap threads with a Teflon dispersion or a molybdenum disulfide enamel remain possible remedies. The procedure of Johansson and Torok for producing water-repellent surfaces on glass insulator bodies would be expected, in the present situation, to form low-friction surfaces on the agent-source bottles and bottle caps (5). The treatment involves dipping in a dilute silicone oil solution, followed by a high-temperature bake. Silicone baking enamels would serve similarly. The "freezing" of bottle caps, recorded in the case of CG and CS bottles, almost certainly indicates attack on the aluminum cap by hydrogen chloride resulting from agent hydrolysis. Bottle cap sticking became so severe at the NRL Tropical Exposure Site that pliers were frequently required to remove the caps. A more durable coating is therefore required if the CG bottle is retained. However, the fact that phosgene and CX are able to escape past the Teflon seal and reach the bottle cap, together with the air pressure test, indicate that the Teflon seal is not gas tight. The significance of this degree of leakage for other agents is not clear at the present, and a further study should be made using the proposed molded seal (1). However, the general design and performance of the seal appear good, with the exception of the failure to contain CG and CX vapors completely, and the probable slow inleak of atmospheric water vapor. Therefore, no major alteration in design is recommended now, other than a change to the new moldable FEP Teflon. Injection molding of the seal components with FEP Teflon will result in a drastic reduction in the cost of the seal and, at the same time, permit a better mating of the Teflon surfaces. This latter change should reduce, if not entirely eliminate, the apparently characteristic leakage of the present machined Teflon bottle seal.

It is believed that one of the elements of the E17 packaging system may contribute to the instability of the agents. This is the glass wool which is used to prevent loss of sorbent or solid agent from the bottles. The hydrophilic surface of the glass fibers represents a large surface on which sorbed water and agent can react. Converting the glass fiber surface to a hydrophobic one would in no way interfere with the mechanical function of the glass wool, and might increase materially the stability of the agent present. The procedure of Johansson and Torok (5) would serve effectively to treat the glass wool.

#### Selection of Agents

The number of CW agents in the 1954 model of the E17 set is believed excessive by almost any standard, including agents for which the detector kits cannot test effectively (PS and CN) as well as several obsolete or obsolescent agents (CK, ED, AC, HN, L, CG, and CX). It is believed that if Navy personnel learn to perform the kit tests for V, G, and H agents with precision and rapidity, detection skills will be at an optimum as far as operational identification-detection of CW vapors at sea is concerned. Even if it is anticipated that other classes of CW agents will occasionally be encountered, it is still preferable to use available training time in perfecting detection techniques for the high-probability agents. Tests for the low-probability agents are provided and described in the M18

Table 6  
Chemical Warfare Agents Contained in Gas Identification Training Sets\*

M1 (1942)	M2 (1946)	E9 (1952)	E17 (1954)	E17R1 (1962)	E17 (1963)	E17 Modified (proposed)
H	HD	HD	HD	HD	Nerve Agent	HD
H	H	HN1	HN1	HN3	Nerve Agent	G
L	L	L	L	L	Nerve Agent	V
CG	CG	CG	CG	CG	Nerve Agent	HD + G
DM	DA	CK	CK	CK	HD	HD + V
CN	CN	CN	CN	CN	HD	G + V
	AC	GA	GA	GA	CG	HD
		GB	GB	GB	CG	G
			ED	CS	AC	V
			AC	AC	AC	HD + G
			PS	Bleach	Bleach	HD + V
			CX	CX	Bleach	G + V

\*Repetition of an agent symbol indicates duplicate bottles in set.

detector kit, and the color changes are adequately illustrated in the M18 instructions; no drills for the agents or samples of them in a Gas Identification Training Set are required.

In addition to the above-mentioned advantages in training and readiness which would follow simplification of the E17 set, it is presumed that a cost reduction would occur also. Thus, by a design philosophy similar to that of the M15 detector kit, emphasizing simplification, a very wide distribution of the training set would become feasible. Table 6 lists the agents used in previous agent identification training sets, the E17 (1954), the E17R1, the E17 (1963) model still under development, and in a version proposed by NRL for maximum usefulness to the Navy.

