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NATIONAL BUREAU OF STANDARDS REPORT

6A140

MONTHLY PROGRESS REPORT
MOBILE LABORATORY PROJECT

FC

by

W. C. Wolfe
A. K. Hannel
and
W. M. Walton

ASTIA

DEC 18 1958



U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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U. S. DEPARTMENT OF COMMERCE

Charles Sawyer, Secretary

NATIONAL BUREAU OF STANDARDS

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10. Building Technology. Structural Engineering. Fire Protection. Heating and Air Conditioning. Exterior and Interior Coverings. Codes and Specifications.
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13. Ordnance Development. Mechanical Research and Development. Electromechanical Fuze. Technical Services. Missile Fuzing Research. Missile Fuzing Development. Projectile Fuze. Ordnance Components. Ordnance Tests. Ordnance Research.
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This is a 15 Page Document

NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT
0502-30-3205

November 14, 1952

NBS REPORT
6A14C

MONTHLY PROGRESS REPORT
MOBILE LABORATORY PROJECT

by

W. J. Wolfe
A. E. Hamel
and
W. W. Walton

Surface Chemistry Section
Chemistry Division

To

Chemical Corps
Chemical and Bacteriological Laboratories
Army Chemical Center, Md.

Contract No. DA-18-107-CML-2783



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I. ANALYSIS OF CW AGENTS AND HYDROLYSIS PRODUCTS

A. Preliminary work on methods.

In order to measure volumes of liquids, as for the peroxide fusion in Part III of the Analytical Scheme, we calibrated a capillary tube of the size to be used for such measurements. The inside diameter of the tube selected was 1.5 mm, as determined by the gauge of nichrome wire which would just fit inside the tube. The tube was weighed and then clean, dry mercury added with a capillary dropper. The height of the mercury column was measured, using millimeter graph paper, and the tube reweighed. The weight of the mercury, at 27°C, was 1.3008 g and the height of the mercury column was 59 mm. The density of mercury at 27°C is 13.5290 and the volume of 1.3008 g of mercury at this temperature is 0.095 ml or 96 lambda. The volume of a column of 59 mm mercury in a 1.5-mm capillary tube is 96 lambda and the volume of 1 mm of mercury in a 1.5-mm capillary tube is 1.6 lambda. This compares with a value of 1.70 cu. mm (approx. 1.7 lambda), reported by Schneider, Qualitative Organic Microanalysis, John Wiley & Sons, N.Y., 1945, p. 6.

In the Analytical Scheme it is sometimes necessary to adjust the pH of solutions within one pH unit. For this purpose, we tested "Accutint" pH papers, manufactured by Anachemia, Ltd., Montreal, Canada, and sold by E. H. Jargent Company. We also tested "pHydrion" paper, a well known substitute for "wide-range indicator paper". The papers were tested on buffer solutions made from tablets supplied by the Coleman Electric Company, Maywood, Illinois. A Beckman pH meter was used to check the pH of the buffer solutions. The results are recorded in Table 1.

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TABLE 1

pH Values

Indicated on vial	Found by meter	Found by phyrion paper	According to "Accutint" papers					
			Wide range		Fractional range			
			Paper No.	pH found	Paper No.	pH found		
2.00	2.08	2	A	1-2	20	2.0		
			B	1-2	30	2.1		
					40	2.1		
					50	2.0		
					Accepted value	2.1		
3.60	3.48	4	A	3	30	3.3		
			B	4	50	3.3		
					60	3.9		
					70	3.9		
					Accepted value	3.9		
					Accepted value	4.3		
4.10	4.00	4	A	4	60	4.3		
			B	4	70	4.2		
					Accepted value	4.3		
					Accepted value	5.3		
5.40	5.20	5	B	6	70	5.3		
					80	5.3		
					90	5.2		
					100	5.3		
					Accepted value	5.3		
							100	7.0
							110	7.0
		120	6.9					
8.10	3.28	8	B	9	Accepted value	6.9		
					120	8.4		
					130	8.5		
					140	8.5		
					150	8.6		
					Accepted value	8.5		
9.8	9.59	10	B	11	160	9.9		
					170	9.8		
					180	10.1		
					Accepted value	9.9		
							Accepted value	9.9

Apparently the "phyrion" paper is more satisfactory than the "wide-range" Accutint" paper. However, a combination of "phyrion" and "fractional range Accutint paper" is satisfactory for determining pH within one unit.

