

**METHODS OF TESTING GAS MASKS AND ABSORBENTS<sup>1</sup>**By A. C. FIELDNER,<sup>2</sup> G. G. OBERFELL, M. C. TEAGUE AND J. N. LAWRENCE

Received March 17, 1919

**INTRODUCTION**

The development and manufacture of gas masks and absorbents depend to a large degree upon accurate and properly designed tests which simulate, as far as possible, actual conditions under which the mask is to be used. These tests are subdivided into three groups:

1—Absorbent tests, which are used in the development and control of absorbent production.

2—Machine canister tests, used in the development of canisters and in controlling the uniformity of filling the canisters at the factory.

3—Actual man tests, in which men wear the masks in gas chambers to determine the life of the canister, maximum protection, leakage, comfort and fit of the facepiece.

**ABSORBENT TESTING**

The ultimate test of the value of an absorbent is, of course, the actual man test of the complete mask. However, for the development and control of absorbents a much more precise test is made by placing a definite quantity of absorbent in a glass tube at approximately the same depth as that used in the final canister and at a rate of flow equal to that of a man doing moderate work. For the present army canisters these fixed dimensions of the standard tube test are:

- 10 cm. depth of layer
- 2 cm. diameter of tube
- Rate of flow of 500 cc. per min. per sq. cm. cross-sectional area
- 8-14 mesh material
- 50 per cent relative humidity
- As nearly as possible a room temperature of 25° C.

In general, the gas concentrations are 1000 p. p. m. by volume, assuming the material to be a perfect gas at 25° C. and 760 mm.

The service time is taken as the time in minutes to 99 per cent efficiency.

The absorption value of any material may be considered under two heads: 1—Activity; 2—Capacity.

The tube test can be varied to throw emphasis upon either one of these factors, although it does not in any case completely separate them. The capacity of the absorbent is usually measured by making the tube test against a rather dilute mixture of gas and air, so that the service time is long. For soda lime, capacity is measured by testing against 1000 p. p. m. of trichloromethylchloroformate, whereas activity is measured by testing it against 10,000 p. p. m. phosgene, activity being measured by the ability of an absorbent to take up high concentrations of gas. It is also possible to secure relative figures on activity and capacity by considering the 99 per cent point as a measure of activity and a comparison at a lower point, say 80 per cent, as a measure of capacity. In the production of absorbents for industrial purposes, it is, of course, advisable to test the material against two or three representative gases such as may be met with in the industries. One of these should be a weak

sulfur dioxide; another, a rather stronger acid gas such as chlorine or phosgene; and for materials that depend entirely upon adsorption, such as charcoal, the gas should be one that is taken up by adsorption only, such as chlorpicrin or carbon tetrachloride.

Another important property of absorbents that are used in masks which are subject to considerable handling and rough usage, is hardness. A special method will be described for making this test as well as the more important tube tests.

**STANDARD METHODS FOR TESTING ABSORBENTS****DETERMINATION OF MOISTURE**

A 5-g. sample of the material is heated 2 hrs. at 150° C. in a Freas electric oven. The covered capsule or dish is cooled in a desiccator and weighed to the nearest milligram. The loss in weight is moisture.

**SCREEN ANALYSIS**

All screen analyses are made with 10-in. Tyler standard screen-scale screens, shaken in nests on the Ro-tap shaker.

Soda lime is screened by placing a 400-g. representative sample in the top screen and shaking 5 min.

Charcoal is screened by placing a 500-g. sample on the top screen and shaking 10 min.

**DETERMINATION OF HARDNESS OR RESISTANCE TO ABRASION<sup>1</sup>**

A 70-g. sample of the material is screened on 8- and 14-mesh screens for 5 min. on a standard Ro-tap machine. Tyler standard screen-scale screens, of 10 in. diameter are used.

Fifty grams of this material which have passed through the 8-mesh and are retained on the 14-mesh screen, are weighed out and placed in the bottom pan of the Ro-tap machine. Fifteen steel ball bearings, 1/2 in. in diameter, and 15 balls of 3/8 in. diameter are then placed carefully on top of the absorbent material and the pan is covered and shaken for exactly 30 min. on the Ro-tap machine, at a speed of 1750 r. p. m. of the driving shaft. The bottom of the pan should be slightly concave upward and have no dents or irregularities.

At the end of 30 min. the contents of the pan are carefully brushed into a 20-mesh screen, the ball bearings dusted off and removed, and the screen shaken for 3 min. on the Ro-tap machine. The weight of absorbent remaining on the screen multiplied by two gives the hardness number.

The test with steel balls of the number and size given above is best adapted to materials ranging between 50 and 80 hardness. If the hardness is above 80, the dispersion is not sufficient to distinguish adequately between different degrees of hardness, and likewise where more than 50 per cent of the material passes through a 20-mesh screen, the cushioning effect of the large amount of fines decreases the severity of the test. Hence, in new types of absorbents a different scale may be required, which is preferably ob-

<sup>1</sup> Method devised by Major Robert E. Wilson of Defense Chemical Research Section.

<sup>1</sup> Published by permission of the Director of the Chemical Warfare Service, U. S. A.

<sup>2</sup> In charge Gas Mask Research Section, Research Division, Chemical Warfare Service, U. S. A.

tained by changing the size of the balls so that the average material of that type passing through the screen will be approximately 60 or 70 per cent.

A second hardness scale used in this laboratory for softer material consists in using 30 steel balls of  $\frac{3}{8}$  in. diameter.

#### DETERMINATION OF THE EFFICIENCY OF ABSORBENTS AGAINST VARIOUS GASES

The standard tube test methods described herein were developed at the American University Experiment Station for determining the absorption value of charcoal and soda lime under conditions closely approximating the actual service conditions of their use in gas mask canisters.

These methods have been in constant use in a number of different laboratories of the Chemical Warfare Service, and, when carefully followed, give closely comparable results.

Acknowledgment is due to a great many persons for improvements in various details of the test and especially to Capt. J. B. Dickson and the Coördinating Committee on Analytical and Research Methods,<sup>1</sup> for carefully standardizing the apparatus and methods.

**PRINCIPLE OF THE TEST**—The absorbent under test is filled into a sample tube of specified diameter (2 cm.) to a depth of 10 cm. by the standard method for filling tubes, and a standard concentration (usually 1000 or 10,000 p. p. m. by volume) of the gas in air of definite (50 per cent) humidity is passed through the absorbent at a rate of 500 cc. per sq. cm. per min. The concentration of the entering gas is determined by analysis. The length of time is noted from the instant the gas-air mixture is started through the absorbent to the time the gas or some toxic or irritating reaction product of the gas begins to come through the absorbent, as determined by some qualitative test. Quantitative samples of the outflowing gas are then taken at known intervals and from the amount of gas found in the sample the per cent efficiency of the absorbent at the corresponding time is calculated.

Per cent efficiency =

$$\frac{\text{P. p. m. entering gas} - \text{p. p. m. effluent gas}}{\text{P. p. m. entering gas}} \times 100$$

These efficiencies are plotted against the minutes elapsed from the beginning of the test to the middle of the sampling period corresponding to that efficiency point. A smooth curve is drawn through these points and the efficiency of the absorbent is reported as so many minutes to the 100, 99, 95, 90, 80, etc., per cent efficiency points.

**APPARATUS**—Like most gas analysis apparatus, tube testing boards are susceptible of infinite variation in detail, and may combine any number of absorption tubes on one board. Unless a laboratory has a very large volume of work a two-tube board furnishes sufficient testing capacity for a single operator. The one described is the result of much experience in de-

signing tube test units and embodies the best features of the various boards at this station.<sup>1</sup>

The board unit itself, as shown in Fig. I, may be used for any gas or vapor by simply attaching to the gas supply line a tank of the compressed gas in question, or, if the gas cannot be stored under sufficient pressure, by attaching one of the three types of gas supply devices shown in Fig. II.

**DESCRIPTION OF STANDARD TWO-TUBE BOARD AS USED FOR LIQUEFIED GASES IN CYLINDERS**—The standard two-tube board shown in Fig. I is 31 in. wide and 30 in. high. It is made of 1 in. soft pine lumber and has a base of the same material projecting 8 in. at the front and rear and 6 in. to the left. The glass tubing is 6 to 8 mm. outside diameter, and the stopcocks are of liberal bore, in order to reduce resistance to flow to a minimum. Stopcocks T,T should have a bore of at least 3 mm.

This apparatus is operated entirely under pressure. Air from a pressure line enters the apparatus through the cock W, and bubbles through the humidifying bottles, 2, 3, and 4, containing sulfuric acid and water of the proper specific gravity to give the desired humidity (usually 50 per cent). Bottles 1 and 5, containing glass wool, serve as traps. From the humidifiers the air is conducted to the mixing chamber M at the top of the board, where it is mixed with the toxic gas from supply tank A, and then passes through a glass tube and T to the two sample tubes, P,P, and out through the effluent analyzing bottles to waste. The rate of flow of the gas-air mixture through each sample tube is accurately regulated to 500 cc. per sq. cm. per min., by the stopcock U, and the flow meter Q. The concentration of the entering gas is taken by directing the gas mixture into gas washing bottles, 8 and 9, by proper adjustment of stopcocks V and S. The gas mixture can also be directed into the waste line on the back of the board by reversing stopcock S. The effluent gas from the sample tubes P may be passed alternately to analyzing bottles, 6 or 7 on the left, and 10 or 11 on the right, by the large bore (3 mm.) three-way stopcocks T,T. R and R are water manometers for measuring the pressure drop through the absorbent.

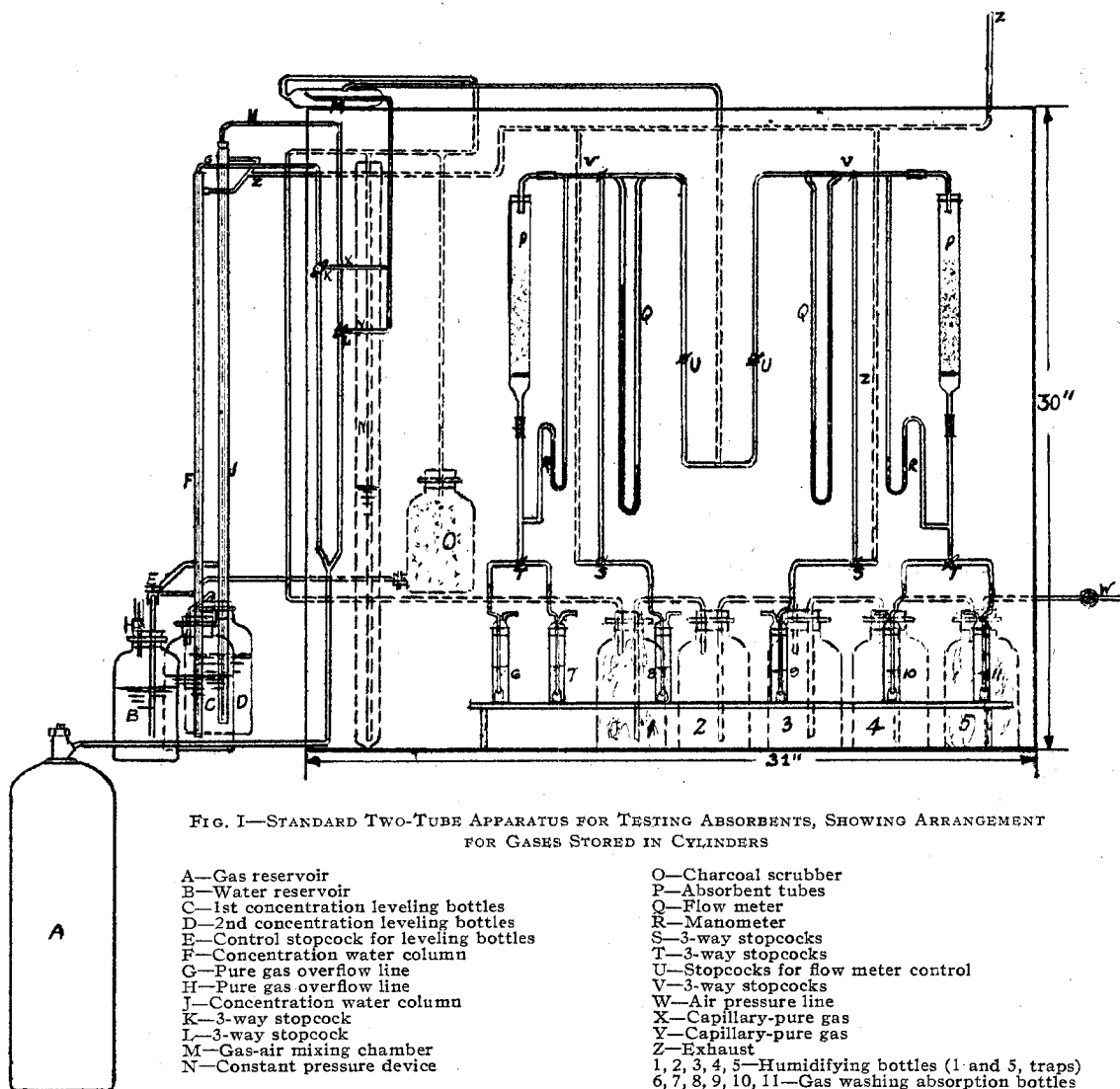
Fluctuations in the air pressure line are controlled by the constant pressure water column N, which is connected to the air line between the humidifying bottles and the mixing chamber.

The toxic gas flow from cylinder A to the mixing chamber M is automatically controlled and maintained constant by a very ingenious automatic compensating flow meter, designed by R. P. Mase, of this laboratory.<sup>2</sup> This flow meter, when once set for a given concentration, as for example 10,000 p. p. m., will automatically deliver this concentration in the mixing chamber M to within 0.5 per cent, regardless of any pressure fluctuations that may take place in the mixing chamber due to increase of resistance in the absorbent tube or in the gas washing bottles.

<sup>1</sup> This board does not include temperature control which may be required if the room temperature cannot be kept fairly constant.

<sup>2</sup> THIS JOURNAL, 11 (1919), 294.

<sup>1</sup> Maj. A. C. Fieldner, Maj. R. P. Rose, Maj. R. E. Wilson, Capt. John B. Dickson, Lt. W. N. Watson, and Lt. J. F. Walsh.



In the apparatus shown in Fig. I, there are two automatic flow meters arranged in parallel between gas supply tank A and mixing chamber M, (1) the system C, F, G, K, X, for concentrations of 1000 p. p. m., and (2) the system D, J, H, L, Y, for concentrations of 10,000 p. p. m. By this double arrangement the concentration can be instantly changed from one to the other, or the combination may be used for binary gas mixtures. Since the operation of the pair of flow meters is identical it will be sufficient to describe the D, J, H, L, Y system.

**PRINCIPLE AND DESCRIPTION OF AUTOMATIC COMPENSATING FLOW METER**—A constant flow of toxic gas is maintained through the very small constricted capillary tube Y, by imposing a constant differential pressure on the inlet and outlet. This differential pressure is the height of water column from bottom end of tube H to water level in bottle D. The cross section of bottle D is chosen sufficiently large in comparison to cross section of tube J so that no rise of water in tube J will change height of water level in D appreciably.

The quantity of gas transmitted by the automatic flow meter is determined by the size of the constricted

capillary Y and the water level in bottle C. These two factors must be determined by actual trials of concentration by analysis. The final exact adjustment of concentration is made by changing the water level in bottle C by applying suction or air pressure to reservoir bottle B and allowing the liquid to flow through stopcock E in the proper direction to give the desired level.

When the board is in operation, 3-way stopcock; L, is turned so that the gas flows through the constricted capillary Y and at the same time an excess of toxic gas passes through tube H and bubbles out against the column of liquid in J and passes off to waste through the tube Z.

