

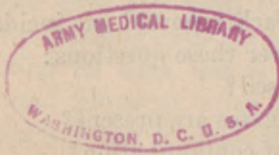
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Identification of Chemical Warfare Agents



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Medical Division
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Identification of Chemical Warfare Agents

*S. Office of civilian defense,
medical division.*

PURPOSE

This bulletin presents information concerning sampling and identification of chemical warfare agents, and methods for delimiting a contaminated area.

It is a compilation of the most useful published tests together with others from private sources available to the U. S. Office of Civilian Defense. All methods of detection which are listed have been tested both in the laboratory and in the field and represent those which have proved to be the most satisfactory. For field testing, it is expected that each local gas defense group will select for each chemical warfare agent one or perhaps two tests which are most suitable for their particular organization. Other tests may be used for further investigation in local gas detection laboratories. No "official" test for each agent has been designated, since individual groups will vary in their facilities for identifying the war gases.

It should be emphasized that speed must be exercised in making field identifications. It is advisable to obtain a tentative identification of the agent quickly, so that definite information will be available in advising other Services. In general, it will be best to make a preliminary identification by odor and to confirm this impression by a single specific test. After this has been done, the Gas Reconnaissance Agent is in a position to forward specific information to the Control Center. Complete analyses and further confirmatory tests can be made in either a local or Chemical Warfare Service laboratory.

IMPORTANCE OF RECONNAISSANCE

In connection with each reported gas incident, the Gas Reconnaissance Agent must answer these questions:

1. Has a gas been used?
2. If so, what gas or gases are present?
3. What is the area of contamination?
4. (After decontamination.) Has decontamination been complete?

The first question must be considered, since in the confusion of an air raid war gas may be reported by mistake, when another type of gas alone may be present, e. g., gas from HE bombs, broken gas mains, bombed chemical plants, etc. It is also possible that the enemy might attempt to create panic by using some unfamiliar, spectacular, but otherwise harmless substance. It has been found by the British that the Gas Reconnaissance Agents have a very important function during

air raids even though war gas has not been used. Hardly a raid occurs but that war gas is reported and a Gas Reconnaissance Agent disproves its presence.

Information as to the gases used will be of value to the Emergency Medical Service in connection with first aid measures and treatment of gas casualties. The gases present will also determine the decontamination and evacuation steps required.

The contaminated area will consist of two parts, particularly if persistent vesicant agents are used:

1. An area of liquid contamination, which acts as a source of vapor production and will require decontamination.

2. An area of dangerous vapor concentrations, the size and shape of which will be determined by the source of vapor evolution, nature of the gas, temperature, terrain, wind velocity, and other factors. In this area, people will become casualties unless they take proper individual precautions or unless evacuation can be carried out quickly.

DETERMINATION OF THE PRESENCE OF WAR GAS

SUBJECTIVE OBSERVATIONS

The presence of odors characteristic of the known chemical warfare agents or of strange new odors unexplainable under the existing conditions is presumptive evidence that a war gas is present. The usual rules for smelling should be observed, namely:

1. Do not inhale deeply. Sniff, since smelling is done by the nose alone.

2. First smell, then reflect.

3. After smelling, breathe out strongly through the nose several times. Do not make a new odor test until the effects of the last have disappeared. Repeated smelling dulls the perception.

4. Smoking before making an odor test is not advisable, since smoking dulls the perception. However, after a thorough reconnaissance by smelling, a smoking test may be made, since phosgene, hydrogen cyanide, and hydrogen sulfide are readily recognized by their disagreeable smoke-taste reaction.

5. If the odor is unfamiliar, associate each perception with a descriptive adjective or familiar odor in order to facilitate future recognition. A thing is odorless only when no perception is sensed.

Studies in detection by odor have shown that some persons have exceedingly poor perception, while others have very sensitive reactions to certain types of odors; however, a person may be sensitive to one type of odor and not to another. By proper training, certain persons who have sensitive odor perception can become exceedingly expert at detecting very small amounts of odoriferous substances.