The similarities and differences between the four E-17 sets in Table 6 should be kept in mind. All four present an identical appearance; case, bottles, bottle caps, Teflon seals, and plastic bottle-liners are identical. All differences, therefore, are in the agents and agent sorbents. The E17R1 set made only a few changes but they were a step in the right direction. Two of the obsolete agents of the E17 (1954) set, ED and PS, were removed and replaced by CS and VX. The VX addition was a "paper" change only, however, and a bottle of STB bleach was placed in the set pending development of a suitable VX source. Another noteworthy change in the E17R1 was the elimination of activated charcoal as an agent sorbent. Its replacement was shavings of polymethylmethacrylate plastic, which have been found by the CRDL to afford markedly improved HD stability.

After study and review of the E17R1 set by various Army commands in 1962-63, a decision was made by the Army to eliminate more of the obsolete or obsolescent agents, and HN, L, CK, CX, and CS were removed in the 1963 model. CS is a standard agent but was considered to serve no useful purpose in the set. Agents CG and AC, although obsolete, were retained in the set. They are among the most stable sources in the E17, and their removal would have left the 12-compartment E17 case with only two CW agents, HD and a nerve agent (the same GF which originally was used in the E17 to represent GA and GB). The provision of four GF sources is not for multiple simultaneous use. Since a satisfactorily stable G agent source has not been developed, it is intended that the four

bottles will be used sequentially: the second bottle will not be opened until the first is no longer serviceable, etc. No explanation was made available for the exclusion of VX or another V agent from the 1963 model. VX is the most important single CW agent known to the U. S. and her allies, and is radically different from the other standard nerve agent GB in its vapor pressure and its ability to penetrate human skin. Both the M18A1 and M15A1 detector kits have the ability to distinguish between the V and G series of agents. As far as the fitness of an E17 set for Navy use is concerned, it appears that a mistake has been made in the lack of provision in the 1963 model to demonstrate realistically the abovementioned ability of the detector kits. Separate G and V sources are required for this important segment of CW detection training.

The E17 modified set listed in Table 6 does not now exist; it is a proposal, but it is believed to be a highly realistic, practical, and pedagogically sound set for Navy use. Its 12 bottles supply six sources each of HD, G, and V, thus allowing multiple simultaneous use. For example, an instructor could use one bottle to demonstrate a test in an M15A1 kit, and immediately thereafter five teams of students could use the remaining five bottles of the chosen agent (HD, G, or V) to practice the test themselves with individual M15A1 kits. Thus, the proposed E17 modified for Navy use would be designed for collateral use with six M15A1 kits. Many ships would not muster this many M15A1 detector kits. Therefore, it may be desirable to manufacture a six-place GITS, half the size of the E17 models. Such a six-place set, if outfitted with the following agent sources: HD, G, V, HD/G, HD/V, and G/V; would still provide three sources each for HD, G, and V tests. Further, two six-place sets, at little more than the cost of one twelve-place set, in many Navy and Marine Corps situations would provide double the training opportunities of a twelve-place set. The mixed agent concept is believed to be operationally realistic. Mixed CW agents were the rule in World War I, and their use must be expected in the future whenever an attacker believes that the additional effort in mixing the agents is more than compensated by the abnormal difficulties to which the defense would be subjected.

The list of agents proposed for the E17 modified set, given in Table 6, is based on several factors:

1. It matches exactly the principal detection capabilities of the M15A1 detector kit.
2. In six bottles it provides three sources of each of three agents – in effect a total of nine sources in six bottles. A corresponding advantage applies in a twelve-bottle set.
3. It provides mixtures with which to drill trainees in the principle that a positive test for one agent does not exclude the possibility of the presence of other agents.
4. By its agent complement, it impresses on the trainee the CW agents which he is most likely to encounter.
5. The use of three mixed-agent bottles allows the replacement of one or two of them by bottles of new or newly emphasized agents while still retaining an example or two of the mixture principle.

#### Miscellaneous Factors

Inasmuch as the word "gas" has fallen into disfavor as an abbreviation of chemical warfare agent, it is suggested that the full name of the E17 set be changed to Chemical Warfare Agent Identification Training Set. If the resulting acronym is not acceptable, the name Chemical Agent Training Set is suggested.

The extensive treatment given olfactory detection in the E17 set instructions is believed unwise, and should be drastically reduced. A statement similar to the following would be suitable: "The most dangerous CW agents have very faint odors when present in attack concentration. Even these faint odors can be concealed by normal battle smells, camouflaged by mixture with stronger smelling CW agents or diluents, or simply overlooked. Therefore, detection of CW agents by odor is generally unreliable and unsafe, and should be resorted to only under very exceptional circumstances. To test your sense of smell, remove cap from bottle and sniff once the inside of cap only. Do not touch the inside of the cap or plastic closure with your hand or nose."