The column of water in J is held by the pressure of the air over the liquid in the level bottle D. This air space communicates with the main air line just ahead of the mixing chamber and, therefore, has the same pressure, practically, as at the outlet end of the constricted capillary Y. Hence, any change in pressure on the outlet of Y is immediately compensated by an equal change in pressure on the inlet end due to change in level of water column J which governs the inlet pressure; thus all pressure fluctuations in the

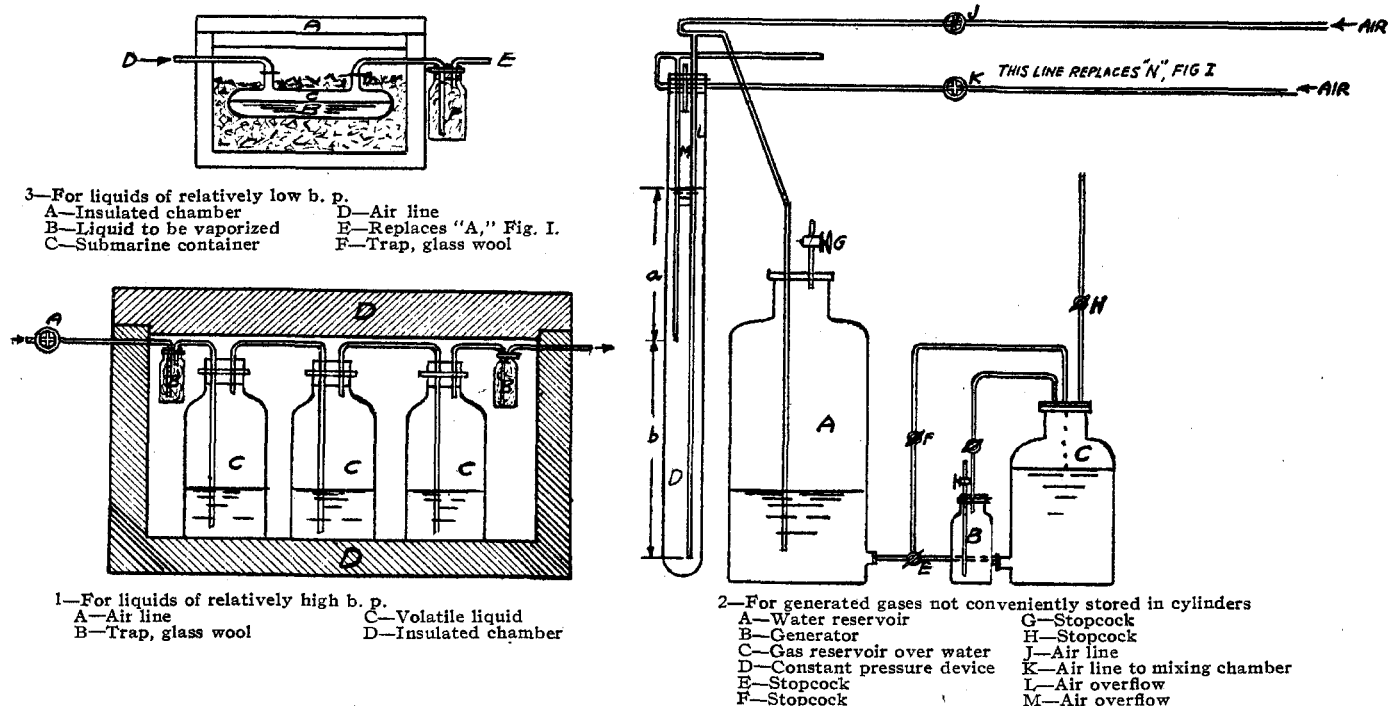


FIG. II—ACCESSORIES FOR FEEDING VARIOUS TYPES OF GASES AND VAPORS

line are instantly compensated and a constant and steady differential pressure is maintained over capillary Y, insuring a definite and uniform concentration. Charcoal bottle O prevents diffusion of gas into the main air line.

**MODIFICATION FOR LOW-BOILING LIQUIDS**—Liquids such as hydrocyanic acid are most conveniently vaporized by passing a slow stream of *dry* air over the surface, the container being packed in cracked ice and water at 0° C. A convenient form of such apparatus is shown in Fig. II-3. This container simply takes the place of the compressed gas tank. The air-vapor flow is regulated by the same automatic compensating flow meter shown in Fig. I.

**MODIFICATION FOR HIGH-BOILING LIQUIDS**—Vaporizing apparatus for liquids such as chlorpicrin, carbon tetrachloride, and trichloromethylchloroformate is shown in Fig. II-1. It differs from the previous apparatus only in that the liquid is placed in a five-bottle train. The three middle bottles contain the liquid and the two end bottles serve as traps. The entire train is packed in ice. The air, previously dried, is bubbled through the liquid and then through the automatic flow meter.

**METHOD OF FILLING TUBES**—The absorption tube and funnel (Fig. III-1) are supported in a vertical position with the funnel stem "centered" in the tube. The distance from the apex of the funnel to the upper layer of absorbent when the absorption tube is filled to the desired height should be 15 cm.

The entire sample of absorbent is poured on a large sheet of rubberized fabric and thoroughly mixed by rolling the sample. This may be done by lifting opposite corners of the fabric alternately. Smooth out the sample on the fabric and take portions with a spoon at regular intervals of space over the entire

surface of the sample so as to insure having a representative portion for test.

The absorbent is poured from the spoon, a few grains at a time, so that no grains have a free fall through the orifice of the funnel. The time for filling a 2.00 cm. tube to a height of 10 cm. should be not less than 1½ min. The tube should not be tapped at any time. Connect the tube to a strong suction and draw air through it at least 5 times to remove dust. In removing dust from the absorbent the stopcock C is turned on quickly for 1 or 2 sec. and quickly closed.

**PROCEDURE IN MAKING TESTS**—Having obtained the desired concentration and diverted the flow of gas-air mixture through the stopcocks V and S to the waste, the absorption tube, filled as previously described, is placed in position, the time noted, and the gas-air mixture passed through the absorbent by proper adjustment of stopcocks V and T. The proper amount of gas-air flow is regulated by the stopcock T. The bottle 6 or 7 may contain the solution or reagent to be used as the qualitative test. Upon noting a qualitative test the time is again recorded and the gas-air flow diverted alternately, generally for 5-min. intervals through bottles 6 and 7, which now contain solutions for quantitative analysis of the effluent gas. The efficiency of the absorbent is calculated as follows:

Percentage efficiency =

$$\frac{\text{P. p. m. entering gas} - \text{p. p. m. effluent gas}}{\text{P. p. m. entering gas}} \times 100$$

The time elapsed to any efficiency point is recorded as time in minutes from the beginning of the test to the middle of the sampling period corresponding to that efficiency point. A time efficiency curve with time in minutes as abscissa and percentage efficiency

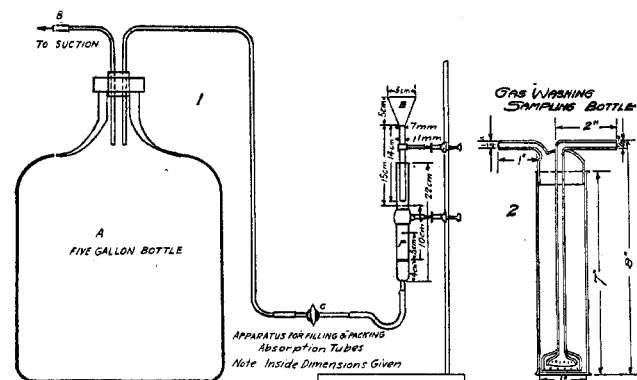


FIG. III—APPARATUS FOR FILLING SAMPLE TUBES

as ordinate is drawn as a smooth curve through the points whose values were obtained by analysis of the effluent gas. The time in minutes read from this curve and corrected to the nominal concentration is reported for the 100, 99, 95, 90, and 80 per cent efficiency points.

#### METHODS OF ANALYSIS

Concentrations of entering and effluent gas are determined by directing the entire gas-air stream (1571 cc. per min.) through 1 or 2 gas washing bottles in series, each containing 25 or 50 cc. of the proper absorbing solution, for an exact period of time—2 min. for entering gas and 5 or 10 min. for effluent gas. Since the gas-air stream has a volume of exactly 1571 cc. per min., the total sample volume becomes 1571 multiplied by the number of minutes that the gas stream was passed through the absorption bottle. The standard solutions are usually made up to an exact normality, so that 1 cc. of the solution is equal to exactly 100 or 1000 p. p. m. of the gas at 25° C. and 760 mm. for a 1-min. sample, *i. e.*, 1571 cc. Actually, the sampling time is usually 2, 5, or 10 min., and the result is then divided by the number of minutes of sampling. The absorbent solutions used for various gases are briefly described below.

#### CHLORINE

**QUALITATIVE**—Two per cent potassium iodide solution to which starch has been added.

**QUANTITATIVE**—A 4 per cent potassium iodide solution is used for entering concentrations and a 2 per cent potassium iodide solution for effluent concentrations. The gas-air mixture is aspirated through the potassium iodide solution and the iodine thus liberated is titrated with 0.1286 *N* sodium thiosulfate. 1 cc. of 0.1286 *N* sodium thiosulfate equals 1000 p. p. m. chlorine (by volume 25° C. and 760 mm.) for a 1-min. sample.

#### PHOSGENE

**QUALITATIVE**—The use of moist Congo-red paper as an indicator depends upon the presence of hydrochloric acid (resulting from hydrolysis of carbonyl chloride) in the effluent gas and the consequent change in color of the paper from red to blue. The phosgene test paper, which also responds to chlorine and hydrochloric acid, is prepared as follows:

Dissolve 5 g. of *p*-dimethylaminobenzaldehyde and 5 g. of diphenylamine in 100 cc. 95 per cent alcohol. Dip filter paper in the solution and dry in the air. When dry, keep the test paper away from light. These papers thus preserved do not deteriorate and will detect readily one part per million of phosgene. Both chlorine and hydrochloric acid act on the papers so that in testing for phosgene one must take precautions first to remove any chlorine or hydrochloric acid which may be present.

Another qualitative test solution which responds to both hydrochloric acid and phosgene is prepared by adding a few drops of potassium chromate and a few drops of neutral silver nitrate to alcohol. A salmon colored solution is obtained which is changed to yellow by presence of hydrochloric acid or phosgene.

**QUANTITATIVE**—The gas samples are absorbed in 50 cc. of alcoholic sodium hydroxide, prepared by dissolving 40 g. sodium hydroxide in 125 cc. of distilled water and adding this solution to 875 cc. of 95 per cent ethyl alcohol. The absorbent solution is neutralized with nitric acid (approximately normal), using phenolphthalein as an indicator, and the chloride ion is determined by titration with 0.1286 *N* silver nitrate, using sodium chromate as an indicator. 1 cc. 0.1286 *N* silver nitrate equals 1000 p. p. m. phosgene (by volume at 25° C. and 760 mm.) for a 1-min. sample.

#### SULFUR DIOXIDE

**QUALITATIVE**—A 2 per cent potassium iodide solution to which a few drops of starch-0.01286 *N* iodine has been added.

**QUANTITATIVE**—Entering concentrations are determined by passing the gas through two absorption bottles, the first of which contains an accurately measured amount (approximately 30 cc.) of 0.1 *N* iodine solution, and the second, 25 cc. of 2 per cent potassium iodide solution, and titrating the liberated iodine with 0.1286 *N* thiosulfate solution (1 cc. = 1000 p. p. m. sulfur trioxide).

Effluent concentrations are determined by absorbing the gas in 25 cc. of 3 per cent aqueous sodium hydroxide. This solution is first neutralized with hydrochloric or sulfuric acid, using phenolphthalein as indicator, then titrated with 0.01286 *N* iodine (1 cc. = 100 p. p. m. sulfur dioxide), using starch as indicator.

#### CARBON DIOXIDE

**QUALITATIVE**—Approximately 0.2 *N* barium hydroxide solution.

**QUANTITATIVE**—The gas is absorbed in 0.1286 *N* barium hydroxide solution. Titrations are made with 0.1286 *N* hydrochloric acid or oxalic acid of the same normality, using phenolphthalein as an indicator. 1 cc. equals 1000 p. p. m. (by volume at 25° C. and 760 mm.) for a 1-min. sample.

#### TRICHLOROMETHYLCHLOROFORMATE

**QUALITATIVE**—Methyl red has given the most satisfactory qualitative test. The color change is from yellow to red. The solution is prepared by adding 10 drops of a 5 per cent solution to 100 cc. of alcohol and then adding 1 cc. 0.2 *N* sodium hydroxide solution.

**QUANTITATIVE**—The gas is absorbed in 50 cc. of alcoholic sodium hydroxide solution. This solution is prepared by dissolving 40 g. of sodium hydroxide in 125 cc. of distilled water which is then added to 875 cc. of 95 per cent alcohol. 1 cc. 0.0257 *N* silver nitrate = 100 p. p. m. (by volume at 25° C. and 760 mm.) for a 1-min. sample.

#### CHLORPICRIN

**QUALITATIVE**—The gas is decomposed by passing it through a quartz tube heated to about 400° C. Chlorine, one of the decomposition products, is detected by means of a 2 per cent potassium iodide solution to which starch has been added.

**QUANTITATIVE**—The gas is absorbed in 50 cc. of a 1 per cent solution of sodium peroxide in 50 per cent alcohol. This solution is made up daily by adding sodium peroxide to ice water in the ratio of 2 g. of the peroxide per 100 cc. of water. Just prior to use, 25 cc. of this solution are diluted with 25 cc. of 95 per cent alcohol. The chloride-ion resulting from decomposition of chlorpicrin is titrated with 0.01929 *N* silver nitrate in the neutral solution by using sodium chromate as an indicator. 1 cc. of 0.01929 *N* silver nitrate equals 100 p. p. m. (by volume at 25° C. and 760 mm.) for a 1-min. sample.

#### HYDROCYANIC ACID

**QUALITATIVE**—The gas is passed through a 2 per cent sodium bicarbonate solution to which starch and enough iodine have been added to produce a visible blue. Decolorization of this solution shows the break point.

**QUANTITATIVE**—Samples of the gas are absorbed in a 2 per cent sodium bicarbonate solution and titrated with 0.01286 *N* iodine solution, using starch as an indicator. 1 cc. 0.0129 *N* iodine solution equals 100 p. p. m. (by volume at 25° C. and 760 mm.).

#### ACCELERATED TEST FOR CHARCOAL

The accelerated test differs from the standard long test only in concentration, rate, and relative humidity of the gas-air mixture and in end-point of test. The rate of 1000 cc. per min. is obtained by using with the standard two-tube apparatus a 1.41 cm. internal diameter absorption tube instead of a 2.00 cm. tube. The relative humidity of the gas-air mixture which is zero and the concentration which is about 7500 p. p. m. are obtained by drying the entire air supply to the apparatus with concentrated sulfuric acid and then passing it through chlorpicrin maintained at 0° C. by an ice-water mixture. The effluent gas is passed into the air supply of a gas burner which contains a copper wire spiral or coil so situated that the copper wire is subjected to all gradations of temperature. A new multiple opening type of burner has been developed. It consists of a flat top burner with a ring of small holes near the outside. A piece of No. 18 copper wire bent into the form of a ring is laid over these holes. A green coloration of the flame is taken as the qualitative test for chlorpicrin in the effluent gas mixture. The green flame appears at a concentration somewhat less than 35 p. p. m. The time in minutes (recorded to the nearest tenth of a minute)

from the beginning of the test until a green coloration of the flame is noted is taken as the service time (or activity) of the charcoal. The green color of the flame is enhanced by use of a dark background.