It cannot be emphasized too strongly that it is essential for the odors of all common war gases to be learned thoroughly so that recognition will be instantaneous. A periodic review of the ability to recognize war gas odors by use of a sniff set prepared with the real agents is strongly recommended. It should be realized, however, that all war gases do not have distinct odors, e. g., adamsite, diphenylchlorarsine and the nitrogen mustards, so that odor alone cannot be relied upon for detecting gas. Furthermore, the possibility that the enemy might mix the agents or disguise odors should be borne in mind.

Certain visual indications may be of help in determining the presence of gas. If a bomb or shell has been dropped, the size of the crater and area of destruction will be almost negligible if the principal charge is a war gas rather than a high explosive. The usual gas bomb will produce no significant crater as contrasted to a crater 18-25 feet in diameter for a 250 kg. (550 lb.) high explosive bomb.

The presence of a cloud, smoke, or oily mist should be regarded with suspicion. Chlorine produces a yellow or yellowish-green cloud. Phosgene or diphosgene in strong concentrations gives a white cloud. High concentrations of the sternutators produce visible smokes. Liquid vesicants when released as a spray will give an oily mist, which can be recognized best by viewing toward a source of light. Care should be taken not to look upward or to otherwise expose oneself to falling vapor.

Any pools, drops of liquid, or wet oily ground should be noted. If a spray is suspected, observe all flat, open surfaces for the presence of dark-colored liquids indicative of vesicants or liquid lacrimators. On dry, porous surfaces (brickwork, cement, stonework), such liquids are rapidly absorbed, leaving a dark oily stain. On dry earth or roads, they appear as wet oily patches, while on wet surfaces, they spread and give a slightly iridescent effect similar to that produced by gasoline or oil. These liquids rapidly penetrate wood, especially along the grain, but remain as liquid drops and splashes on painted surfaces for a considerable time. They do not penetrate metals, glass, glazed tile, etc., and remain in droplet form on green foliage and grass for some time. In water, the bulk sinks to the bottom, but a thin, somewhat iridescent film remains on the surface.

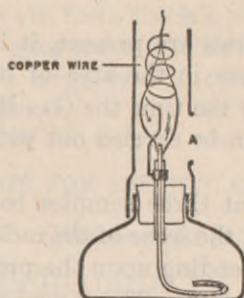
CHEMICAL METHOD

Chemically, it is possible to obtain evidence for the presence of war gases by means of the Beilstein test. This test is used to detect minute quantities of the halogens, although cyanides give the same reaction. Since most of the existing war gases contain halogen (or cyanide), the presence of halogen (or cyanide) vapor is indicative of danger. It is possible, though, that halogen may be present in

a sample or in the atmosphere but not in the form of a war gas; hence specific tests must also be carried out.

The Beilstein test is dependent upon the production of a green color when halogen or cyanide vapor reaches a flame in which a copper wire is heated. The color is due to the volatilization of copper salt in the flame. Because this test is so sensitive, extreme care must be exercised to keep the wire free from any traces of halogen. The wire should be heated in halogen-free air until no color is seen before making a test.

A convenient method for applying this test is by means of an alcohol lamp equipped with a special hood. The construction is illustrated below.



This hood allows use of the lamp under windy field conditions. A sample to be tested may be placed at A. The mild draft produced by the lamp draws the vapors into the flame, making the test very sensitive. The production of a green-colored flame when the lamp is placed in a suspected area is evidence of halogen or cyanide, hence of the possibility of war gas. Such an arrangement of the lamp allows its use either in testing the air at an incident or for later investigation of samples which have been taken.

SAMPLING

In general, it is best to remove a sample from the contaminated area and return to safety before applying tests. Approach to the gassed area should be made from the upwind side. Samples should be taken as quickly as possible while the sampler avoids personal contamination.

The type of samples taken will depend upon the particular situation. If liquid has been used, an excellent sample can be obtained by absorbing a droplet on a piece of filter paper, or by removing contaminated water or soil. In general, any contaminated material will serve satisfactorily as a sample, for example, contaminated bomb fragments, or leaves, grass, gravel, brickwork, wood, twigs, etc.