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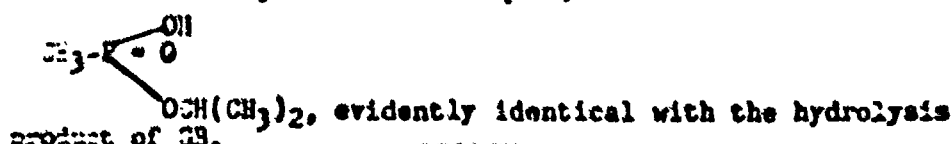
Solutions were made up containing chlorine, arsenic, phosphorus, sulfur, boron, fluorine and nitrogen, in ionic form and tests for these elements, were performed as described in Part III of the Analytical Scheme. On the basis of these tests and on tests made with agents and hydrolysis products, further revisions were made in the Analytical Scheme. These revisions will be included in the final form of the Scheme, now in preparation.

3. Experiments with warning agent.

As a safety measure, some of the operations in the Analytical Scheme were performed on butyl mercaptan, a well known warning agent. This substance is easily detectable in air at a concentration of about 8.1×10^{-5} parts per million or 22 gamma per liter of air, according to Katz and Talbert, U. S. Bureau of Mines Technical Paper 1402 (1930). The lethal concentration of G-agents is not over 50 gamma per liter of air. The material was transferred, using a capillary dropper, to ampoules and to capillary tubes and a peroxide fusion was conducted, all in a good hood. No odor could be detected by any one of three persons at any time. A capillary tube containing butyl mercaptan was carried out of the hood into the room and centrifuged. While removing the tube from the hood, odor was detectable only in the vicinity of the tube. However, considerable odor was noted on centrifuging. A capillary tube containing 1-2 lambda of butyl mercaptan was crushed on the floor of the room (about 2000 cu. ft. of air space). The odor was strong at first and was estimated, from the tables of Katz and Talbert, *op. cit.*, to correspond to a concentration of about 1-5 parts per million. After evacuating for about 10 minutes with the hood fan, no odor was detectable.

4. Analysis of hydrolysis products of G-Agents.

Two compounds were obtained from the Army Chemical Center, identical with hydrolysis products of G-Agents. One of these compounds was a liquid, with the formula

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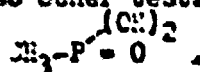
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Our work indicates, however, that GB reacts with two moles of sodium hydroxide to form a dibasic acid. This compound is a viscous liquid, soluble in water and insoluble in petroleum ether. A sodium peroxide fusion was performed on the liquid, as in the Analytical Scheme pp. 72-4 and the fusion solution was tested for phosphorus as in the Scheme pp. 91-2, beginning at line 12 in (3) Preliminary tests for arsenic and phosphorus. The revised Analytical Scheme will clarify the preliminary test for phosphorus in the absence of iodine. No further test was performed on this compound.

The other compound from the Army Chemical Center was a light-tan, crystalline solid, soluble in water and ether but insoluble in petroleum ether. No other tests were performed. The formula was given as



D. Analysis of MW toxic agents.

In order to test the procedures in the Analytical Scheme a complete analysis was made on mustard gas (ii). Some clay soil, 175 grams, was contaminated with 0.30 ml of crude mustard gas from the Army Chemical Center. The soil was then extracted with petroleum ether and the extract evaporated as in Part I of the Analytical Scheme. The residue was distilled in the micro fractionating still as on pp. 33-37 of the Scheme. About 60 lambda of distillate was obtained (37 mm in a capillary tube 1.5 mm i.d.). The distillation temperature was determined from a potentiometer reading of 4.3 millivolts to be 100°C at 11 mm pressure. The distillate was fused with sodium peroxide and analyses for chlorine and sulfur made as recorded in Table 2. Functional group analyses are included in Table 3.

Analyses according to Parts III and IV in the Analytical Scheme were performed on the mustard gas distillate mentioned above and also on the following agents obtained from the Army Chemical Center: GA, GB, ethyl fluoracetate, HN1, Lewisite and H-1217. Table 2 is a summary of the ultimate analysis, as in Part III of the Analytical Scheme and Table 3 summarizes the functional group analyses as in Part IV of the Scheme.

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TABLE 2

<u>Agent</u>	<u>Elements found by qualitative tests</u>	<u>References, pages in the Analytical Scheme</u>
GA	Phosphorus (preliminary test) Phosphorus (detection) Nitrogen	81-82 83-84 87-88
GB	Phosphorus (preliminary test) Phosphorus (detection) Fluorine	81-82 83-84 87
Ethyl fluoroacetate	Fluorine	87
HX1	Chlorine Nitrogen	76 87-88
Lewisite (L)	Chlorine Arsenic	75 82-83
Mustard gas (H)	Chlorine (from halogen precipitate) Sulfur (from solution obtained from precipitation of halogen group)	76-79 85
RL-1217	Iodine (on fusion solution) Iodine (from halogen precipitate) Nitrogen	75 77-78 87-88

In every case, all elements other than carbon, hydrogen and oxygen, indicated by the structural formulas, were detected by the qualitative tests.