An accelerated test of charcoals against carbon tetrachloride has also been used. It differs from the accelerated chlorpicrin test only in rate and concentration. The rate, which is 500 cc. per min., is obtained by using the standard two-tube apparatus. The concentration, which is about 40,000 (theoretical about 44,000 p. p. m. at 760 mm. pressure), and the relative humidity, which is zero, are obtained by drying the entire air supply to the apparatus with concentrated sulfuric acid and then passing it through carbon tetrachloride maintained at 0° C. by an ice-water mixture. The break point is sharper than that of the accelerated chlorpicrin test, due to the fact that there are more chlorine atoms in the carbon tetrachloride molecule and to the fact that the test time is shorter.

#### STANDARD METHODS FOR TESTING CANISTERS

##### LEAKAGE TEST

The canister filled with chemicals and completely assembled is subjected to an air pressure of 15 in. of mercury; leaks are detected by immersing the canister in water.

##### RESISTANCE TO FLOW TEST

The resistance to flow of the completely assembled canister is determined at a standard rate of flow of 85 l. per min. The canister is attached to A (Fig. IV)

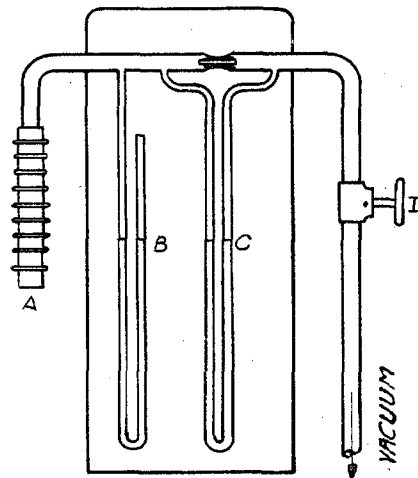


FIG. IV—APPARATUS FOR MEASUREMENT OF PRESSURE DROP

and the flow of air is adjusted by valve D to a rate of 85 l. per min. as indicated by flow meter C. The pressure drop or resistance is measured in inches or millimeters by the water column in manometer B. The resistance should not exceed 3 in. of water.

#### DETERMINATION OF EFFICIENCY OR LIFE OF CANISTERS AGAINST VARIOUS GASES

The efficiency or "life" of a canister in "gas" may be determined by an actual man test or by machine test; experience has shown that both methods are absolutely essential in development work. Each new design of canister must be submitted to exhaustive man tests in a gas chamber against all gases for which it is proposed, and, furthermore, these tests should be made in

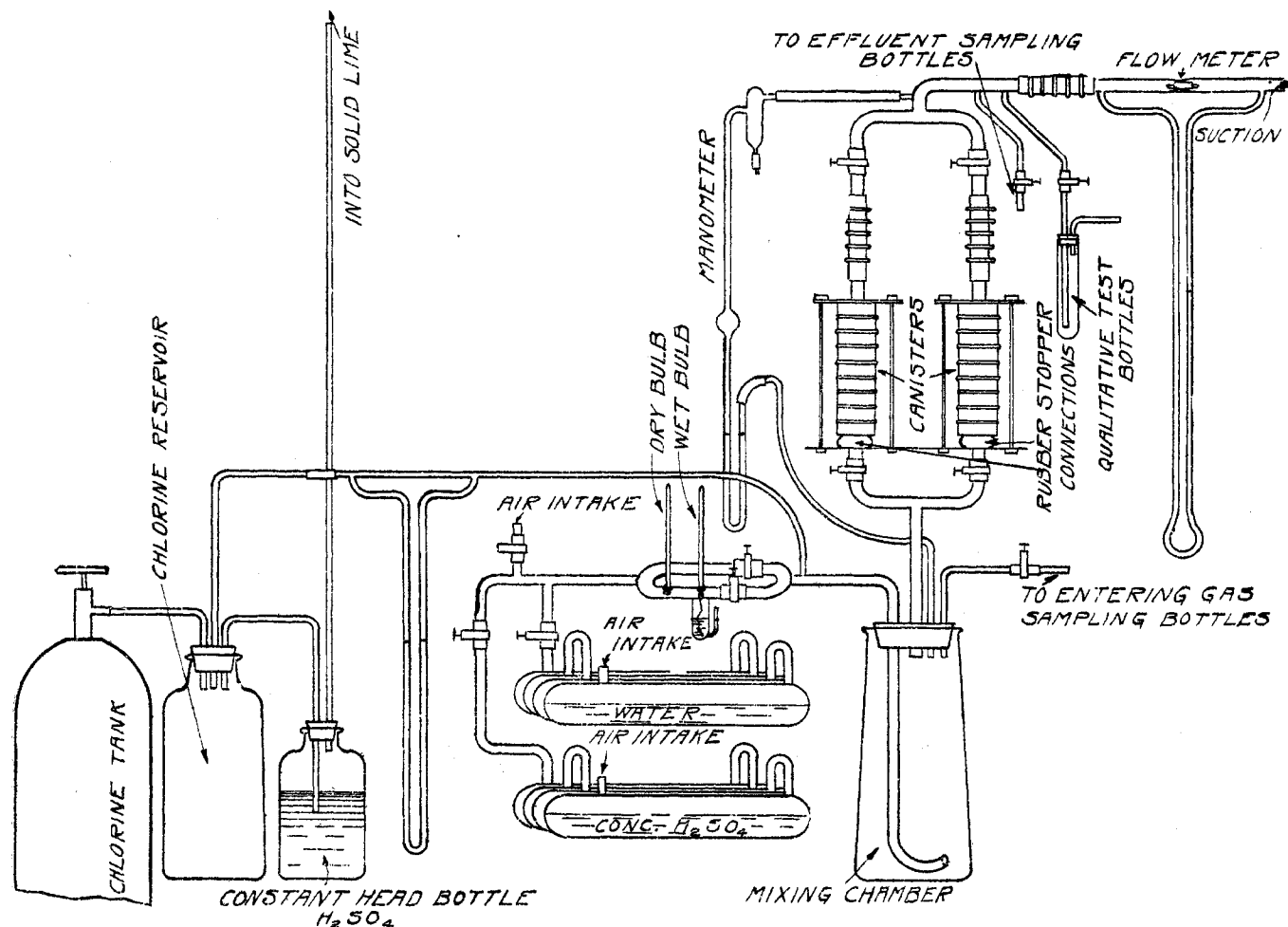


FIG. V—APPARATUS FOR TESTING CANISTERS AGAINST CHLORINE

several different concentrations including the maximum concentration that is likely to be encountered in using the mask. Deep breathing or maximum work tests must also be made to insure adequate protection under all conditions of use.

Owing to the variable breathing rates of men, and to their different degrees of sensitivity to gas, man tests are too variable to be of much use in control testing such as is required in the factory where the canisters are filled with absorbents. For this purpose machine tests are used in which a constant flow of gas at a definite concentration is maintained through the canister by suitable suction and pressure pumps. The first trace of gas penetrating the canister is detected by chemical means and the amount penetrating is determined at definite intervals of time.

The particular gases used in the machine test will depend upon the absorbents: charcoal fillings are tested against chlorpicrin; soda-lime fillings against chlorine and hydrocyanic acid; and combinations of these two fillings against chlorine (or phosgene), chlorpicrin, and hydrocyanic acid. Other gases of the same general types may, of course, be substituted, especially if the canister is to be used against them, e. g., sulfur dioxide instead of hydrocyanic acid. Methods for such other gases will suggest themselves from the four standard methods described below.

#### METHOD OF TESTING CANISTERS AGAINST CHLORINE

SOLUTIONS—(1) 0.00818 *N* sodium thiosulfate. Make up a solution of approximately 1.3 g. per l. Allow it to stand for a week and dilute to required strength as shown by its action against standard 0.0100 *N* iodine solution.

(2) Two per cent potassium iodide solution. Dissolve 20 g. potassium iodide in 1 l. water.

(3) Starch solution. Bring 1 l. of water to boiling and add 5 g. starch. Filter hot, bring again to boiling, and pour into flask. Insert stopper containing one long tube and one short one having a pinchcock attachment. Invert, and use by drawing off solution through short tube.

APPARATUS—The apparatus is shown in Fig. V. Pure chlorine gas from a tank of liquid chlorine is supplied to a reservoir bottle which is connected to a constant pressure bottle containing sulfuric acid. From the constant pressure bottle the chlorine passes through a flow meter to a mixing jar. At the same time, air of 50 per cent relative humidity is drawn into the mixing jar, and the air-gas mixture is then drawn through the canister to be tested and a large flow meter on which can be read the total volume.

The stand is built to hold two canisters which are connected into the line in parallel between the mixing jar and flow meter. Stopcocks or pinchcocks on soft

rubber tubing placed on either side of each canister permit changing the air stream from one to the other without stopping the flow. The canisters rest on large rubber stoppers (No. 12) fitted on to the tubes from the mixing jar clamped in place by bolts passing through the iron stand. The connection from the top of the canisters to the  $\frac{3}{4}$  in. glass manifold leading to the main flow meter is made by means of a piece of heavy-wall flexible tubing such as is used in making connection between the canister and face-piece of the completed gas mask. There are three small side tubes in this glass tube which leads from the canister. One of these is used for drawing off effluent samples; from the second, the gas for the qualitative test is aspirated; and the third is connected to the other side of the manometer which measures the pressure drop across the canister.

The samples for the determination of the concentration of chlorine in the entering and effluent air are drawn through absorption bottles by means of a large water-filled aspirator from which is run, into a calibrated flask, a volume of water equal to the volume of the sample required.

A three-canister apparatus, such as that used for chlorpicrin, can be substituted for the two-canister type described if it is desired to run a greater number of tests. Such an apparatus permits the testing of two canisters simultaneously. (See Method of Testing the Efficiency of Canisters against Chlorpicrin.)

**PROCEDURE**—The canister is first examined for leaks according to the method already described.

The pressure drop, in inches of water across the canister at 85 l. per min., is then determined.

With an old canister in position on the stand the apparatus is started. The air flow is adjusted to 32 l. per min., as shown by the main flow meter, and the relative humidity of the entering air is brought to 50 per cent by governing the relative amounts drawn over the sulfuric acid and the water tubes, the resulting value being determined from the wet and dry bulb hygrometer. (The humidity is frequently checked.) Next, the concentration of the air-gas mixture is brought to the desired value (5000 p. p. m. by volume for routine tests) by varying the rate of flow of the chlorine as shown by the small flow meter, the concentrations being actually determined by analyzing samples of the entering mixture.

When the relative humidity and concentration have been adjusted to the desired values, the canister to be tested, and which has been placed in the vacant position is quickly connected into the line and the old one cut out by means of the stopcocks above and below each canister, without interrupting the air flow. This causes the least possible dislocation of the concentration and makes the testing continuous.

The time of starting the test is recorded. Effluent gas at 500 cc. per min. is immediately started through the qualitative test bottle containing about 50 cc. of 2 per cent potassium iodide solution and a little starch solution. During the time which this qualitative test is running, the main flow should show 31.5 l. per min.,

making a total of 32 l. per min. Samples of entering air, 500 cc. in volume, are taken every 20 min. throughout the run. The pressure drop across the canister at 32 l. per min. is observed from the manometer about three times during the run and for any increase noted.

After the qualitative test has indicated a break, the concentration of chlorine in the effluent mixture is determined, 2-l. samples being taken successively until the efficiency of the canister has dropped below 90 per cent. The efficiency is calculated on the basis of the nearest entering concentration.

The time from the beginning of the test to the first appearance of blue in the qualitative test bottle is recorded as the time at 100 per cent efficiency, and the time in minutes from the beginning of the test to the middle of the sampling period of each effluent sample is plotted against the per cent efficiency.

Per cent efficiency =

$$\frac{\text{P. p. m. entering gas} - \text{p. p. m. effluent gas}}{\text{P. p. m. entering gas}} \times 100$$

A smooth curve is drawn through these points and the corrected times to such efficiency points as 100, 99, 95, 90, and 80 per cent are read from the curve.

In comparing the results of canister tests it is most convenient to use these specified points. The elapsed times must be corrected to the nominal concentration at which the test is being run, as the actual average concentration may fluctuate up to plus or minus 5 per cent. This correction is made according to the linear inverse proportion:

Corrected time =

$$\text{Observed time} \times \frac{\text{Actual average concentration}}{\text{Nominal concentration}}$$

The average temperature of the entering gas mixture is recorded and also the pressure drop through the canister at 85 l. per min. at the beginning and end of the test.

The machine is checked on alternate days by testing a canister of a previous packing date and comparing the result with the average tests of that date.

**ANALYSIS**—The concentration of chlorine in both the entering and effluent mixture is determined by aspirating samples through about 50 cc. of 2 per cent potassium iodide solution. By making use of a "petticoat" bubbler (shown in Fig. I) one absorption bottle is sufficient to recover all of the chlorine in the entering gas sample. Half-liter samples of entering gas and 2-l. samples of effluent gas are used; these are taken at the rate of about 1 l. per min. The iodine thus liberated is titrated with 0.00818 *N* sodium thio-sulfate solution and the results calculated to parts (volume) per million at 25° C. and 760 mm.

**CALCULATIONS**—Standard solutions are made of such strength that 1 cc. of solution is equivalent to 100 p. p. m. of chlorine at 25° C. and 760 mm. in a 1-l. sample.

The calculation is as follows:



- 1 equivalent chlorine =  $\frac{24.45}{2}$  = 12.23 l. at 25° C. and 760 mm.  
 1 l. *N* sodium thiosulfate = 12.23 liters chlorine at 25° and 760 mm.  
 1 cc. 0.01 *N* sodium thiosulfate = 0.1223 cc. chlorine at 25° C. and 760 mm.  
 In a liter sample 1 cc. 0.01 *N* sodium thiosulfate = 122.3 p. p. m.  
 1 cc. 0.00818 *N* sodium thiosulfate = 100 p. p. m. in a liter sample at 25° C. and 760 mm.  
 For a sample of *A* liters,  
 1 cc. 0.00818 *N* sodium thiosulfate =  $\frac{100}{A}$  p. p. m.  
 5000 p. p. m. = 14.51 mg. per l.

## METHOD OF TESTING CANISTERS AGAINST PHOSGENE

SOLUTIONS—(1) Alcoholic sodium hydroxide, chlorine-free. Five hundred cc. of aqueous sodium hydroxide solution (35 g. per 100 cc. water) diluted to 9 l. with 95 per cent alcohol.

(2) 0.00818 *N* silver nitrate. Approximately 1.5 g. per l. of water, diluted to 0.00818 *N* as shown by titration against standard 0.01 *N* sodium chloride solution.

(3) Potassium chromate indicator. Use 50 g. of neutral potassium chromate in a little distilled water. Add enough silver nitrate to produce a slight red precipitate. Filter and make up to one liter.

(4) Pieces of filter paper 1 cm. × 2 cm. are placed in water and 3 or 4 drops of strong Congo-red solution added. Paper is allowed to soak 1 hr. The best depth of color must be determined by experience.

(5) *p*-Dimethylaminobenzaldehyde test paper, while not being used as a qualitative test paper, due to the fact that the color change is not marked in low concentrations of hydrochloric acid, which first appears in the effluent air, has been found to be valuable in locating gas leaks in the machine. (Its preparation has been described under absorbent testing.)

(6) Phenolphthalein solution. One per cent in 95 per cent alcohol.

APPARATUS—The apparatus is nearly identical with that used for chlorine. The phosgene is taken from an iron cylinder similar to that used for chlorine. The excess gas is absorbed in alcoholic sodium hydroxide. For the qualitative test a 4 in. glass tube  $\frac{3}{4}$  in. in diameter, containing a test paper, is used instead of the absorption bottle of the chlorine apparatus. The effluent quantitative samples are drawn off by means of a small flow meter instead of an aspirator.