#### CONCLUSIONS

Developmental models of the Gas Identification Training Set (GITS) are inadequate in resistance to degradation of the CW-vapor sources by atmospheric water vapor. It is believed, however, that this hydrolytic degradation can be greatly reduced by dissolving the agents in a plastic or polymer which has a very low solvency for water.

The GITS can be further improved by removing CW agents of secondary or minor importance and adding a V agent vapor source. Sources containing G/V, G/H, and H/V are also desirable from the standpoint of operational realism.

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## APPENDIX A

### GLOSSARY OF CHEMICAL WARFARE AGENT AND RIOT CONTROL AGENT SYMBOLS\*

- AC Hydrogen cyanide; HCN. Nonstandard in U.S.
- CG Phosgene; carbonyl chloride;  $\text{COCl}_2$ . Standard B in U.S.
- CK Cyanogen chloride;  $\text{CNCl}$ . Standard B in U.S.
- CN Chloroacetophenone;  $\text{C}_6\text{H}_5\text{COCH}_2\text{Cl}$ ; a riot control agent. Standard B in U.S.
- CS O-Chlorobenzalmalonitrile;  $\text{ClC}_6\text{H}_4\text{CH}(\text{CN})_2$ ; a riot control agent. Standard A in U.S.
- CX Phosgene oxime; dichloroformoxime;  $\text{Cl}_2\text{CNOH}$ . Nonstandard in U.S.
- DA Diphenylchloroarsine;  $(\text{C}_6\text{H}_5)_2\text{AsCl}$ ; a riot control agent. Nonstandard in U.S.
- DM Adamsite; diphenylaminochloroarsine; chlorophenarsazine;  $\text{NH}(\text{C}_6\text{H}_5)_2\text{AsCl}$ ; a riot control agent. Standard A in U.S.
- GA Tabun; ethyl phosphorodimethylamidocyanidate;  $(\text{CH}_3)_2\text{N}(\text{C}_2\text{H}_5\text{O})\text{CNPO}$ . Nonstandard in U.S.
- GB Sarin; isopropyl methylphosphonofluoridate;  $\text{CH}_3(\text{C}_3\text{H}_7\text{O})\text{FPO}$ . Standard A in U.S.
- GF Cyclohexyl methylphosphonofluoridate, a member of the G agent group.
- H Mustard gas; impure material made during World Wars I and II by Levinstein process. H is about 70 percent pure mustard, has a stronger odor than HD (see below), and is unstable in storage. Standard B in U.S.
- HD Mustard gas, commercially pure; bis(2-chloroethyl) sulfide;  $(\text{ClC}_2\text{H}_4)_2\text{S}$ . The symbol originally signified H purified by distillation, but now applies to any mustard gas of 92 percent purity or better, regardless of method of preparation. Standard A in U.S.
- HN1 A nitrogen mustard; ethyl bis(2-chloroethyl) amine;  $(\text{ClC}_2\text{H}_4)_2\text{NC}_2\text{H}_5$ . Nonstandard in U.S.
- HN3 A nitrogen mustard; tris(2-chloroethyl) amine;  $(\text{ClC}_2\text{H}_4)_3\text{N}$ . Nonstandard in U.S.
- L Lewisite; dichloro (2-chlorovinyl) arsine;  $\text{ClC}_2\text{H}_2\text{AsCl}_2$ . Obsolete in U.S.
- PS Chloropicrin; nitrochloroform;  $\text{NO}_2\text{CCl}_3$ . Nonstandard in U.S.
- V A group of nerve agents characterized by low volatility and exceptional skin penetration.

\*Standard A indicates type classification as the most advanced and satisfactory items currently available to fill operational requirements; Standard B, as items which have limited acceptability to fill operational requirements, and which are normally used or issued as substitutes for standard A items. Obsolete items no longer have any degree of standardization in the U.S. Nonstandard items have never had any degree of standardization in the U.S.