Samples of air should also be taken in certain cases. This can be accomplished by aspirating air (near the ground in the grossly contaminated area) through a small tube of activated, granular silica gel. Aspiration of the air can be carried out most conveniently by means of a rubber bulb; however, other devices may be used. One method is to use the suction produced by emptying a container of liquid. Another method is to utilize the suction produced by the engine of an idling automobile, in which case connection should be made to the hose attached to the windshield wiper. Also, a small bicycle pump with the valves reversed can be used to pull air through a silica gel tube. Samples of air should be taken in every case of suspected nonpersistent gas and in the case of persistent gas when liquid samples are difficult to obtain.

When nonpersistent gases are present, it is advisable to carry out rapid indicator paper tests in the area of high vapor concentration if any such area exists by the time the Gas Reconnaissance Agent arrives. This obviously can be carried out with greater safety if vesicants are not present.

It is recommended that three samples be obtained. One sample may be used for testing at the scene of the incident or be sent to the gas detection laboratory, depending upon the procedure of the particular local gas defense organization. The second sample should be held in reserve. The third sample is for analysis by War Department facilities. Reference should be made to the Office of Civilian Defense Circulars, Protection Series No. 17 and No. 26, for details concerning labeling and disposal of it.¹

Treatment of Samples

The type of sample taken will determine how it should be treated. Samples of liquid absorbed on filter paper or contaminated dirt or gravel may be used directly. Samples, particularly those containing interfering impurities, may be extracted with a suitable solvent, for example, acetone, benzene, ether, or ligroin, and after decantation from any solid present, the extract can be used as the test material. Ligroin has been found to be especially suitable for extraction, although ether possesses the advantage of being readily removed by evaporation.

A sample adsorbed on silica gel may be used without further treatment after removal from the containing tube (be sure to remove grains only from end of tube where gas entered) or may be extracted with a suitable solvent.

¹ At the present time War Department facilities are available only at the Massachusetts Institute of Technology and Edgewood Arsenal.

Reactions may also be carried out directly in the silica gel tube. For example, addition of the aniline hydrochloride reagent directly into the tube will produce the color characteristic of chlorine (if present) on the silica gel grains. With careful technique, two reagents may be introduced successfully from opposite ends of the tube.

Samples of contaminated earth, leaves, twigs, etc., may be used as a source of vapor evolution for adsorption of this vapor on silica gel. Air from the sample can be aspirated through an adsorption tube and the tests carried out as described above when they are of sufficient sensitivity. This procedure is especially convenient when the sample contains impurities which might obscure the color expected.

When tests are to be carried out on sensitized papers, a convenient procedure is to aspirate air from the sample (by means of a rubber bulb) through a small glass tube containing the test paper. This assures the passage of a large volume of air containing gas vapor directly over the test paper.

TESTS FOR SPECIFIC GASES

Chlorine (CL)

1. ANILINE HYDROCHLORIDE.

Reagent: 2 ml. of aniline, 8 ml. of concentrated hydrochloric acid diluted with 40 ml. of water.

Use: Filter paper dipped into the reagent on exposure to chlorine changes from colorless to red and later to blue.

Chemistry: Oxidation of aniline to various dyes, the end product of which is nigrosine.

Sensitivity: No data.

Limitations: Any strong oxidizing agent interferes. Bromine is, however, negative. Nitrogen dioxide slowly gives a green color.

2. STARCH-IODIDE PAPER.

Reagent: A paste of 2.5 g. of soluble starch in 5 ml. of cold water is poured into a liter of boiling water and the boiling is continued for 5 minutes. After the addition of 5 g. of potassium iodide, the solution is cooled. Filter papers dipped into this liquid and dried are stored in a brown bottle.

Use: For most sensitive results, the paper is moistened before use. Chlorine changes the paper from colorless to blue.

Chemistry: Liberation of iodine, which gives the typical blue color with starch.

Sensitivity: 0.014 mg. chlorine per liter (4 p. p. m.) in 3 to 5 seconds, just over the threshold for irritation.

Limitations: Bromine and nitrogen dioxide give a blue color.

Phosgene (CG)

1. HARRISON'S REAGENT.

Reagent: 5 g. of p-dimethylaminobenzaldehyde and 5 g. of colorless diphenylamine are each dissolved in 50 ml. of carbon tetrachloride and mixed. Filter papers are dipped immediately into this solution and dried. The papers are stored in tightly sealed brown bottles.

Use: The dry paper when exposed to phosgene changes from colorless to yellow.