Phosgene readily corrodes brass. The controlling valve is, therefore, protected by means of a soda-lime tower placed in line just after the main flow meter.

A three-canister apparatus such as that used for chlorpicrin can be substituted for the two-canister type described if it is desired to run a greater number of tests.

PROCEDURE—The procedure is the same as that for chlorine. Moistened Congo-red paper is used in the qualitative test. The quantitative samples are absorbed in alcoholic sodium hydroxide solution. The tests are run at 32 l. per min. and 5000 p. p. m. The main flow meter is set at 30 l. per min., while 2 l. per min. are passed through a qualitative tube or a quantitative sampling bottle. Fifty per cent relative humidity is used. Half-liter and 2-l. samples are taken for the entering and effluent concentrations, respectively.

NOTE—Phosgene is readily absorbed by the rubber connections. To avoid error in the qualitative test and effluent concentration determinations, the rubber connections at the top of the canisters are changed for every test and thoroughly washed with alcohol before being replaced.

ANALYSIS—The gas samples are absorbed by aspirating through 50 cc. of alcoholic sodium hydroxide solution in which the phosgene is hydrolyzed to sodium carbonate and sodium chloride. One absorption tube with petticoat bubbler is sufficient. Phenolphthalein is added and the excess sodium hydroxide neutralized with approximately normal nitric acid. The chloride is determined by titration with 0.00818 *N* silver nitrate, using potassium chromate solution as indicator.

The use of moist Congo-red paper as an indicator depends upon the presence of hydrochloric acid in the effluent gas and the consequent change in color of the paper from red to blue.

CALCULATIONS—The calculations are substantially the same as for chlorine.

- 1 cc. 0.00818 *N* silver nitrate = 100 p. p. m. phosgene at 25° C. and 760 mm. in a 1-l. sample.  
 5000 p. p. m. = 20.225 mg. per l.

## METHOD OF TESTING CANISTERS AGAINST HYDROCYANIC ACID

SOLUTIONS—(1) Sulfuric acid. One volume concentrated sulfuric acid (1.84 sp. gr.) to one volume water.

(2) Sodium cyanide. Approximately 25 g. to 100 cc. of water.

(3) Sodium carbonate. 20 g. per l.

(4) Sodium bicarbonate. 20 g. per l.

(5) Iodine. 0.0164 *N*, in potassium iodide solution.

(6) Starch solution. 5 g. per l.

APPARATUS—The apparatus, shown in Fig. VI, is similar to that used for chlorine, except that the gas is supplied from a generator instead of a cylinder. The canister stand is built to hold two canisters. T-tubes, carrying the entering and effluent gas, and soft rubber connections are used which permit shutting off either canister by means of pinch clamps.

A three-canister apparatus such as that used for chlorpicrin can be substituted for the two-canister type described if it is desired to run a greater number of tests.

The generator is a 5-l. bottle which is placed in an ice bath. Dropping tubes for the sulfuric acid and sodium cyanide, a tube for passing in air to carry over the gas, an outlet tube, and a siphon are inserted into the bottle through a large rubber stopper. A small flow meter is provided to control the rate at which air enters the generator. The air incidentally serves to agitate the liquid and provide thorough mixing. Bottles containing dilute sulfuric acid, sodium cyanide solution, and water are placed on a shelf directly above the generator and connected to it by siphon tubes. A small compressor forces the air through the generator.

PROCEDURE—To start a run, the ice bath is filled, an inch of water run into the generator (to assist in hold-

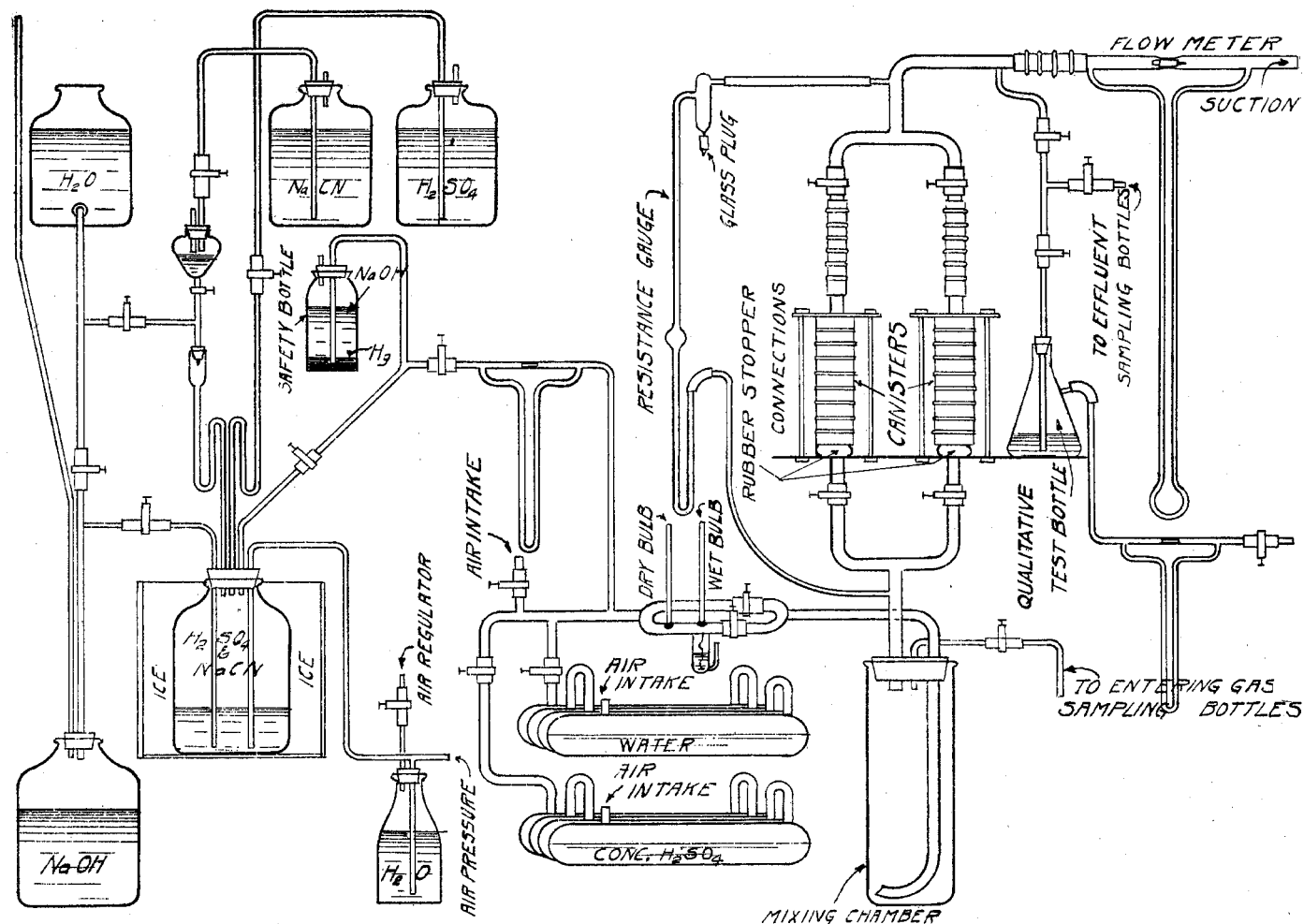


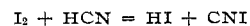
FIG. VI—APPARATUS FOR TESTING CANISTERS AGAINST HYDROCYANIC ACID

ing in solution the sodium sulfate formed), followed by 750 cc. of dilute sulfuric acid (1 : 1) and about 50 cc. of the cyanide solution. The pumps are then started and air at 32 l. per min. is drawn through the apparatus with an old canister in the line, and at the same time air at 1 to 1.5 l. per min. is forced through the generator and into the mixing chamber. After the addition of the first 30 cc. of cyanide solution (which should take about 5 min.) the flow of solution is gradually cut down during a period of 10 min. to approximately 36 drops per min., where it is held constant. In this way it ordinarily takes  $\frac{3}{4}$  hr. to get up concentration. This has been found to give a concentration of 2000 p. p. m. at 32 l. per min. If the concentration is unsatisfactory, it may be changed by varying the rate of air flow through the generator, and the rate of dropping. The concentration, with moderate care in controlling the air flow and dropping rate, can be kept within 5 per cent of that desired.

When the concentration is satisfactory, the canister to be tested is opened to the line, by removing the pinch clamps above and below it, and the old one cut out. Effluent gas at 500 cc. per min. is aspirated through the qualitative test bottle containing 50 cc. of 2 per cent sodium bicarbonate solution, a little starch, and sufficient iodine solution to give the whole a distinct blue tinge. The flow, as indicated by the large flow meter, is set at 31.5 l. per min. to bring the

total through the canister to 32 l. per min. Samples for determining the concentration of entering and effluent gas are aspirated through 50 cc. of 2 per cent sodium carbonate solution in an absorbing cylinder, 5-l. samples of effluent and 1 l. samples of entering gases being taken by means of a flow meter and an aspirator bottle, respectively. The hydrocyanic acid is determined by titration with standard iodine solution, using starch as an indicator. Results are calculated to parts per million of gas at 25° C. and 760 mm.

**ANALYSIS**—Samples are absorbed in a 2 per cent aqueous sodium carbonate solution and the hydrocyanic acid titrated with 0.0164 *N* iodine solution, using starch as an indicator, the reaction being as follows:



**CALCULATIONS**—1 l. *N* iodine solution =  $\frac{24.45}{2}$  l. hydrocyanic acid at 25° C. and 760 mm.

1 cc. *N* iodine solution = 12.23 cc. hydrocyanic acid at 25° C. and 760 mm. = 12,230 p. p. m. hydrocyanic acid at 25° C. and 760 mm.

1 cc. 0.01636 *N* iodine solution = 200 p. p. m. at 25° C. and 760 mm. in a 1 l. sample.

2000 p. p. m. = 2.22 mg. per l.

#### METHOD OF TESTING CANISTERS AGAINST CHLORPICRIN

**SOLUTIONS**—*Absorbing Solution*—Equal volumes of 2 per cent sodium peroxide solution and 95 per cent alcohol mixed just before using.

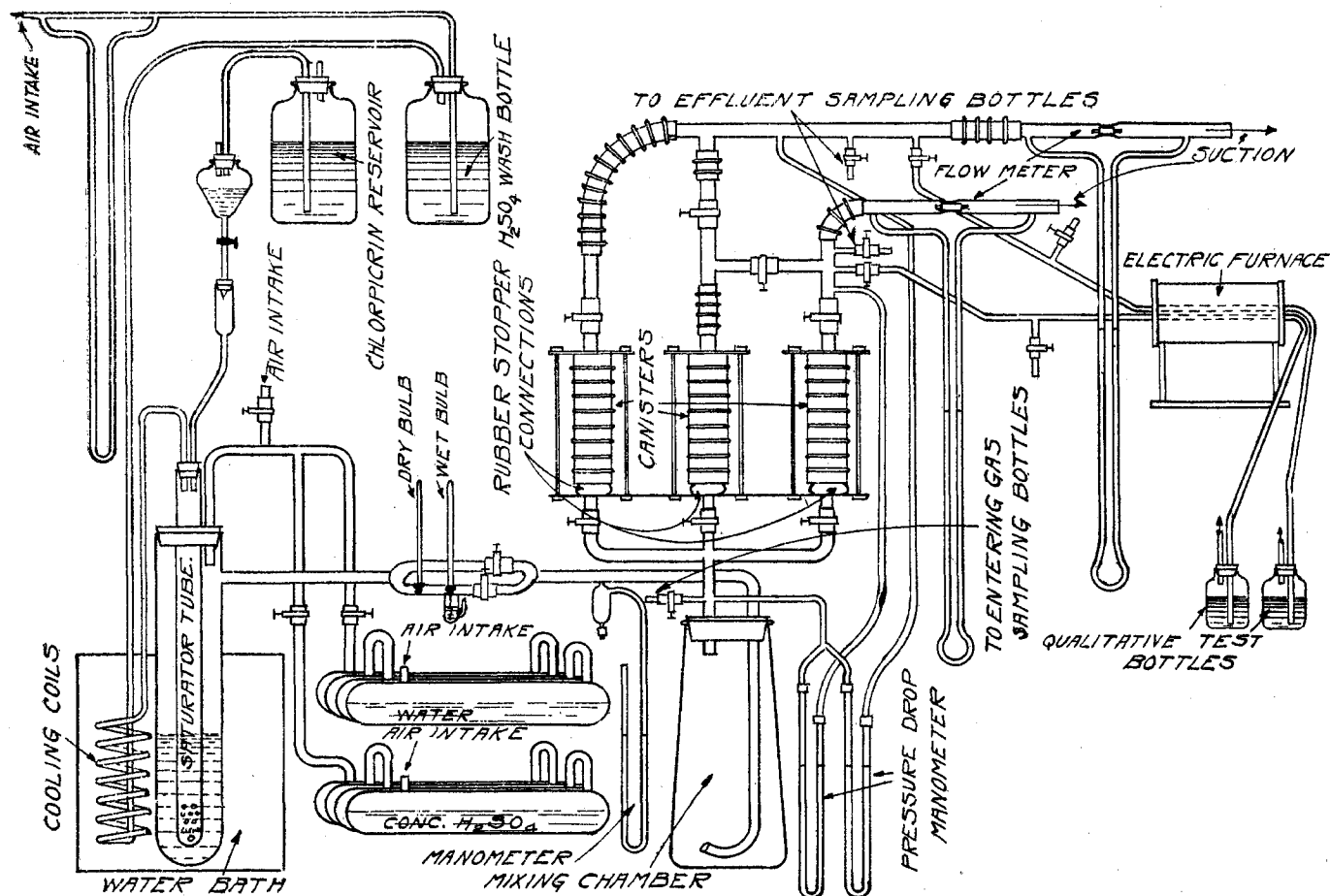


FIG. VII—APPARATUS FOR TESTING CANISTERS AGAINST CHLORPICRIN

**Standard Solution**—Silver nitrate solution, 0.0123 N 2.3 g. per l. standardized against standard sodium chloride solution and diluted to 0.0123 N.

**Potassium Chromate Indicator**—Use 50 g. of neutral potassium chromate in a little distilled water. Add enough silver nitrate to produce a slight red precipitate. Filter and make up to one liter.

**Qualitative Test Solution**—Potassium iodide solution, 20 g. potassium iodide per l. Starch solution, 5 g. starch per l.

**APPARATUS**—The apparatus used is shown in Fig. VII. It is similar in part to those previously described. The chlorpicrin is contained in a glass tube 5 cm. in diameter and 60 cm. long, and is maintained at 25° C. in a water bath. Air at constant rate (regulated by a flow meter) is bubbled through the liquid after having been previously passed through a coil in the water bath. The height of liquid is held constant by dropping liquid steadily from a dropping funnel sealed in the top of the tube. A siphon tube is also sealed in for the purpose of removing the liquid when desired. The bath is agitated by an air pump to insure even temperature.

An additional volume of air, sufficient to make the total 32 l. per min. per canister, is drawn into the line through the humidity control and, together with the chlorpicrin-air mixture, passes by the humidity indicator and into the mixing chamber. The mixing jar is a glass cylinder 10 cm. in diameter and 40 cm. high,

closed with a wooden stopper sealed in with sealing wax. By means of rubber tubing and a screw clamp at the air intake, the pressure inside the mixing jar is kept at 2 cm. of water below atmospheric. This is determined by a manometer, one side of which is connected in the line. (The slight reduction in pressure is merely to insure no possible exit of gas through the large air intake.)

The iron stand and the method for clamping the canister in place is the same as on the chlorine machine, except that it is arranged for three canisters in parallel. After leaving the mixing chamber the air stream is divided so that two canisters may be tested simultaneously, yet independently of each other. The third canister may be substituted at will for a canister on which the test has been completed, thus avoiding interruption between tests (see diagram). The gas passes from each canister to a large flow meter and thence through a needle valve to the suction pump.