## APPENDIX B

## CHEMICAL WARFARE AGENT IDENTIFICATION TRAINING

The only instrument available in World War I for detecting toxic gas and vapors was the human nose. Even after the introduction of the M9 and Mark 1 detector kits in 1944, the U.S. Navy Defensive Chemical Warfare Manual still rated the olfactory sense as the principal means of CW detection (B1). This manual stated: "There are three methods by which a chemical warfare agent can be detected and identified. They are odor, observation, and gas detection devices. . . . it is of utmost importance that gas personnel be able to detect and identify the odors of chemical warfare agents." Although statements similar to this one were sometimes qualified by warnings on the lack of reliability of the sense of smell as a CW detector, odor-recognition of the CW agents occupied an important place in CW training until the mid-fifties (B2).

The official policy of leaning heavily on the sense of smell for CW identification-detection was supported at the beginning of World War II by two training devices. These were the Gas Identification Set, Instructional, M1, commonly called a "sniff set"; and the Gas Identification Set, Detonation, M1. The former was a case of seven small bottles containing the following agents, either pure or sorbed on charcoal: HD (2 bottles), PS, L, DM, CN, CG (from triphosgene decomposition). The detonation set was also used for training in CW odor recognition, but was designed for outdoor training only. Glass vials of the agents, HD, L, PS, and CG, either as pure liquids or in solution were shattered by electric blasting caps and the liquid atomized and vaporized into the air. Groups of 15 to 20 men placed downwind from the detonation experienced together the odor of the agent detonated.

Although the M1 sniff set was very widely used in CW training, none of the World War II training manuals appear to have appreciated its value as a source of CW vapors for detector kit demonstration and training. It is noteworthy that the value of a vapor source in demonstrating the functioning of a detector kit was recognized, however, in the design of the earliest U.S. detector kit, the M1/M4. This kit, for detecting mustard gas only, contained a bottle of mustard-reacting liquid with which to test or demonstrate the response of the kit (B1).

Near the end of World War II, the M1 sniff set was replaced by the M2 model (B3). In the new set, one bottle of low purity (Levinstein) mustard was replaced by distilled mustard (HD), the U.S. agent Adamsite was replaced by an analog preferred by the Axis nations (DA), and a bottle containing potassium or sodium cyanide was added. This compound yields AC by slow hydrolysis in traces of moisture.

By the early 1950's work was underway to update the M2 identification set by improved packaging (glass bottles were replaced by aluminum ones and the canvas carrier by a styrenated rubber case), and by changes in the selection of agents (B3).

The new set of eight agents included HD, HN, L, CK, CG, CN, and simulants for GA and GB. Documents related to the new set, designated the E9, make it clear that the set was intended solely for olfactory training.

The E9 set was never standardized (B4). Probably the principal factor in its rejection was its design for and strong emphasis on CW detection by odor. By this time it was generally recognized that training men to attempt CW detection by odor was not only likely

to fail in its objective of developing a useful olfactory detection ability, but also involved risks of toxic effects. These handicaps to CW odor detection followed the introduction of the G agents which were characterized both by very faint odors and a new order of toxicity among war gases.

By 1954, three main factors, beside the inadequacy of the E9 set, served to emphasize the Navy's need for an up-to-date CW agent set for identification training. The first factor was the very wide acceptance and distribution of the E27R4/M15 detector kit, developed by the Army Chemical Corps for the Navy as a kit for high-probability agents only. It was distinctly possible that many Navy and Marine Corpsmen would be called on to use the kit with essentially no prior training. For these men especially, but also for other users, exploitation of the training fundamental "learn by doing" required that a set of sources of CW-agent vapors be readily available for use in conjunction with the detector kit. With the M15 or M15A1 detector kits and CW identification training sets readily available in approximately equal numbers to ship and shore personnel, optimum conditions would exist for frequent practice and drill in CW detection. Considering an identification training set as an item complementary to M15 kits, the identification set would be expected to contain exclusively or principally the CW agents for which the kit itself was designed. In addition, the factor of complementarity between the identification training set and the detector kit suggested that the former ideally would correspond roughly in size, weight, and cost with the detector kit.

The second factor which gave emphasis to the Navy's need for a set of CW-agent-vapor sources was the E21 G-agent automatic alarm. Essentially the entire production of this pioneer device, ultimately some 680 units, was purchased by the Bureau of Yards and Docks (BUDOCKS) in pursuance of its obligations for the passive defense of Navy installations in the U.S. and overseas. As with the detector kit, a source of G-agent vapor is highly desirable, if not indispensable, to test realistically the overall response of the alarm and to train operators. Certain other chemicals will give the G-agent reaction (Schoenemann reaction) used in the alarm, but the genuine agent is a more convincing and respected training aid than a nontoxic substitute. At present, a red-spotted strip of white plastic is inserted into the optical system of the alarm to simulate the colored spot resulting from the true G-agent reaction. This is a satisfactory test of the optical, photoelectric, and electronic components of the alarm, but does not check the somewhat less dependable liquid transfer, mechanical, and chemical functions. G-agent vapor checks the entire alarm.