Chemistry: Unknown, but may be dependent upon hydrogen chloride liberated.

Sensitivity: 0.004 mg. per liter or 0.9 p. p. m.

Limitations: Hydrogen chloride, bromine, chlorine, diphosgene, triphosgene, and lewisite give a yellow color. Nitrogen dioxide gives a yellow to green color.

2. NITROSO REAGENT.

Reagent: Two solutions are prepared and retained in separate bottles: (a) 0.25 g. of 1,2,4-nitrosodiethylaminophenol is dissolved in 50 ml. of benzene, (b) 0.20 g. of m-diethylaminophenol is dissolved in 50 ml. of benzene. The 1,2,4-nitrosodiethylaminophenol cannot be purchased from the usual supply houses, but can be readily prepared by the following procedure: 0.50 g. of m-diethylaminophenol (from the Eastman Kodak Co.) is dissolved in 15 ml. of water containing 1 ml. of concentrated hydrochloric acid and cooled to 0° C. A 0.22 g. portion of sodium nitrite is dissolved in 5 ml. of water and added to the phenol solution in small portions. The additions are made below the surface of the liquid, and the temperature is maintained at 0° C. at all times. This addition requires about 20 minutes. The mixture is made nearly neutral by the addition of a solution of 2 g. of sodium acetate in 5 ml. of water, and then extracted with 50 ml. of benzene. The separated benzene layer is filtered through a dry filter paper and is ready for immediate use as reagent (a).

Use: Equal parts of solutions (a) and (b) are mixed and applied to a piece of filter paper. Exposure of this paper to phosgene changes the color from brown to green.

Chemistry: Formation of a colored "Michler's" ketone.

Sensitivity: 0.0008 mg. per liter or 0.2 p. p. m.

Limitations: Specific for phosgene. The nitroso solution is somewhat unstable and must be renewed every 3 to 4 months.

Di- and Tri-Phosgene (DP, TP)

These two gases give the same yellow color with Harrison's reagent as phosgene but do not respond to the nitroso reagent.

Chloropicrin (PS)

1. RESORCINOL TEST.

Reagents: 10 percent alcoholic potassium hydroxide, resorcinol crystals.

Use: (a) To a drop of liquid, add 3 ml. of the alcoholic alkali, boil for 30 seconds, add a few crystals of resorcinol and heat again. The production of a red colloidal suspension which may become a heavy red precipitate indicates chloropicrin. (b) Bubble vapor through 3 ml. of alcoholic alkali for 2 minutes and proceed as usual. Or heat resorcinol and alcoholic potassium hydroxide in an evaporating dish in an atmosphere of chloropicrin vapor. A red ring appearing at the edge of the liquid indicates a positive reaction.

Chemistry: Production of a triphenylmethane dye.

Sensitivity: No data.

Limitations: Chloroform gives a similar red color.

2. DIPHENYLAMINE TEST.

Reagents: (a) 2 g. of barium diphenylamine sulfonate in 100 cc. of water, (b) concentrated sulfuric acid.

Use: Bubble vapor through 1 ml. of concentrated sulfuric acid. Add 2-3 drops of water to the acid, allow to stand one minute, and add 2 drops of the diphenylamine reagent. Chloropicrin gives a blue color.

Chemistry: Formation of nitrous acid from chloropicrin by hydrolysis. The nitrous acid gives the typical blue color with diphenylamine.

Sensitivity: 0.002 mg. or 0.3 p. p. m. for 1 liter of air.

Limitations: Oxides of nitrogen, stannic chloride, and titanium tetrachloride interfere.

3. NITRITE TEST.

*Reagents:*² (a) Dissolve 1 g. of sulfanilic acid in hot water, cool, and dilute to 100 ml. (b) Boil 0.5 g. of alpha-naphthylamine hydrochloride in 100 ml. of water kept at constant volume for 10 minutes. Decolorize with activated charcoal, if necessary. (c) Sodium ethylate solution made by dissolving 3 g. of sodium in 100 ml. of alcohol. (d) Glacial acetic acid.

Use: The sample is extracted with 1-2 ml. of sodium ethylate solution which is then acidified with glacial acetic acid. To this is added 1 ml. of alpha-naphthylamine solution. Development of a rose or pink color indicates the presence of chloropicrin.