A manometer for measuring the pressure drop across each canister is connected in the line by means of side tubes on each side of the canister.

The qualitative test is made by using two quartz tubes filled with broken porcelain and heated in an electric or gas furnace to 400° C.

**PROCEDURE**—The bath agitator is started and the furnace heated.

With two old canisters in the train, air at 32 l. per min. is drawn from the mixing jar through each. At

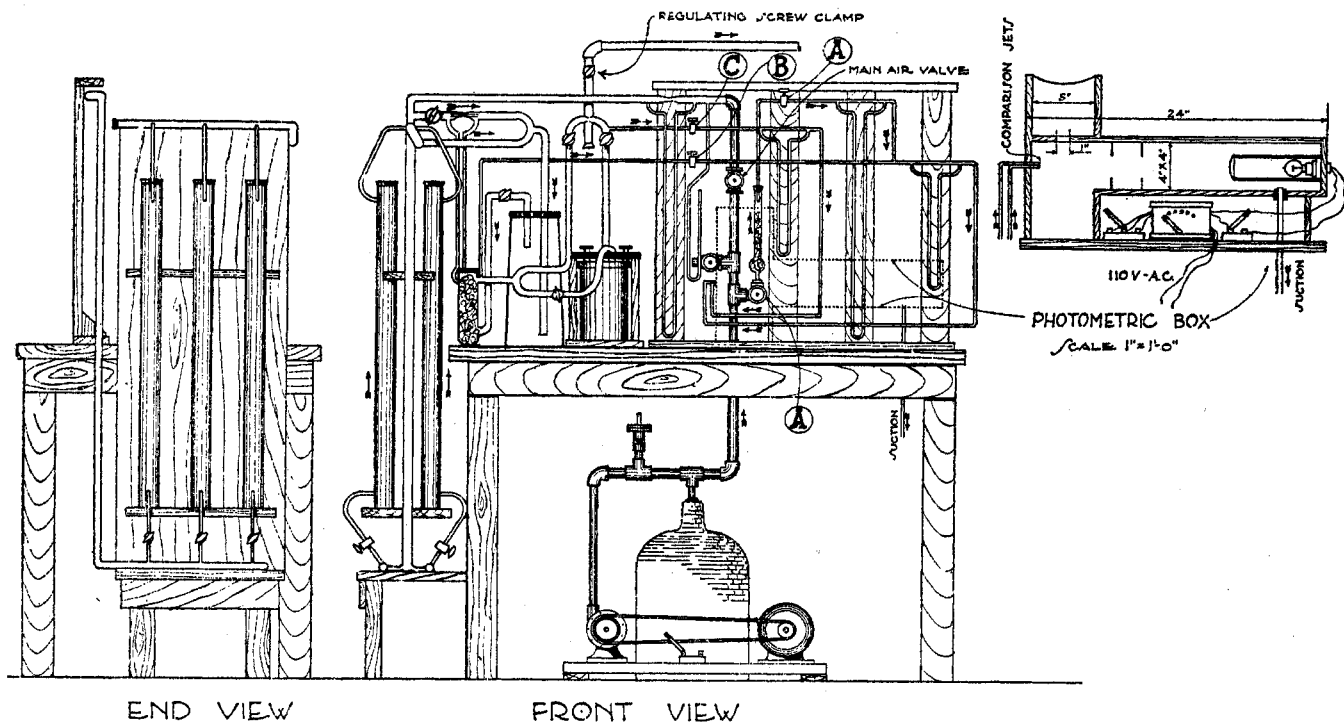


FIG. VIII—APPARATUS FOR TESTING CANISTERS AGAINST SMOKE

the same time, air is started through the chlorpicrin at such a rate as will give the desired concentration of chlorpicrin in the final gas-air mixture (2500 p. p. m. for routine tests). Liquid chlorpicrin is added from the dropping-funnel as fast as it is vaporized in the tube. The temperature is kept between 23° and 28° C. and the humidifier adjusted to give 50 per cent relative humidity. The screw cock on the air intake is closed till the pressure within the apparatus is 2 cm. of water below atmospheric.

In the meantime, the third, but new, canister has been inserted in place. When the desired concentration has been reached, the flow through one of the old canisters is switched through this new one and its test thus started. Another new canister is now inserted in place of this released old one and the flow of gas changed as above. For convenience, the canisters are inserted so as to "break" at different times. The pressure drop of each canister is taken at 85 l. per min. before and after the test. The time and temperature are noted at the beginning of the test. Every 20 min. during the test the entering gas concentration is determined by aspirating 1-l. samples through the two absorbing bottles, each containing a mixture of 25 cc. of 2 per cent aqueous sodium peroxide solution and 25 cc. of 95 per cent alcohol and analyzing as described below. Effluent gas is aspirated through the furnace and the qualitative test bottle, containing about 50 cc. of 2 per cent potassium iodide solution and a few drops of starch solution, at 1 l. per min. The large flow meter is set to deliver 31 l. per min. while the qualitative test is being taken, in order that the total flow through the canister may be kept constant at 32 l. per min. When the first positive qualitative test is obtained a 2-l. effluent sample

is taken, followed by at least two others before the canister has fallen to 90 per cent efficiency.

**PRECAUTIONS**—The furnace used in the qualitative tests has been found to hold up small amounts of chlorine and consequently gave a false test. To overcome this it is necessary to draw air through the furnace for about 20 min. between tests. Delays are avoided by turning off the qualitative test immediately after the "break" and at once starting the air through the furnace.

**ANALYTICAL METHOD**—The analytical method depends on the formation of sodium chloride by the decomposition of chlorpicrin by alcoholic sodium peroxide solution.

Samples of entering and effluent gases of known volume are aspirated through two bottles, each containing 50 cc. of a half and half mixture of 2 per cent (by weight) sodium peroxide solution and 95 per cent alcohol.

The solution is washed out into an Erlenmeyer flask, neutralized with nitric acid, and titrated immediately with silver nitrate solution, using potassium chromate indicator.

**CALCULATIONS**—1 g. equivalent chlorpicrin vapor =  $\frac{24.45}{3}$  l. at 25° C. and 760 mm.

∴ 1 l. *N* silver nitrate =  $\frac{24.45}{3}$  l. chlorpicrin vapor at 25° C. and 760 mm.

1 cc. 0.01 *N* silver nitrate = 0.0185 cc. chlorpicrin vapor at 25° C. and 760 mm.

∴ 1 cc. 0.01 *N* silver nitrate = 81.5 p. p. m. chlorpicrin vapor at 25° C. and 760 mm. in a 1 l. sample.

∴ 1 cc. 0.0123 *N* silver nitrate = 100 p. p. m. chlorpicrin vapor at 25° C. and 760 mm. in a 1 l. sample.

2500 p. p. m. = 16.61 mg. per l.

METHOD OF TESTING THE FILTERING EFFICIENCY OF CANISTERS AGAINST TOBACCO SMOKE<sup>1</sup>

PRINCIPLE OF THE METHOD—"Tobacco Smoke" is generated by passing air over ignited sticks consisting of a mixture of tobacco, rosin, and potassium nitrate. The smoke thus produced is passed through the canister at a rate of 85 l. per min. A part of the entering smoke is by-passed and diluted with a measured amount of air; the mixture is then passed through a flow meter (maintained at 1500 cc. per min.) and allowed to issue from a jet alongside a similar jet of the effluent smoke. A constant amount of this effluent smoke (1500 cc.) per min. also issued from a jet. A strong beam of light is directed lengthwise upon these two jets of smoke. The Tyndall beams are observed laterally and the relative amounts of air and entering smoke varied until the two streams of smoke appear bright. Inasmuch as the same volume of smoke-air passes through both jets, the amount of air required to dilute the unfiltered smoke and produce the same optical density in the two streams is a measure of the efficiency of the filter. The scale in the dilution meter is arranged to give the percentage efficiency of the canister directly; this may be calculated from the formula

Percentage efficiency =

$$100 \times \frac{\text{Cc. air used for dilution}}{\text{Total cc. (1500)}}$$

APPARATUS—The apparatus is shown to scale in Fig. VIII, arrows indicating the direction of flow of air and smoke. Air under pressure passes through a flow meter and enters a main to which are connected three pairs of vertical iron cylinders, 5 cm. in inside diameter and 90 cm. long. A part of the air may be by-passed and then mixed with the smoke stream in the mixing chamber. Inside each cylinder is hung a tobacco stick which is ignited at the bottom when the machine is put in operation. The smoke produced by the combustion of these sticks passes into a main which delivers it to a jar, which jar acts as a settling chamber. From the settling chamber the smoke passes through a filter bottle which contains a loose plug of cotton for removing any large particles of smoke, tarry matter, etc. Before the settling chamber and the tar filter is placed a by-pass through which the smoke can be directed to the exit while the apparatus is being adjusted and the canisters changed.

From the tar filter a small part of the smoke passes through a flow meter, adjusted to give 1500 cc. per min. after dilution with air, and then into the optical box. The rest of the smoke passes into the canister jar in which the canister is placed, and is forced through the canister at a rate of 85 l. per min. After having passed through the canister, 1500 cc. are metered and led into the optical box through a jet, and the remainder is discarded through the regulating valve. A connection and a valve, A, are provided, in order that air from the compressed air line can be metered by the "percentage flow meter" and used to dilute the unfiltered smoke mixture before the diluted mixture is

metered and passed into the optical box through the second jet.

The optical box consists of a wooden box of the shape and dimensions shown in the drawing. It is painted dull black inside in order to prevent reflection of light. The smoke jets are illuminated by a lamp (6 to 8 v., 24 c. p.) and condensing lens system situated at the end of the box opposite that through which the smoke enters. A slight suction on an outlet near the lamp removes the smoke from the box. The glass tips from which the smoke issues are of 3 mm. inside diameter. A rheostat or transformer is used in series with the lamp in order to regulate the brightness of illumination of the smoke jets, a bright illumination being used in matching the jets in the case of a good canister and a poorer illumination in the case of a poorer canister.

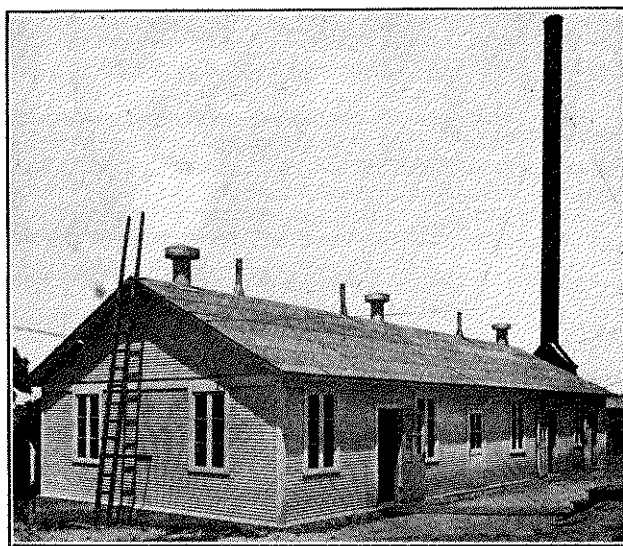


FIG. IX.—EXTERIOR OF MAN-TEST LABORATORY

One of the most important parts of the apparatus is the percentage flow meter. This should be carefully calibrated, and the diluted smoke flow meter checked against it before each run in order to avoid any error due to plugging of this flow meter with smoke. The effluent smoke flow meter should also be checked against this diluting air flow meter.

PROCEDURE—(a) *Adjustment of Apparatus*—About one end of a tobacco stick, wrap a wire so as to form a loop; evenly ignite the opposite end, hang the stick on the upper stopper by the loop so that the upper end is about 10 in. from the stopper, and then tightly insert the stopper in the smoke cylinder. Fill the six tubes in this manner. With the regulating screw clamp partly open and the by-pass around the canister box open, the main air valve is opened until a differential corresponding to 85 l. per min. shows on the main flow meter. Usually a maximum of 30 l. per min. is allowed to pass through the smoke tubes (5 l. per tube); the balance is by-passed and meets the smoke stream in the mixing chamber.

The regulating screw clamp and the main air valve are so adjusted that 85 l. are passing with a pressure

<sup>1</sup> Method worked out by Lt. H. C. Arnold, P. M. Horton, and E. S. Longfellow.

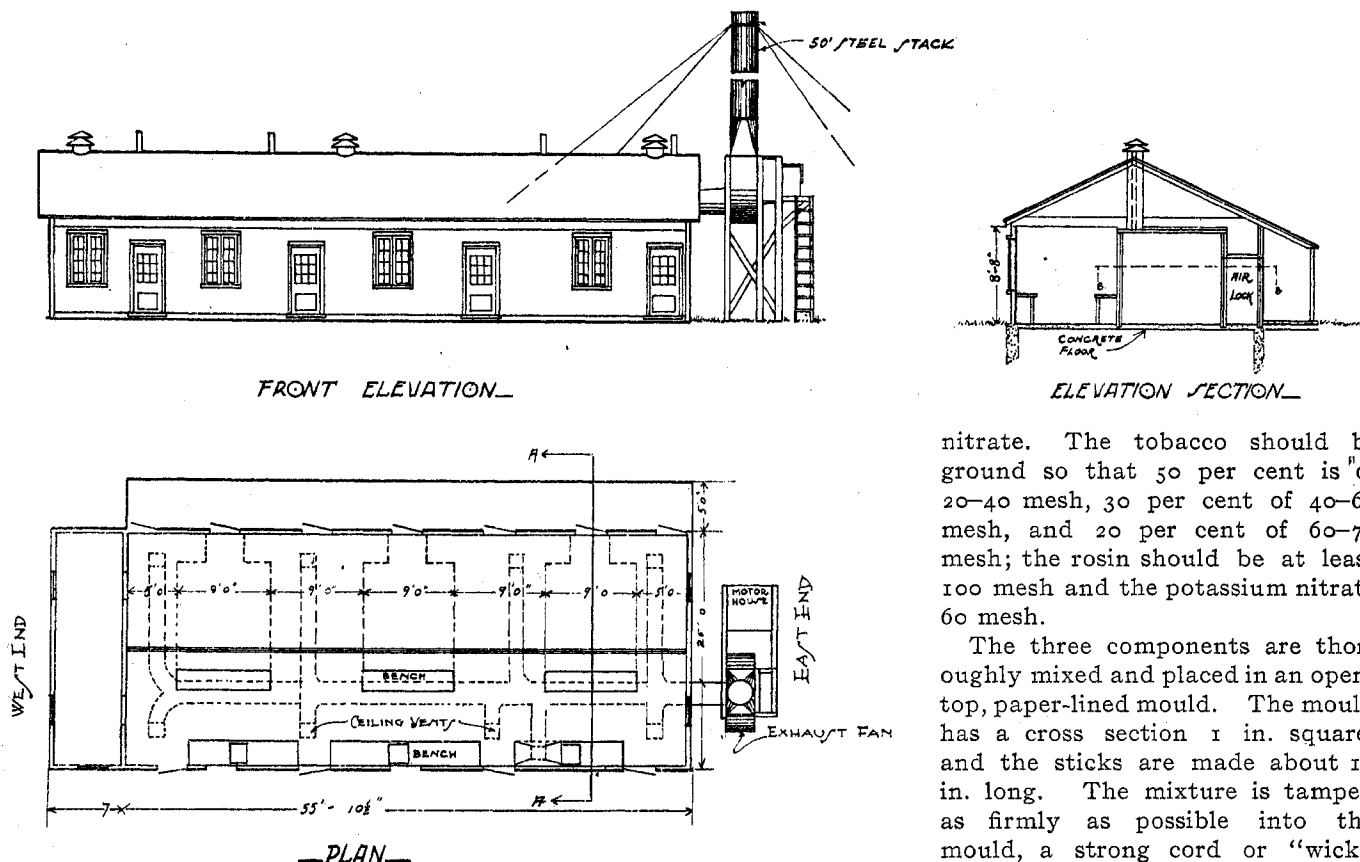


FIG. X—ELEVATION AND PLAN OF MAN-TEST LABORATORY

head of 80 mm. of mercury, shown by an open end manometer placed just before the main flow meter. This head is necessary to operate the measured smoke sample flow meters. When smoke is passing uniformly through the apparatus, close the by-pass around the canister box, thus directing the smoke through the canister under test.