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TABLE 3

Agent	Analytical Scheme Test No.	Pages	Name of test	Results
GA	1	103-105	Acidity or basicity	Neutral
	2	105-106	Hydrolysis to acidic compounds	Positive; weakly acidic; no change on heating
	3	106-107	Iodide test for oxidizing agents	Negative; colorless solution
	4	107-108	ICl test for reducing agents	Negative; very faint pink color
	20	131-132	Hydrolysis to ammonia	Positive; rust colored precipitate immediately on heating
	3c	151-152	Test for alkoxyphosphines with acetic acid and HBr	Negative; light yellow color
	4c	153-154	Test for cyanophosphines with ferrous sulfate	Positive; dark greenish-blue color
GB	41	154-155	Test for phosphoric acid formation with HBr	Positive; copious fine, yellow precipitate within one minute
	42	155	Azino fluorophosphates; test for azine formation with HCl	Positive; immediate large, yellow crystals
	1	103-105	Acidity or basicity	Positive; weakly acidic immediately; strongly acidic on standing
	3	106-107	Iodide test for oxidizing agents	Negative; colorless solution
	4	107-108	ICl test for reducing agents	Negative; light orange color

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TABLE 3 (Cont'd)

Agent	Analytical Scheme Test No.	Pages	Name of test	Results
GB	33	145-146	Test for fluoride ion formation with sodium hydroxide	Positive
	34	146-147	Test for fluoride ion formation with hydrochloric acid	Positive
	36	148-149	Test for fluoride ion formation with sodium iodide in acetone	Positive; yellow color
	38	151-152	Test for alkoxyphosphines with acetic acid and HBr	Positive; red-brown final color
	41	154-155	Test for phosphoric acid formation with HBr	Negative; no precipitate
Ethyl fluoroacetate	8	112-113	Test for hydroxamic acid formation with hydroxylamine	Positive
	13	116-122	Test with DBJ reagent	Very weak or negative; faint pink color on heating; increased on adding second portion of base; no initial color or precipitate
	14	122-123	Sodium ethoxide test	Positive; immediate precipitate; no color
	15	123-124	Silver perchlorate test	Negative; no precipitate on heating
	36	148-149	fluoride ion test with sodium iodide in acetone	Positive; yellow color
	HNI	1	103-105	Acidity or basicity
2		105-106	Hydrolysis to acidic compounds	Positive; weakly acidic on heating
4		107-108	ICI test for reducing agents	Positive

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TABLE 3 (Cont'd)

<u>Agent</u>	<u>Analytical Scheme</u>	<u>Pages</u>	<u>Name of test</u>	<u>Results</u>
HN1	5	108	Iodine test for reducing agents	Positive but weak
	13	118-122	Test with DB3 reagent	Positive; strong final and initial colors; precipitate and disappearance of color on heating
Lewisite (L)	1	103-105	Acidity or basicity	Strongly acidic; immediate deep red color
	4	107-108	ICI test for reducing agents	Positive; dark purple color
	5	108	Iodine test for reducing agents	Positive but weak; light yellow color
	13	118-122	Test with DB3 reagent	Immediate precipitate, which increased after 30 seconds; no change on heating; light red color on adding base
	37	149-151	H ₂ O ₂ test for primary, secondary and tertiary arsenicals	Positive; dibasic acid formed, indicating primary arsenical (First end point, 37 lambda 1 N NaOH; total, 57 lambda 1 N NaOH)

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TABLE 3 (Cont'd)

<u>Agent</u>	<u>Analytical Scheme</u>		<u>Name of test</u>	<u>Results</u>
	<u>Test No.</u>	<u>Pages</u>		
Mustard gas (H)	1	103-105	Acidity or basicity	Neutral on stand- ing
	2	105-106	Hydrolysis to acidic compounds	Positive; strongly acidic immediately and more acidic on heating
	13	112-122	Test with DB3 reagent	Faint violet lor on 30 sec. in. standing; lark purple color on heating (darker than color stand- ard); even darker on adding base; no precipitate at any time
	15	123-124	Silver perchlorate test	Positive; immediate precipitate
	23	135-138	Sulfonium salt formation with methy. iodide	Positive; deep blue final color
	25	138-139	Hydrolysis to alkyl mercaptans or hydrogen sulfide	Negative; yellow color
TL-1217	3	106-107	Iodide test for oxidizing agents	Negative; color- less solution on heating
	4	107-108	Iodine test for reducing agents	Positive
	5	108	Iodine test for reducing agents	Negative; deep orange color more intense than color standard
	7	108-110	Carbon dioxide evolution	Positive; white precipitate in 2-3 minutes