Chemistry: Sodium ethylate decomposes chloropicrin with the formation of nitrite. The nitrite diazotizes the sulfanilic acid which couples with alpha-naphthylamine to form a dye.

² Reference should be made to a modification of this reagent by Shinn, M. B., Ind. Eng. Chem., Anal. Ed. 13, 33 (1941).

Sensitivity: 0.001 mg. or 0.14 p. p. m. for 1 liter of air.

Limitations: Nitrites.

Hydrogen Cyanide* (AC)

1. BENZIDINE-COPPER ACETATE TEST.

Reagent: (a) Dissolve 2.86 g. of cupric acetate monohydrate in water and dilute to 1 liter. (b) Dilute 475 ml. of saturated benzidine acetate solution with 525 ml. of water.

Use: Filter paper should be dipped in an equal mixture of (a) and (b) and exposed to the suspected vapor. Production of a blue color indicates hydrogen cyanide.

Chemistry: Unknown.

Sensitivity: 0.020 mg. per liter or 16 p. p. m.

Limitations: No data.

2. METHYL ORANGE-MERCURIC CHLORIDE TEST.

Reagent: Papers are prepared by dipping them in a solution of 2.5 g. of mercuric chloride, 0.60 g. of methyl orange and 50 ml. glycerol in 750 ml. of water and then drying. The papers are stored in a brown bottle.

Use: Exposure of the dry paper to hydrogen cyanide produces a pink to red color.

Chemistry: The hydrogen cyanide reacts with the mercuric chloride to form insoluble mercuric cyanide and hydrochloric acid. The acid changes the methyl orange to red.

Sensitivity: No data.

Limitations: Any strong acid will produce the red color. Hydrogen sulfide gives a black color.

Arsine* (SA)

MERCURIC BROMIDE TEST.

Reagent: (a) Standard mercuric bromide papers purchasable from supply houses (see Gutzeit test for arsenic), (b) aqueous lead acetate, and (c) cotton are needed.

Use: Suspected vapor is aspirated through a small glass tube containing a cotton plug moistened with lead acetate, followed by a mercuric bromide paper. The production of a yellow to brown color on the paper indicates arsine present.

Chemistry: Arsine combines with mercuric bromide to form various brown complexes. The lead acetate is used to remove any hydrogen sulfide present, which would form black mercuric sulfide.

Sensitivity: 0.002 mg. of arsine or 0.6 p. p. m. for 1 liter of air.

Limitations: Stibine.

* This substance has been included since British publications on war gas identification have indicated it as a possible agent in the event of an attack.

Mustard Gas (H)

1. SODIUM IODOPLATINATE.

Reagent: (a) A solution of sodium iodoplatinate is made by adding 1 ml. of a 5 percent platinum chloride solution to 5.3 ml. of a 5 percent sodium iodide solution and diluting to 180 ml. with water. The reagent should be left 24 hours before use. (b) A starch solution is prepared by rubbing 1 g. of soluble or arrowroot starch to a smooth paste with cold water and adding to 200 ml. of boiling water. After cooling, 2 drops of toluene are added as a preservative.

Use: Vapor is aspirated through a tube containing glass wool impregnated with 5 percent aqueous acetic acid. To the tube is then added 3 drops of sodium iodoplatinate followed by 2 drops of starch solution. The formation of a blue color indicates mustard.

Chemistry: The platinum is reduced to a valence of plus two with the liberation of free iodine, which gives the typical blue color with starch.

Sensitivity: 0.005 mg. of mustard or 0.7 p. p. m. for 1 liter of air.

Limitations: Chlorine, nitrous fumes, and other oxidizing agents also give a positive result, while the arsenical vesicants in high concentrations tend to bleach the iodoplatinate.

2. YABLICH'S REAGENT.

Reagent: 1 g. of selenium dioxide (or its equivalent of selenous acid) is dissolved in 100 ml. of 50 percent aqueous sulfuric acid.

Use: Vapor is bubbled through 2 ml. of the reagent for a few minutes, or a small amount of a solid sample may be added to the reagent. The mixture is warmed nearly to boiling. The production of a red color or precipitate indicates mustard.

Chemistry: Reduction of selenium to the free element.