(b) *Manipulation for Test*—Check the diluted smoke flow meter by opening the air dilution valve A until 1500 cc. shows on the percentage flow meter. The differential on the diluted smoke flow meter then shows 1500 cc. per min. and should be maintained at this height throughout the test (the scale is made adjustable for convenience).

While closing A, open B correspondingly, so that the diluted smoke flow meter remains constant at 1500 cc. per min. Adjust these two valves until the two smoke streams appear equally dense when observed in the optical box. The intensity of the light should be adjusted to correspond with the density of the smoke, the denser the smoke the less the illumination required for the highest precision in matching. To check the observation, two other readings should be taken, one slightly above and the other slightly below the true match.

The total time for passage of smoke through the canister should probably not exceed 5 min. Usually it will vary from 1 to 3 min.

**DISCUSSION**—The tobacco sticks are made of 63 per cent tobacco, 30 per cent rosin, and 7 per cent potassium

nitrate. The tobacco should be ground so that 50 per cent is of 20–40 mesh, 30 per cent of 40–60 mesh, and 20 per cent of 60–70 mesh; the rosin should be at least 100 mesh and the potassium nitrate 60 mesh.

The three components are thoroughly mixed and placed in an open-top, paper-lined mould. The mould has a cross section 1 in. square, and the sticks are made about 15 in. long. The mixture is tamped as firmly as possible into the mould, a strong cord or “wick” then inserted in the center to give strength to the finished stick, and

the stick heated in an oven until it begins to smoke. (The rosin melts and forms a binder for the rest of the material.) As soon as the stick has hardened the paper may be removed.

Tobacco smoke is not a clogging smoke and no error is encountered due to change in nature of filter because of absorption of smoke in the short time necessary for a test. The concentration of the testing cloud may vary slightly from time to time, but produces no effect upon the relative filtering efficiency of the canister.

#### METHOD OF CONDUCTING MAN TESTS AND DESCRIPTION OF CHAMBER

**GENERAL**—The man test laboratory at the American University provides facilities for determining the efficiency of gas masks and other protective devices, such as gas-proof clothing. Here concentrations of various gases are established in suitable chambers and the anti-gas apparatus tested directly on men.

Drawings and photographs of the laboratory and auxiliary apparatus are given in this report.

**DESCRIPTION**—The man-test laboratory (Fig. IX) is a one-story building, 56 ft. in length and 25 ft. in width. The main part is occupied by three gas chambers, laboratory tables, and various devices for putting up and controlling gas concentrations in the chambers. A small part at one end is used as an office and storeroom.

Good ventilation is of great importance in a laboratory of this nature. This is secured by means of a

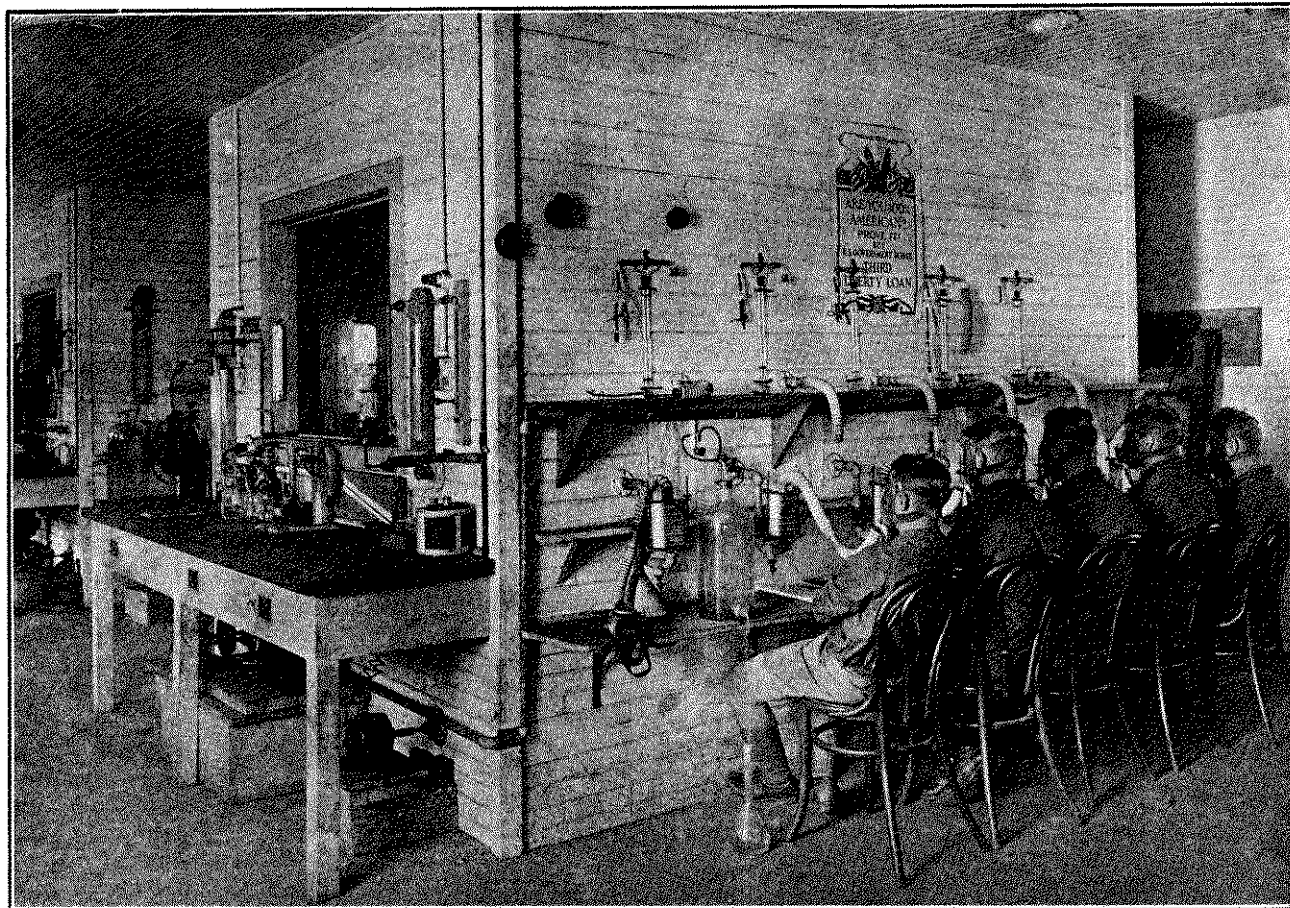


FIG. XI—INTERIOR OF MAN-TEST LABORATORY

6-ft. fan connected to suitable ducts (see Fig. X). The fan is mounted on a heavy framework outside and at one end of the building. The fan is driven at a speed of about 250 r. p. m. by a 10-h. p. motor. The main duct is 33 in. square and branches off into nine smaller ducts, each 16 in. square, extending to all parts of the building. A connection is also made to a small hood used when making chemical analyses.

The gases, fumes, etc., drawn out by the fan, are forced up and out of a stack 30 in. in diameter, extending upward 55 ft. above the ground level.

The stationary laboratory tables are provided with the usual facilities for making chemical analyses, and extend along one side of the building directly opposite the gas chambers. Here the analytical work necessary in controlling the concentration of the gas in the chambers is performed.

The main features of each of the three gas chambers are identical. Auxiliary pieces of apparatus are used with each chamber, the type of apparatus being determined by the characteristics of the gas employed. The features common to all chambers will be described first, and then the special devices will be set forth.

**FEATURES COMMON TO ALL CHAMBERS**—Each chamber is 10 ft. long, 8 ft. wide, and 8½ ft. high, having, therefore, a capacity of 680 cu. ft. or 19,257 l. (see Fig. XII). The floor is of concrete, and the walls and ceiling are constructed on a framework of 2 × 4 in. scantling, fin-

ished on the outside with wainscoting and on the inside with two layers of Upson board (laid with the joints lapped) covered with a ½ in. layer of special cement plaster laid upon expanded metal lath. The interior finish is completed by two coats of acid-proof white paint. The single entrance to the chamber is from outside the laboratory, and is closed by two doors, with a 36 × 40 in. lock between them. These doors are solid, of 3-ply construction, 2½ in. thick, with refrigerator handles, which may be operated from either inside or outside the chamber. The door jambs are lined with ⅜ in. heavy rubber tubing to secure a tight seal.

At the end of the chamber opposite the doors, a pane of ¼ in. wire plate glass, 36 × 48 in., is set into the wall, and additional illumination may be secured by 2 headlights, 12 in. square, set into the ceiling of the chamber and of the air-lock, respectively, and provided with 200-watt Mazda lamps and Holophane reflectors. Beneath the large window, a heavy built-in table, 24 in. wide, extends across the width of the chamber outside, for holding sampling devices or any other apparatus necessary for the tests. Openings into the chamber, five in number, are spaced across this end beneath the window and 9 in. above the table top. Two of these openings are of 1½ in. pipe and the remaining three of 1 in. pipe threaded on the exterior to receive connections. Beneath the tables

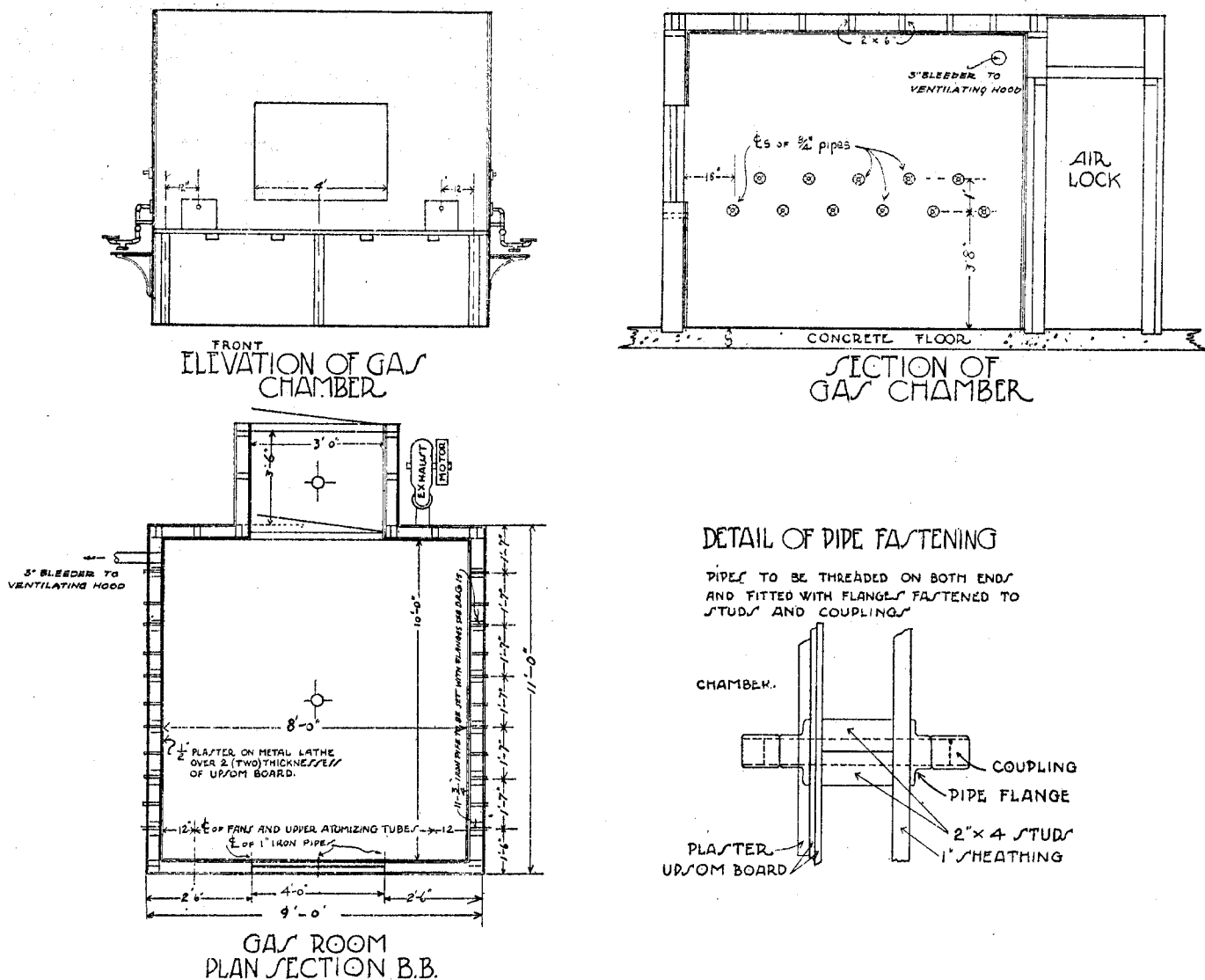


FIG. XII—ELEVATION AND PLAN OF GAS CHAMBERS

are installed fan motors and an air pump for producing either pressure or vacuum in the chamber. The fans for keeping the concentration uniform, two for each chamber, are 16 in. in diameter, 6-bladed, mounted in adjacent corners opposite the door; their shafts, running through stuffing boxes, are coupled directly to small  $\frac{1}{8}$ -h. p. motors having a speed of 1725 r. p. m. and mounted under either end of the table outside the chamber. Between these is placed a Leiman (size B) air pump, driven by a  $\frac{1}{2}$  h. p. motor and delivering 10 cu. ft. air at a speed of 600 r. p. m. An expansion tank is provided, and, by means of suitable connections, either the intake or exhaust of the pump may be applied to the chamber. These pumps are employed for a number of purposes to be described later.