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TABLE 3 (Cont'd)

Agent	Analytical Scheme Test No.	Pages	Name of test	Results
TL-1217	8	112-113	Hydroxamic acid formation with hydroxylamine	Negative; no change in color
	13	116-122	Test with 15% reagent	Negative; no color or precipitate
	14	122-123	Sodium etheride test	Negative
	15	123-124	Silver perchlorate test	Insoluble in benzene; could not perform test
	17	125-128	Chlorosauric acid test	Soluble in water; red-brown precipitate; silver oxide gave basic solution to wide-range indicator (pH = 8), indicating a quaternary salt.

Some of the results with the G-Agents are of particular interest. For example, GB gave a positive test and GA a negative test for alkoxyphosphines with the glacial acetic acid-hydrogen bromide reagent. (Test No. 38). Both GA and GB contain an alkoxy group attached to phosphorus but differ in the other groups about the phosphorus atom. Both have the coordinate covalent phosphorus-oxygen linkage but GA has dimethylamino and cyano groups and GB has a methyl group and a fluorine atom attached to phosphorus. Evidently the ease with which alkoxy groups are split off from phosphorus depends on the nature of other groups attached to the phosphorus atom. On the other hand, GA was readily split by heating with 48% aqueous HBr to give phosphoric acid (Test No. 41), while GB gave a negative test. The negative result with HBr is not surprising, as the methyl

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group should not readily be split off from the phosphorus atom.

Test No. 33, for fluoride ion formation with sodium hydroxide, is misleading as written. The addition of the alkaline reaction mixture of GB and NaOH solution to the thorium-alizarin lake (see Analytical Scheme pp. 145-6) gave a red color. This might be interpreted as a negative test, since fluoride ion should destroy the lake and give a colorless or light yellow solution. The red color, however, is due to the amount of sodium hydroxide introduced, which turned the alizarin indicator red. Addition of acid decolorized the solution and addition of excess thorium nitrate to the acid solution restored the red lake.

The reaction of GB and 6 M sodium hydroxide solution was also carried out on a larger scale, with measured amounts of the reactants and standardized alkali solution. After reacting for two minutes at 100°C as usual, the mixture was titrated with standard acid solution. The results showed that two moles of sodium hydroxide reacted per mole of GB, indicating the formation of a dibasic acid. This would necessitate the splitting off of both the fluoride atom and the isopropoxy group.

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II. REVISION OF THE ANALYTICAL SCHEMES

- A. Corrections and additions made in Amendment No. 1, US Report 6A125, September 2, 1952.
- B. Further corrections and additions.
 1. Revision of 2. Physical properties of toxic agents, Analytical Scheme pp. 13-23.

This list is to be transferred to the end of the first volume of the Scheme and will be expanded to include newer agents. Information on the newer agents has been requested from the Army Chemical Center.

2. Classification of CW toxic agents.

In order to complete the Analytical Scheme and make possible a systematic analysis of field samples, we are preparing a summary, which will make possible identification of field samples with a minimum of effort and time. The system which we are using is believed to be an improvement on the "Tabular Summaries" in FLN 1-2-16 Revised, July 1944. Our system for the classification of toxic agents is similar to that used by Mulliken and Huntress in their work on the identification of organic compounds. Compounds are first classified according to elementary composition. Under each class are listed responses to the functional group tests in Part IV of the Scheme for various agents and functional groups belonging to that class. The elementary composition of field samples would be determined by carrying out the procedures in Part III of the Analytical Scheme. Each sample would then be located in its proper class according to our system, and identified by its responses to the tests.

3. Quantitative Analysis for Acidic and Basic Elements.

This subject is to be covered in a separate manual, since the quantitative procedures are to be performed in the Base Laboratory and not in the Mobile Laboratory. A draft of this manual has been prepared, using material from FLN 1-2-4 Revised, August 1944.

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III. LIST OF MATERIALS AND EQUIPMENT

The final additions and corrections to the list of materials and equipment for the Mobile Laboratory have been completed and sent to the Chemical Corps, Army Chemical Center, Maryland.

Respectfully Submitted,

W. W. Walton

W. W. Walton
Project Leader.

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