Sensitivity: 0.005 mg. per liter or 0.7 p. p. m.

Limitations: Any reducing agent gives a positive result. Of these, hydrogen sulfide is the one most likely to be encountered.

Nitrogen Mustards (HN)

At the present time no methods for detecting these agents are available for public distribution. It may be stated, however, that they give an alkaline reaction with the usual acid-alkaline indicator papers. This test may also be carried out by aspirating vapor through silica gel impregnated with an acid-alkaline reagent. The presence of an alkaline material along with the absence of positive tests for other agents may be used as a preliminary method of detection.

The Arsenicals (DM, DA, CDA, L, ED)

1. GUTZEIT TEST FOR ARSENIC.

Reagents: (a) Concentrated hydrochloric acid, (b) zinc shot (arsenic-free), (c) 5 percent aqueous copper sulfate, (d) 5 percent aqueous

lead acetate, (e) standard mercuric bromide papers (purchasable from supply houses), and (f) cotton are needed.

Apparatus: A small bottle provided with a small diameter 3-inch vertical delivery tube is required.

Use: In the bottle is placed 5 g. of zinc and enough copper sulfate solution to cover it. After 1 minute the liquid is drained off and replaced by 10 ml. of water. The contaminated sample may be added directly to this mixture, or vapor may be aspirated through the water. In the meanwhile, a small plug of cotton moistened with aqueous lead acetate is inserted in the delivery tube followed by a mercuric bromide paper. Care should be taken to leave a little of the paper extending outside the tube to act as a blank. Then, 5 ml. of concentrated hydrochloric acid is added to the bottle and the delivery tube put into place. The presence of arsenic is indicated by a yellow to brown color on the mercuric bromide paper.

Chemistry: Arsenic in the sample is converted to arsine, which forms various brown complexes with mercuric bromide. The lead acetate is used to remove any hydrogen sulfide present, which would form black mercuric sulfide. The copper catalyzes the reduction, eliminating preliminary oxidation of the sample.

Sensitivity: 0.002 mg. of arsenic.

Limitations: Antimony.

Diphenylaminechlorarsine (Phenarsazine chloride) (Adamsite) (DM)

1. GUTZEIT TEST will show arsenic to be present.
2. BEILSTEIN TEST will be strongly positive.
3. SULFURIC ACID TEST.

Reagent: (a) Concentrated sulfuric acid, (b) 5 percent potassium nitrate.

Use: Adamsite in cold concentrated sulfuric acid produces a red color. Addition of a drop of potassium nitrate solution changes the color to purple or blue.

Chemistry: The reason for the red color is unknown. Addition of nitrate produces the typical blue color with diphenylamine, which is an impurity always present in adamsite.

Sensitivity: No data.

Limitations: Brombenzylcyanide gives a red color in sulfuric acid turning to a clear yellow with the addition of nitrate.

Diphenylchlorarsine (DA)

1. GUTZEIT TEST will show arsenic to be present.
2. BEILSTEIN TEST will give a strong test for halogen on solid or solvent extracted sample.
3. SULFURIC ACID will produce no color.

Diphenylcyanoarsine (CDA)

1. GUTZEIT TEST will show arsenic to be present.
2. SULFURIC ACID will produce no color.

Lewisite (L)

1. GUTZEIT TEST will show arsenic to be present.
2. BEILSTEIN TEST will be positive on vapor from the sample.
3. SULFURIC ACID will give no color.
4. SLOAT'S REAGENT.

Reagent: To 50 ml. of 20 percent aqueous sodium hydroxide is added 4 g. of arsenic trioxide. A second solution is prepared by dissolving 0.4 g. of copper acetate dihydrate in 10 ml. of water. Both solutions are heated to 85° C., then the latter is added to the former solution under constant stirring. The mixture is cooled rapidly in a cold water bath, whereupon the orange precipitate dissolves. The solution when diluted to 100 ml. is ready for use.

Use: Vapor may be aspirated over a paper freshly moistened in the reagent or the reagent may be added to silica gel grains which have been used to adsorb vapor. The production of a red color indicates lewisite.

Chemistry: The reagent is an alkaline solution of cuprous ions, which forms red copper acetylide with the acetylene liberated from lewisite.