The arrangements for attaching the canisters to be tested are adaptable to a large variety of types of canisters. Fig. XIII shows the device used for the standard army canisters. Along both sides of the chamber the wall is pierced, at a height of 44 in. from the floor, by a series of  $\frac{3}{4}$ -in. pipes (six in all) spaced 18 in. apart and extending about 3 in. through

the wall. On the exterior of the chamber the pipes are bent downward, then horizontally, and finally upward, terminating in a flat plate, A, of lenticular outline. At the proper distance apart to permit the adjustment of a canister between them, two threaded  $\frac{1}{4}$ -in. rods, F, F, extend upwards for 8 $\frac{1}{2}$  in. through the plate, and are provided with wing nuts. To attach the canister, a  $\frac{1}{4}$  in. rubber gasket of the proper shape is laid on the flat plate, the canister placed upon it, and a yoke, B, of  $\frac{1}{8}$ -in. sheet steel laid across the top; then, by turning down the wing nuts, the canister is pressed on the gasket until an air-tight fit is secured. As the vertical rods are threaded for 4 $\frac{1}{2}$  in. of their length, standard type cans of any height from 8 $\frac{1}{2}$  in. down may be accommodated. Small wooden blocks are placed under the yoke when very short styles are tested. For the standard oval-bottom canister with its circular central opening, a simple circular gasket suffices, but by providing several sets of oval gaskets large enough to engage the periphery of the canister, and with central openings of different sizes, it has been found possible to accommodate many experimental types. Short, cylindrical canisters of the



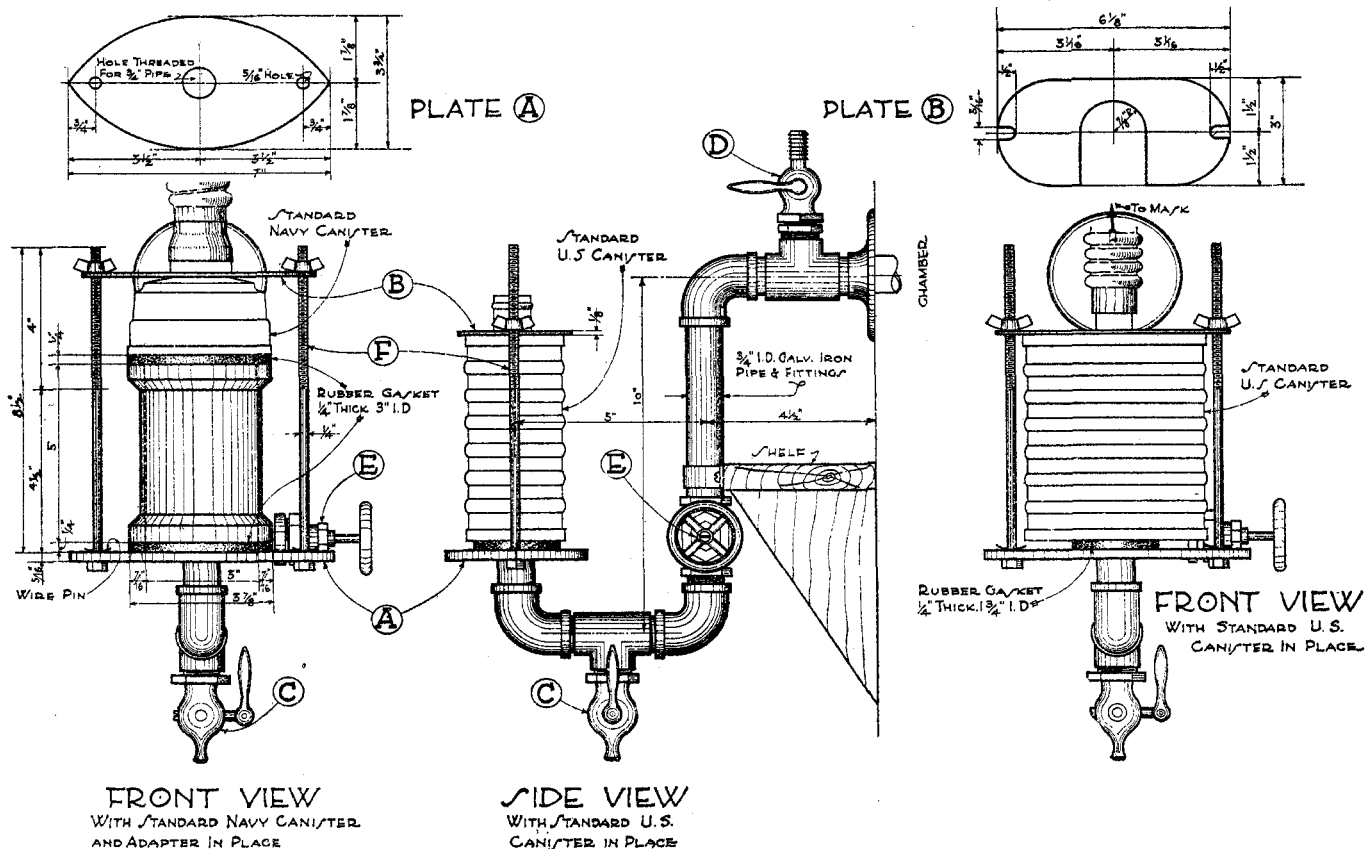


FIG. XIII—DETAILS OF CANISTER HOLDER

German type are attached by means of a cylindrical adapter (Fig. XIII) of heavy iron piping 5 in. high and of the same diameter as the canister. As shown in the figure, the adjustment is the same as for standard types, an additional gasket being inserted between the canister and its adapter. A gate valve, E, set in the piping allows the connection with the gas chamber to be shut off at will and prevents the escape of gas when no canisters are in position. At the lowest point in the bend of the piping, a small petcock, C, is inserted for draining off any liquids which may condense in the tubes. Three of the pipes on each side of the chamber have an additional petcock, D, attached just where they emerge from the wall. These are used for aspirating samples of the gas for analysis, the samples thus obtained being as nearly representative as possible of the concentration inhaled into the canisters. For convenience in collecting samples, two shelves are placed along the sides of the chamber (Fig. XII): a large one, 14 in. wide and 27 in. from the floor for holding the large aspirating bottles, and another 4 in. wide and 12 in. higher up, for holding the gas-washing cylinders in which the samples of gas are absorbed. The larger shelf is used also for holding the papers and magazines provided for the use of men taking the tests.

The obvious advantage of the bracket arrangement described above is that the canisters may be attached or changed at will, without any necessity for disturbing the concentration of gas by entering the chamber to make these adjustments. However, if it is necessary

that canisters should be attached inside the chamber, this is provided for by a second series of 3/4 in. pipes, five on each side, set in at a height of 56 in. from the floor and spaced 18 in. apart, alternating with those of the lower series. These pipes merely extend through the wall and terminate in smooth nipples, 3 in. long, the attachment to the canisters being completed by lengths of flexible tubing. Connections outside the chamber are made in similar fashion with flexible rubber tubing, the tubes being tightly stoppered when not in use. Practically all styles of canisters may be tested inside the chamber by this means.

Arrangements for removing the gas from the chamber consist of a small "bleeder" which allows a continuous escape of small amounts, and a large blower for rapidly exhausting the entire contents of the chamber. The bleeder is 2 1/2 in. galvanized pipe connected with the chamber at one corner near the ceiling. It is fitted with a gate valve and empties into a duct connecting with the large fan which constantly creates a strong draft up the stack at one end of the laboratory. The blower, a Sirocco No. 2, is connected with the chamber at one corner near the floor (Fig. XII) by a 6 in. pipe provided with a gate valve. The blower is driven by a 3/4 h. p. motor at a speed of 700 r. p. m. It is connected so that it draws the gases out of the chamber and delivers them to the large ventilating fan through a separate duct to avoid the corrosive action of the gas on the entire ventilating system.

A sensitive U-tube water manometer with an in-

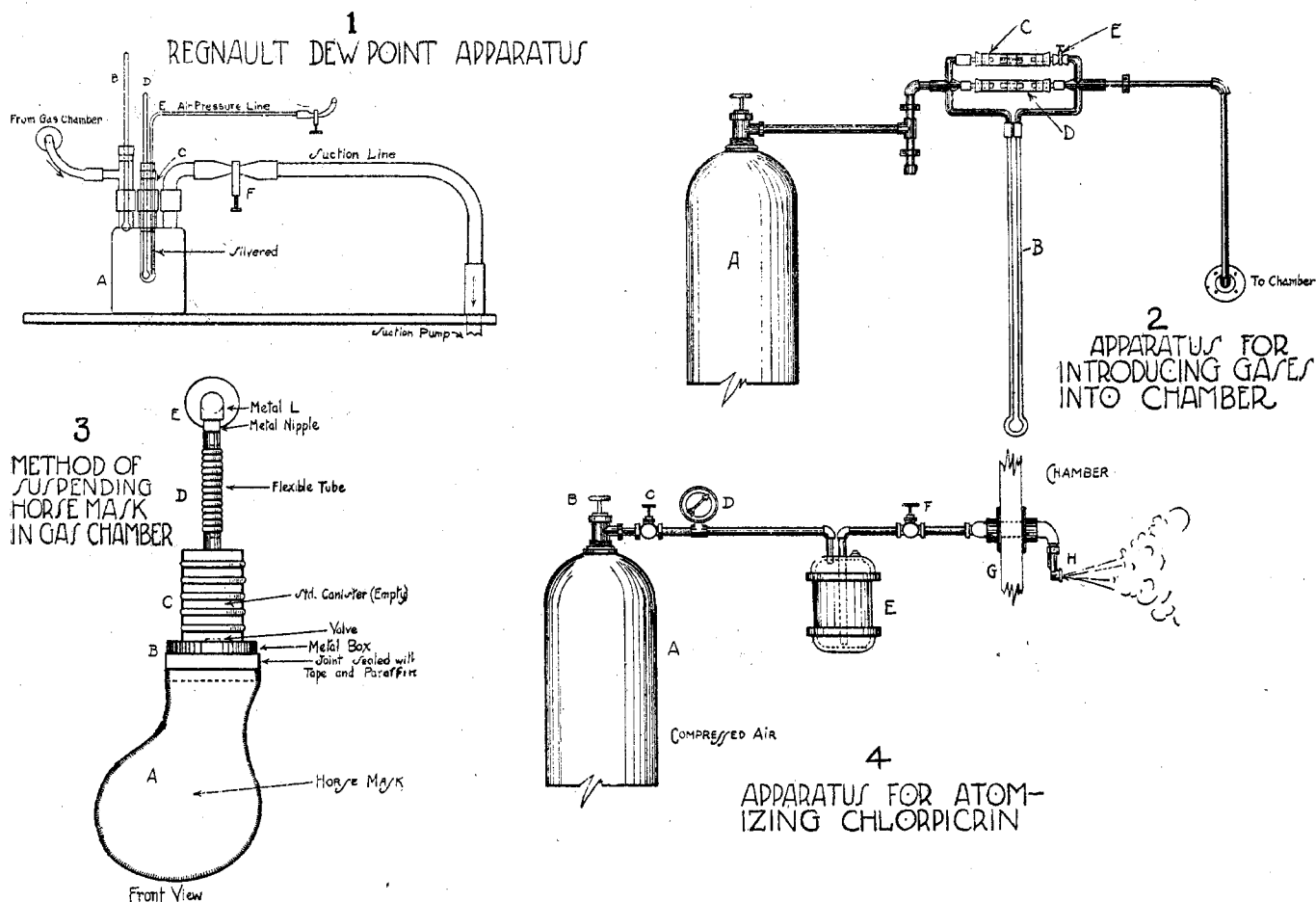


FIG. XIV—APPARATUS FOR INTRODUCING GASES AND DETERMINING DEW POINT

clined arm is connected with the chamber mounted on the wall beneath the window. It records either pressure or suction, and may be read directly to tenths of an inch of water. When running tests, the usual procedure is to open the gate valve of the bleeder until the manometer indicates a pressure within the chamber very slightly less than that of the air outside, thus preventing leakage of the gases out into the laboratory. The gate valve of the large blower is kept tightly closed except when clearing out the chamber after a test.

Other general features of the equipment of each chamber deal with the determination of the physical conditions surrounding the tests, often a matter of considerable importance. The temperature of the gas inside the chamber is easily ascertained by means of a thermometer suspended inside the window in such a position as to be read from the outside. The relative humidity of the mixture of air and gas in the chamber is determined by means of a somewhat modified Regnault dew point apparatus (see Fig. XIV-1) mounted on the built-in table. This apparatus consists of a 3-necked Wolf bottle, A, one opening of which is connected directly to the interior of the chamber and another to the intake pipe of the air pump beneath the table, so that by regulating the valve F, a steady stream of the air-gas mixture may be drawn through the bottle. A thermometer, B, measures the temperature of the mixture as drawn

from the chamber. The central opening of the bottle is closed by a thin-walled test-tube, C, which has been silvered on the inside for a distance of about  $1\frac{1}{2}$  in. from the bottom. A second thermometer, D, extends down into this tube, also a small glass tube, E, drawn out to a capillary and connected to the pressure line of the pump. To operate the apparatus, the test-tube C is partly filled with ether, and the valve F opened, drawing in air and gas from the chamber. Air from the pressure line E is then blown through the ether until it is cold enough to produce a deposition of moisture on the surface of the test-tube, easily visible against the silvered background. The thermometer D immersed in the ether gives a dew point temperature, and the thermometer B the temperature of the air-gas mixture entering the bottle, from which data the relative humidity may be calculated, using the tables furnished in Bulletin 235, of the U. S. Weather Bureau.

It is necessary that the test-tube should be silvered on the inside, as the gases would quickly tarnish the mirror if placed on the outside. The tendency of the bubbling ether to mechanically strip off the thin layer of silver is prevented by protecting it with a very thin coat of water glass (sodium silicate). The apparatus is frequently checked against readings made in the chamber, using a standard sling psychrometer.

SPECIAL APPARATUS HUMIDIFIER—Other items of

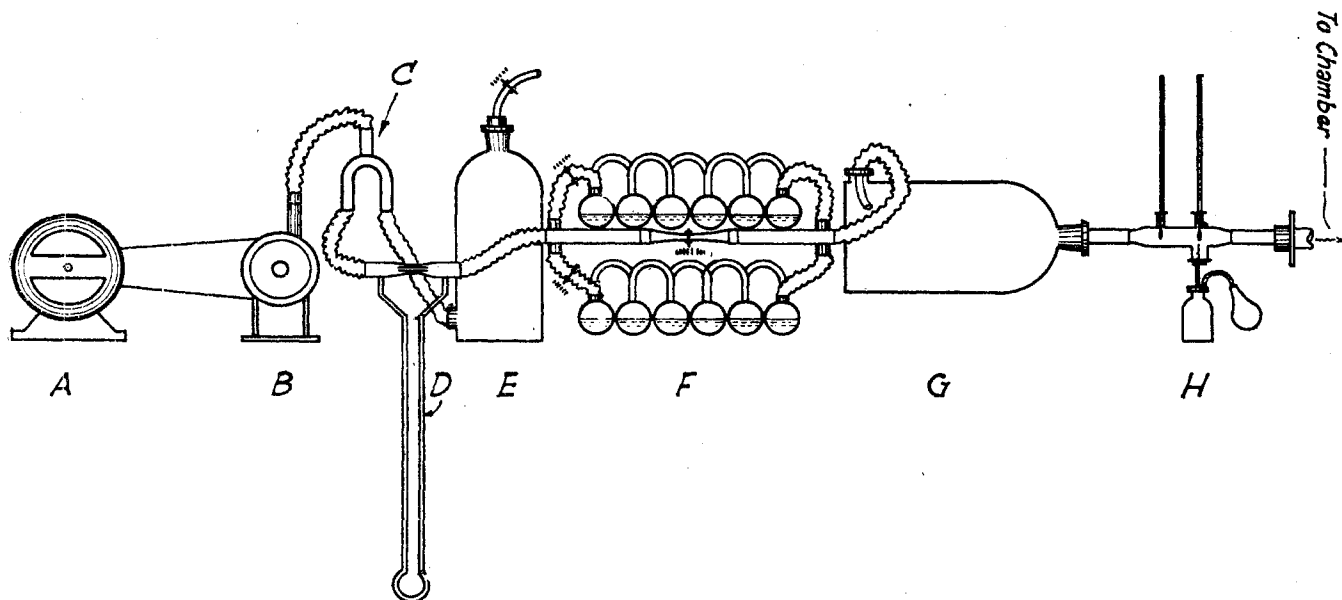


FIG. XV—APPARATUS FOR CONTROLLING HUMIDITY OF AIR IN GAS CHAMBERS

general equipment for the laboratory include a device for humidifying or drying air (Fig. XV) which may be attached to any of the pumps for delivering air of any desired degree of humidity to the chambers. Half the tubes are filled with distilled water, and the other half with concentrated sulfuric acid; by means of a set of clamps, the air from the pump may be sent directly into the chamber or may be shunted through either the drying or moistening tubes, giving a range of from 10 per cent to 90 per cent relative humidity. A wet-and-dry-bulb thermometer installed in the line affords a constant check on the moisture content of the air being forced into the chamber.