The third factor which stressed the Navy's requirement for a CW-agent vapor training set was a recognition that the V-agent group of nerve agents were not only CW agents of the first rank but also that they posed detection problems of a difficulty previously unknown. The very low volatility of the V agents, coupled with their supertoxic effects, made it plain that detection-by-odor had reached a new low in reliability and safety among CW-detection methods. The various nonsensory detection devices therefore achieved a correspondingly increased importance in CW defense, and the need for a complementary set of CW-vapor sources was more evident.

The Army position relative to a CW identification training set is stated by Raun (B3). The proposed set to succeed the E9 would not be used for agent odor training by the Army; instead the Gas Identification Set, Detonation, M1 would be used for this purpose to the extent that odor demonstration or training was required. Further, the proposed set was at all times considered in connection with and as an adjunct to the M9A2 (now M18) detector kit. Because the latter had a ten-agent detection capability, samples of the same agents, or suitable simulants, were considered desirable in the set.

The Navy's need for a GITS which would be directly complementary to the M15 detector kit and the E21 alarm had no parallel in Army requirements. Both of these detection devices were exclusively Navy items in their early procurement and distribution.

For a time there was a legitimate question over the advisability of including genuine nerve agents in the proposed set because of the possible hazards to users. This concern was dispelled by assurances from the Chemical Corps laboratory personnel and by a special report from this Laboratory (B5). This question stimulated discussions on the possibility and desirability of replacing genuine agents in the proposed set by nontoxic materials which would give the correct kit and alarm tests, and afford the true agent odor, where such an odor existed. It was noted that to accomplish this substitution uniformly for all agents was a difficult problem and would require considerable research. Moreover, some of those concerned recognized that this step would, by implication, appear to acknowledge that the CW agents involved could not be handled safely in training operations even under the relatively favorable conditions provided by good packaging of small amounts of the agents. The principal effect of this discussion was to allow the nerve agent GF, which has a longer life in the training set than GA or GB, as a "simulant" for both these agents when suitable artificial odors are added.

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## APPENDIX C

### MECHANISMS INVOLVED IN THE DECOMPOSITION AND STABILIZATION OF MIXTURES OF CW AGENTS AND SORBENTS

No systematic study of the mechanisms involved in the decomposition and stabilization of CW agents mixed with sorbents has been reported. It is axiomatic that these mechanisms must follow well-known laws of physical chemistry. In the absence of experimental data, therefore, it is both reasonable and desirable to formulate tentative mechanisms, using scientific analogy and inference as extensively as possible.

It is believed reasonable to assume that the main chemical reaction causing the decomposition of chemical agents, contained in glass bottles and mixed with comparatively inert materials such as activated carbon and glass wool, is hydrolysis. Oxidation may play a part in the decomposition of the amino nitrogen-containing nerve agents, as it is known that amines are generally oxidizable by atmospheric oxygen. However, the tendency toward hydrolysis is a general one for CW agents, and the hydrolysis rates, or half-lives, are well known.

If hydrolysis is to take place at the standard rate for a given agent, the agent must be dissolved in the water. That is, the individual molecules of agent must be completely interspersed with water molecules. Under these conditions, the rate of reaction (hydrolysis) for the prevailing temperature is at a maximum, and equal to the standard hydrolysis rate. If, however, the agent exists in particles larger than molecular size (the agent is not fully dissolved in the water), then all the agent molecules are not in contact with water molecules and the reaction rate is less than the standard rate. A good example of the latter situation can be cited using mustard gas. This agent has a hydrolysis half-life of about five minutes when dissolved in water at ordinary temperatures. However, mustard gas also has a very small solubility in water, and, therefore, may exist in contact with water or moist materials for surprisingly long periods without hydrolyzing. The story is told that an excavation for a construction job in World War II encountered a World War I dumping area for mustard gas or mustard-contaminated materials. In spite of normal ground moisture, the mustard resisted hydrolysis for over twenty years, and a hazardous degree of contamination still existed. The story is scientifically believable on the grounds of the very low solubility of mustard gas in water, plus the complete absence of mechanical mixing or stirring together of the agent and water.