Sensitivity: 0.01 mg. per liter or 1.1 p. p. m.

Limitations: Specific among the known war gases.

Ethylchlorarsine (ED)

1. GUTZEIT TEST will show arsenic to be present.
2. BEILSTEIN TEST will be positive on vapor.
3. SULFURIC ACID will give no color.
4. SLOAT'S TEST will be negative.
5. MERCUROUS NITRATE TEST.

Reagent: 5 percent aqueous mercurous nitrate solution.

Use: Aspirate vapor through 2 ml. of reagent. Ethylchlorarsine gives a white precipitate turning to gray in a few seconds.

Chemistry: Complex formation with reduction of mercury.

Sensitivity: 1 mg. or 128 p. p. m. for 1 liter of air.

Limitations: Lewisite gives a white precipitate turning to gray in 12 hours. Methylchlorarsine gives a gray precipitate at once.

General Test**PYRIDINE TEST OF HARGER.**

Reagent: (a) Pyridine of good quality, (b) concentrated ammonium hydroxide diluted with four volumes of water, (c) 10 percent

aqueous sodium hydroxide solution, and (d) 5 percent aqueous sodium cyanide are needed.

Use: 1 to 5 liters of vapor are aspirated through 2 ml. of pyridine. Any pyridine lost during this time is replaced. The liquid is divided into two portions. To one part is added 0.5 ml. of water followed by 3 drops of sodium cyanide solution. If chloropicrin is present, the fluid will assume a yellow color. To the second portion of the liquid is added 0.5 ml. of water and the mixture heated to boiling for about 20 seconds. If chloracetophenone or bromacetophenone is present, a yellow color develops. One drop of the ammonium hydroxide solution is now added. If chloracetophenone is present, the color will become more intense. By continuing the boiling with agitation to prevent bumping, any color due to chloracetophenone will mostly disappear after 1 to 2 minutes. The solution is allowed to cool for about 20 seconds and 2 drops of sodium hydroxide solution is added. If mustard is present, a yellow color will form. On standing, the yellow color due to mustard will gradually fade, the disappearance being more rapid if the fluid is heated.

Chemistry: Unknown.

<i>Sensitivity:</i> Chloracetophenone -----	0.01 mg.
Mustard -----	0.002 mg.
Chloropicrin -----	0.003 mg.

Scope: This test will detect three of the known war gases (four, including the vesicant amine of Ward), and most other agents will not interfere.

Suggested Scheme of Analysis

The following procedure is based on the assumption that the sample will contain only one chemical warfare agent. This may not be the case, but the scheme should still handle the analysis of many mixtures. If difficulty is encountered with mixtures, individual tests will have to be applied to confirm or eliminate each gas suspected of being present.

It is suggested that the Gutzeit test be started as soon as possible after a sample is taken. While this test is progressing, both the sulfuric acid test and Harrison's paper should be used. By that time, arsenic will have been found to be present or absent and the rest of the scheme can be followed accordingly. This procedure is adopted to save time.

With the foregoing information, the Gas Reconnaissance Agent should be able to determine whether or not gas has been used at an incident and, if so, what gases are present.

The problem remains to delimit the area contaminated.

If only nonpersistent gases are present, there will be no need for decontamination. However, if a nonpersistent gas is present in a

Scheme of Analysis for Testing of War Gases

TEST FOR ARSENIC

Arsenic Present L, DM, DA, ED, CDA Test with H ₂ SO ₄		Arsenic Absent H, PS, DP, CG, CL, BBC, CN Harrison's Reagent							
Negative L, DA, ED, CDA Sloat's Reagent		Positive DM	Negative H, PS, BBC, CN Test with H ₂ SO ₄			Positive DP, CG, CL Aniline HCl			
Negative CDA, DA, ED Beilstein on vapor* of HgNO ₃			Positive L	Negative H, PS, CN Pyridine-NaOH			Positive BBO	Negative DP, CG Nitroso	Positive CL Con- firm
Negative CDA, DA	Positive ED			NH ₄ OH	NaOH	NaCN		Neg. DP	
			CN	H Con- firm	PS Con- firm				
*CDA and DA are nonvolatile									