Another consideration is that even a CW agent highly insoluble in water can be caused to react with water at an appreciable rate if the agent and water are spread together in layers of molecular thickness. This method of increasing the rate of reaction of two substances of low mutual solubility by establishing large contact areas is demonstrated in the alveoli of the lungs. Oxygen has a very low solubility in water, but rapid reaction and exchange can occur by virtue of the large area of the blood-air interfacial membrane provided in the alveoli. This method is also effective when a CW agent is adsorbed on activated carbon (activated charcoal). Such carbons have surface areas of over a thousand square meters per gram, and adsorb both CW agents and water vapor readily. It is plausible that in the situation of co-adsorption of agent and water on activated carbon, conditions are favorable for relatively rapid hydrolysis of even a water-insoluble agent. From this point of view, the use of activated carbon for many years as the substrate or sorbent for CW agents in gas identification training sets was a poor selection. An exception to this judgment is the case in which the carbon/agent mixture is prepared and maintained in a very dry condition.

The next consideration is that, if an agent can be kept separate from, or out of contact with, water molecules, the agent can be stabilized against hydrolysis. The effecting of this method is not immediately obvious when the agent involved is appreciably soluble in water or strongly hygroscopic like VX and when atmospheric moisture is present. The method can be applied, however, if the agent is dissolved in a liquid, plastic, or polymer which itself has a very low solubility for water.

The methods stated should, as a matter of procedure, be checked against experience before assembling them inductively into a general rule or scientific specification for stable agent-sorbent mixtures. The first two methods can be regarded as sufficiently fundamental in physical chemistry to be considered axiomatic. The third method is supported by direct experience of the instability of activated carbon/agent mixtures in gas identification training sets. It is also supported by a wide variety of evidence in physiological chemistry, catalytic cracking and reforming of petroleum, etc. The fourth method is supported, it is believed, by the observation that mustard gas sorbed in polymethylmethacrylate plastic (e.g., Lucite or Plexiglas) shows much improved stability over mustard gas sorbed in activated carbon.\*

It is believed that sufficient evidence or experience has been cited to establish a reasonable degree of acceptability for the four methods stated above. Therefore, they can be consolidated into a single working standard on which to base future investigation. There are three main requirements for an agent sorbent in a gas identification training set:

1. The sorbent should have an adequate solubility for the agent.
2. The sorbent and agent should not react chemically.
3. The sorbent should have a very low solubility for water. Additionally, it should be actually water repellent to eliminate molecular films of water on its surface.

It is proper to ask how well the currently used sorbents conform to the above requirements and, additionally, how well they are actually succeeding in stabilizing the agent vapor sources. As noted in the body of this report, the 1963 model of the E17 set contains only two liquid CW agents, GF and HD (as far as is known, VX has not yet been added to the set). The substrates for these two agents are understood to be crepe rubber and polymethylmethacrylate. The GF/crepe rubber mixture shows a substandard stability, and four sources per set are thought to be necessary to provide a year's service life. The HD/methacrylate mixture is understood to provide a much improved (compared to HD/carbon) stability, and perhaps an entirely satisfactory one. These data are compatible with the qualitative observation that crepe rubber has an appreciable solubility for water, and that polymethylmethacrylate plastic has a low solubility for water. An indication of the magnitude of these factors is given in data cited by Houwink.† Natural rubber at 100 percent relative humidity may absorb five to ten percent or more water whereas polymethylmethacrylate under similar conditions absorbs 1.5 percent.

The stability characteristics of the agent vapor sources in the 1963 model of the E17 set are: HD, very good, possibly room for improvement; GF, mediocre, much room for improvement; VX, no source offered because of poor stability. These data indicate that there are marked deficiencies in present agent substrates in the E17 Set. Since the three

\*J.A. Stein, B.J. Wood, B.W. Fromm, and D.N. Kramer, "Development of the E17 Gas Identification Training Set (GITS)," U.S. Army Chemical Warfare Lab. Report CWLR 2327, Dec. 1959

†R. Houwink, Editor, "General Theory," Vol. I of "Elastomers and Plastomers," New York: Elsevier, 1950

requirements suggested above purport to offer systematic guidance to selection of improved substrates, some particular types of sorbents which are consistent with the requirements will be reviewed.

It is believed best to consider initially the second and third requirements, i.e., absence of chemical reactivity with the liquid CW agents, and extremely low solubility for water plus water repellency (contact angle of water on substrate as large as practicable, >90 degrees). These two criteria appear to be generally well satisfied by two large classes of compounds, the fluorocarbons and the silicones (pure hydrocarbon oils, greases, waxes or polymers are a possible but somewhat less promising group). Both classes contain oils, waxes, greases, and rubbers; all can be considered as candidate agent sorbents or, in some cases, diluents. The crux of the experimental problem is then to examine the candidates for their ability to dissolve suitable amounts of the desired agents; their extremely low or zero solubility for water can be considered as a class characteristic. This discussion does not suggest that such a search is necessarily easy, or even that success is certain; only that a sound hypothesis points to the silicones and fluorocarbons as a fruitful field for study.

It is believed appropriate to note two alternatives which may offer routes to the desired stable sources of agent vapor even if a silicone or fluorocarbon solvent for HD, GF, and VX cannot be found. These two routes are based on diffusion of the agent through the silicone or fluorocarbon, rather than solubility in them.

The first alternative is to mix or emulsify the agent in a silicone, fluorocarbon, or hydrocarbon grease, wax, rubber, resin, or thixotropic-gel substrate while the substrate is in a reasonably fluid state due to an elevated temperature or low extent of polymerization. A conventional acid-accepting stabilizer for the nerve agents may be incorporated at this stage also. As the substrate reverts or progresses to the semisolid or solid state, a mechanically stable dispersion of agent in substrate will exist. A well-chosen substrate will allow diffusion of the agent out of the substrate while not allowing the diffusion of water to the agent. The rate of mass transfer of the agent through, and out of, the substrate is the resultant of two factors; solubility of agent in substrate and diffusion rate of individual agent molecules in the substrate. These two factors are largely independent, and can be regarded roughly as dependent, respectively, on molecular compositions and molecular shapes. Thus, the agent solubility in the substrate can be very low, but a relatively large diffusion rate can assure a satisfactorily rapid evaporation of the agent from the substrate surface.

The second alternative is somewhat similar to the first, but utilizes diffusion of the agent through a thin film of a silicone, fluorocarbon, or even of a hydrocarbon. It will be recalled that common polyethylene film has the interesting characteristic of transmitting some organic vapors at a relatively fast rate while transmitting water vapor at a very slow rate. Thus, even a polyethylene bag may be usable to contain a CW agent on an entirely inert solid substrate such as glass wool (rendered water repellent) porous or foamed glasses, silicas, metals or organic polymers, or on a relatively inert solid which will combine with traces of water penetrating the bag (e.g., barium peroxide or calcium sulfate). The bag will, in general, allow outward diffusion of the agent faster than inward diffusion of water vapor.

It may be useful to call attention to differences in the agent vapor pressure characteristics of the three classes of agent substrates which have been discussed above. These classes are: the adsorbents, such as activated carbon; the solvents, such as oils, greases, waxes or polymers in which CW agents are soluble; and the absorbents, such as porous or granular inert materials. All three classes of substrates will supply agent vapor at approximately the vapor pressure of the pure agent when they are saturated with agent. However, marked differences in agent vapor pressure appear when partial saturation of the substrate obtains. Since this situation is the normal one in agent/substrate mixtures

in CW-agent identification training sets, these considerations are important in the basic design of such sets. At partial saturation of CW agents on activated carbon, the equilibrium pressure follows a characteristic adsorption isotherm, and is very low at low loadings, but approaches the saturation pressure at moderate and high loadings. The essentially complete retention of agents by carbon at low loadings is necessary in a gas mask canister, but represents unusable agent in an agent identification set.

Agents in the solvent substrates show equilibrium vapor concentrations in accordance with Henry's Law. That is, the vapor concentration will be proportional to the liquid or solid phase concentration or activity. This means that the vapor concentration or pressure will decrease essentially linearly as the concentration of the agent in the substrate decreases. This is a marked improvement over adsorbent substrates. In absorbent substrates there is no decrease of agent vapor pressure or concentrations in the vapor phase as long as any liquid agent remains. Thus, the vapor concentration remains constant, at a given temperature, for 90 to 95 percent of the life of the agent source. Moreover, the equilibrium vapor pressure and concentration are fully predictable from the properties of the pure agent.

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