Tabulation of Individual Tests for War Gases

Name of test	Gas tested	Sensitivity	Interferences
Aniline hydrochloride	Chlorine	No data	Strong oxidizing agents.
Starch-iodide	do	0.014 mg. per liter	Bromine, nitrogen dioxide.
Harrison's reagent	Phosgene	0.004 mg. per liter	Hydrogen chloride, diphosgene, triphosgene, chlorine, bromine, lewisite, nitrogen dioxide.
Nitroso	do	0.0008 mg. per liter	Specific.
Resorcinol	Chloropicrin	No data	Chloroform.
Diphenylamine	do	0.002 mg.	Oxides of nitrogen, stannic chloride, titanium tetrachloride.
Nitrite	do	0.001 mg.	Nitrites.
Benzidine-copper acetate.	Hydrogen cyanide	0.020 mg. per liter	No data.
Methyl orange-mercuric chloride.	do	No data	Strong acids, hydrogen sulfide.
Mercuric bromide	Arsine	0.002 mg.	Stibine.
Sodium iodoplatinate	Mustard	0.005 mg.	Chlorine, nitrous fumes, oxidizing agents.
Yablich's reagent	do	0.005 mg. per liter	Reducing agents.
Gutzzeit	Arsenic	0.002 mg. of As.	Antimony.
Beilstein	All	Very sensitive	Bromide, fluoride.
Sulfuric acid	Adamsite	No data	None.
Sloat's reagent	Lewisite	0.01 mg. per liter	None.
Mercurous nitrate	Ethylidichlorarsine.	1 mg.	Lewisite, methylidichlorarsine.
Harger's pyridine test	Chloroacetophenone.	0.01 mg.	Most war gases do not interfere.
	Mustard	0.002 mg.	
	Chloropicrin	0.003 mg.	

secluded area, the size of the area in which dangerous concentrations remain upon the arrival of the Gas Reconnaissance Agent should be determined. The size of this area should be known so that people can be warned to take suitable protective measures or, if necessary, be evacuated.

The extent of such an area will have to be determined by smell, or, if in doubt, by air sampling followed by the usual tests outlined in this publication.

The presence of persistent vesicants, however, will require knowledge of the approximate area of liquid contamination as a guide in determining decontamination measures to be taken.

Determination of liquid vesicant contamination can be carried out by use of less specific tests which can be applied quickly and frequently. Several materials have been suggested for such tests. One of these is Sudan Red powder, which, when sprinkled over a suspected area, turns red on contact with liquid vesicant agents. Another method is the use of painted detector paper containing a pigment which becomes colored by the solvent action of the liquid. It is suggested that some mechanical device be used to facilitate the use of this paper. A long stick with a nail on the end to hold the test paper is simple and satisfactory.

To determine the completeness of decontamination, samples may be taken and tests carried out for the agent or agents found to be originally present. The Sudan Red powder or detector paper may be used, although it will probably be more satisfactory to apply specific tests for the chemical warfare agents. If bleach or sodium hypochlorite has been used as the decontaminant, the presence of free chlorine may be taken as presumptive evidence that decontamination has been complete. Tests for free chlorine can be made by using aniline hydrochloride or starch-iodide paper as outlined on page 6.

Further information concerning the identification of chemical warfare agents may be found in the following references:

1. Ministry of Home Security, Brit., "The Detection and Identification of War Gases." (American Edition) Chemical Publishing Co. Inc., Brooklyn, N. Y. (1940).
2. Jacobs, "War Gases." Interscience Publishers, Inc., New York, N. Y. (1942).
3. Sartori, "The War Gases," D. van Nostrand Co., Inc., New York, N. Y. (1940).
4. Studinger, *Chem. and Ind.*, 56, 225 (1937).
5. Hoogeveen, *Chem. and Ind.*, 59, 550 (1940).
6. Degand, *J. Pharm. Belg.*, 21, 895 (1939).
7. Maricq, *J. Pharm. Belg.*, 21, 749 (1939).
8. Stainsky and Taylor, *Analyst*, 66, 44 (1941).
9. Cox, *Analyst*, 64, 807 (1939).
10. Hickey and Hanley, *J. Chem. Ed.*, 19, 360 (1942).
11. Bradley, *Chem. Eng. News*, 20, 893 (1942).