**PRESSURE DROP AND LEAK DETECTING APPARATUS—**Another piece of apparatus consists of a combined pressure drop machine and leak tester (Fig. XVI) for measuring the resistance of canisters and testing them for faulty construction. This is mounted on a small table, with the motor and air pump installed on a shelf underneath. The resistance, or pressure drop, of canisters is measured by the flow meter A and the water manometer B. Air is drawn through the canister and the flow meter A at the rate of 85 l. per min., the flow being adjusted by the needle valve. The pressure drop across the canister is read on the water manometer B, one end of which is connected to the suction line, the other open to the air. The reading is generally made in inches, correction being made for the resistance of the connecting hose and the apparatus itself.

Canisters are tested for leaks by the apparatus shown at D in Fig. XVI. The canister is clamped down tightly by wing nuts against a piece of heavy  $\frac{1}{4}$ -in. sheet rubber large enough to cover completely the bottom of the canister and prevent any inflow of air through the valve. Suction is then applied, and a leak is indicated by a steady flow of air bubbles through the liquid in the gas-washing cylinder E. A second gas-washing cylinder, empty, is inserted in the line between E and the canister as a trap for any liquid

drawn back when the suction is shut off. If a leak is shown, it is located by applying air pressure to the canister and then immersing it in water.

In addition to the general equipment so far described, the several chambers are fitted with certain devices for aiding in the particular work for which they are used. These can perhaps best be described by explaining the methods followed for handling the gas most commonly employed in each chamber, although the procedure is typical of a class in every case, except for slight variations.

**DETONATION OF SOLIDS AND LIQUIDS—**The chamber at the east end of the house is used for those gases

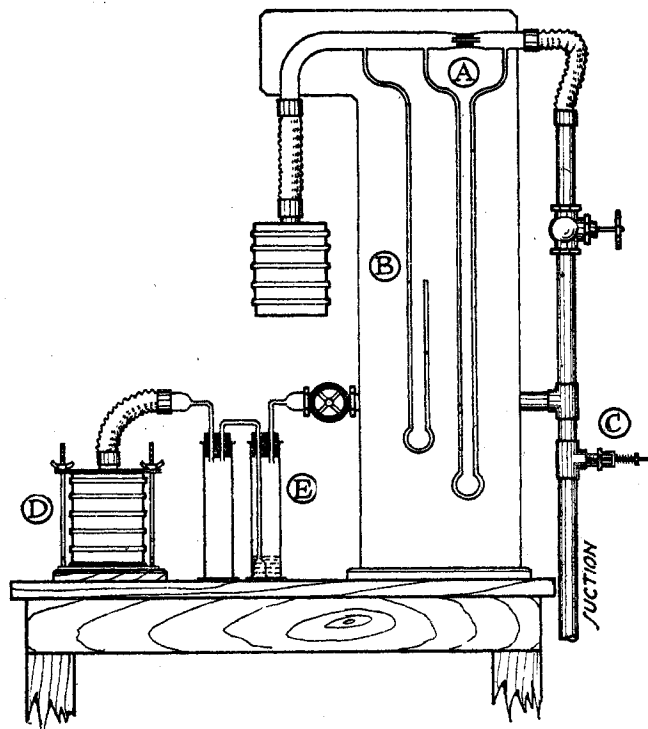


FIG. XVI—APPARATUS FOR DETERMINING PRESSURE DROP AND FOR DETECTING LEAKS IN CANISTERS

whose concentrations are secured by detonating small amounts of the solid or liquid substances. Typical of this class is diphenylchlorarsine. The concentration of this compound is secured by detonating the solid substance, using No. 6 electric detonators enclosed in the gelatin capsules containing the solid. At the beginning of a test, a calculated weight of diphenylchlorarsine sufficient to give the desired concentration is detonated, and every 10 min. thereafter an additional amount of the substance is detonated to reënforce the concentration. Five of these reënforcements are made at 10-min. intervals, and the test allowed to run 10 min. after the last detonator is exploded. Tests of this nature are usually run for 1 hr. The weights necessary to produce the desired concentrations are calculated from the gram-molecular volume of the substance, assuming that a gram-molecular weight of any substance in true gaseous form occupies 24.45 l. under average laboratory conditions.

The gelatin containers holding the compounds to be detonated are No. 10 veterinary capsules of 1 oz. capacity.

The detonators are placed directly inside the capsules, the lids being perforated by melting a hole through the gelatin, using a heated iron rod. A small loop is made in the lead wires just above the blasting cap. This serves to hold the capsule snugly up against the detonator. After the two halves of the capsule have been fitted together, they are held by a short strip of adhesive tape to prevent the jar of other explosions from knocking them apart. Fig. XVII-1 illustrates the appearance of a filled capsule and its detonator ready for use.

In order to protect the chamber walls, canisters, connecting hose, etc., from flying particles of the fulminate caps, which are driven off with great force, guards are provided, a separate one for each detonator, or six in all. These are made of 12-gauge sheet iron (nothing lighter will do) and are constructed as shown in Fig. XVII-2. They are strung on a wire extending through the middle of the chamber from the top of the door casing to the casing of the window opposite, being spaced about 15 in. apart and at the same height from the floor as the canisters to be tested, *viz.*, about 50 in. A system of permanent wiring connects the guards with the exterior of the chamber. Small insulated binding posts mounted on the upper sheet-iron discs make it possible to use short leads (18 in.) on the detonators, and to replace exploded ones rapidly and easily. Outside the chamber the wires connect with small insulated copper plates fastened to the wall and numbered to correspond with numerals painted on the detonator guards, so that the capsules may be detonated in any desired order. Fig. XVII-3 illustrates the method of wiring used.

**SUBSTANCES GASEOUS AT ORDINARY TEMPERATURES—**The middle chamber is used for those substances which are gases at ordinary temperatures and may be introduced into the chamber directly from the ordinary steel storage cylinder. Phosgene is the one most commonly used, and the description of the methods

employed for this gas may be taken as typical of the class. The special equipment of this chamber consists of flow meters particularly designed for getting up and maintaining the desired concentration. These flow meters are permanently attached to the wall, one on either side of the large window. They are constructed almost entirely of iron and copper piping and of glass tubing, joined either by solder or hard De Khotinsky cement, rubber connections being used only where indispensable. The arrangement of the larger flow meter is shown in Fig. XIV-1. It is constructed mainly of  $\frac{1}{4}$ -in. iron piping, with a union (not shown) for the rapid replacement of exhausted cylinders. A gas cylinder is shown in position at A; it is 4 ft. high and 10 in. in diameter. The long U-tube B for measuring level differences is of glass, as are the tubes holding the capillaries C and D. It will be noticed that the apparatus has two capillaries, the upper and larger of which, C, may be closed off at will by means of the clamp E. The other capillary, D, is much finer and gives a much smaller flow of gas for the same level difference in the U-tube. When getting up the initial concentration, both capillaries are open and the gas feeds in rapidly; after the approximate concentration desired has been reached, the larger capillary, C, can be shut off and a small, steady flow of gas maintained through the smaller one, D. For still finer adjustments, a second flow meter of very small capacity is built against the wall on the opposite side of the window. This one has connections of  $\frac{1}{8}$  in. piping, and is coupled by a union to a small 14 × 4 in. phosgene cylinder held in rigid bracket attached to the wall and resting on a small electric hot plate, as it is sometimes necessary to warm the cylinders to secure sufficient pressure. The larger tank is warmed, in cold weather, by means of a gas flame. The U-tubes of both flow meters are filled with "Usoline," an inert material oil not affected by phosgene. With gases like chlorine, no heating of the cylinders is necessary.

In running tests with phosgene, the initial concentration is obtained by feeding in the gas through the large flow meter with both capillaries open; then a steady stream of both air and gas in the desired proportion is fed into the chamber continuously in an amount somewhat in excess of that removed by the men inhaling through the canisters. The Leiman pump mounted beneath the table delivers air through a flow meter into the chamber at the rate of 125 l. per min., while at the same time one or the other of the phosgene tanks is feeding in gas at the proper rate to give the concentration desired. The piping of both tanks is so arranged as to deliver the gas into the chamber just above the air inlet from the pump, insuring rapid mixing. As the canisters are usually tested in lots of ten at a time, and the average breathing rate of a man is about 8 l. per min., it will be seen that a considerable excess of air and gas is being constantly forced into the chamber. This excess is taken care of by the bleeder which carries the surplus mixture off into the stack. The manometer is used to assist in regulating the gate valve until the pressure

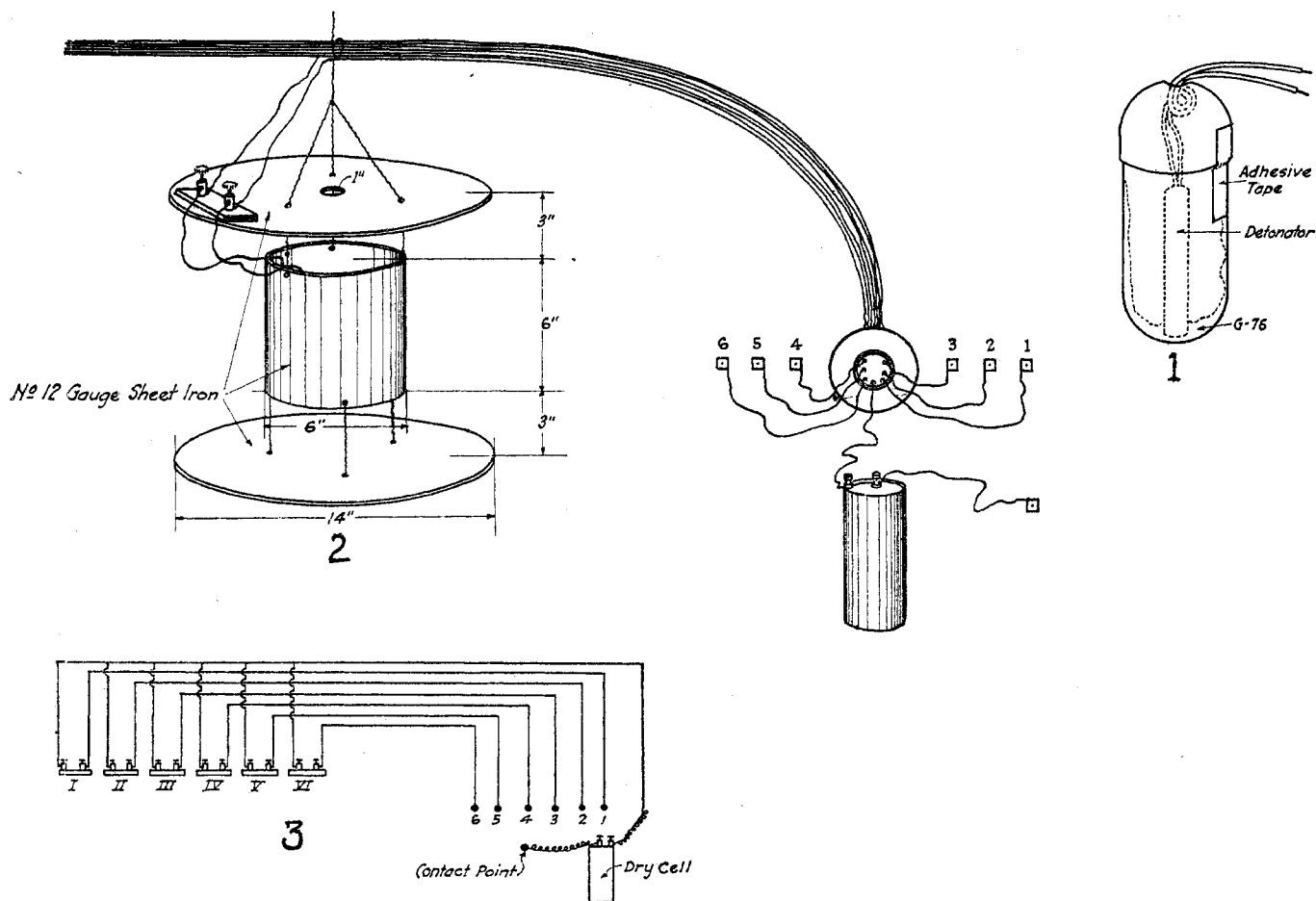


FIG. XVII—APPARATUS FOR DETONATING SOLID AND LIQUID SUBSTANCES

inside the chamber is very slightly less than that of the air outside. This prevents leaks.

**SUBSTANCES LIQUID AT ORDINARY TEMPERATURES—**  
The chamber at the west end of the laboratory is used for putting up concentrations of substances which are in a liquid form under ordinary conditions. This is accomplished by spraying or atomizing them in the chamber. Some atomizers have been specially constructed for the purpose. They have a large capacity and are made of glass. The nozzles are extra long (12 in. or more), extending through the wall and well into the chamber. These atomizers are operated by means of a foot bellows or an automobile foot pump. However, for atomizing chlorpicrin, which is the liquid most commonly used, an apparatus has been devised using compressed air, which works very successfully.

The same apparatus can be used for many other liquids. The arrangement is shown in Fig. XIV-4. A is a tank holding air at a pressure of 1800 lbs. per sq. in. controlled by valve B and a needle valve, C, of more delicate adjustment. The container for chlorpicrin, E, is made of a short section of pipe capped on both ends, with connections as shown. The gauge D indicates the pressure in the apparatus. The ball valve F allows the chlorpicrin to escape into the chamber through the nozzle H which is from an ordinary orchard sprayer and works on the centrifugal principle. To operate, the measured amount of

chlorpicrin is introduced into the container E, through a small hole at the top, which is then tightly closed by means of a screw plug. The ball valve F is closed and valve B opened; then by means of the needle valve C the pressure in E is raised to 150 lbs. per sq. in., as indicated by gauge D. The needle valve is then closed and valve F opened, whereby the chlorpicrin is expelled into the chamber in the form of a fine mist. The concentration is kept up by successive small amounts atomized at regular intervals or by a continuous flow of air and chlorpicrin in the proper proportions, as in the case of phosgene.

**UNIFORMITY OF GAS CONCENTRATION—**To test the efficiency of the fans in keeping a uniform concentration, a survey has been made of one of the chambers, using a concentration of approximately 11,000 p. p. m. of chlorine. After thorough mixing by the fans, 2 l. of the air-chlorine mixture were aspirated through each of the sampling tubes in order to displace all air in the tubes and connections, with the mixture to be analyzed. The absorption cylinders were then attached, and 1 l. samples of the contents of the chamber aspirated through them simultaneously at the rate of 1 l. per min. The analyses are given below. Samples 1 and 4 were taken near adjacent corners nearest the window, and almost directly over the fans; Samples 2 and 5 from the middle of each side; and Samples 3 and 6 from near the corners nearest the door and farthest from the fans.

SAMPLE No.	CONCENTRATION	
	P. p. m.	
1.....	11,325	
2.....	11,300	
3.....	11,230	
4.....	11,315	
5.....	11,275	
6.....	11,225	
MEAN.....	11,278	

The maximum variation from the mean was 53 p. p. m., or 0.5 per cent. It is assumed, therefore, that the concentration is practically uniform in all parts of the chamber.

**METHODS OF CONDUCTING TESTS**—Three general methods of conducting man tests are followed:

(1) Canisters are placed in the brackets outside the chamber or fastened to the wall tubes within the chamber. The subjects of the test remain outside the chamber, and the facepieces of the masks are connected directly to the canisters, in the first case, and to the wall tubes connecting with the canisters, in the second case. The concentration is established and the time noted. Then the men put on the masks and breathe until they can detect the gas coming through the canisters. Reading matter is provided for the men during the test period. When gas is detected, the time is again noted and the time required for the gas to penetrate the canister is reported as the "time to break down" or "service time" of the canister. Ten canisters are tested at one time, and the average of the results for the 10 canisters is taken for that type of canister. Much less accurate results are obtained when the final figure is based on a small number of canisters. This is largely due to the various breathing rates and sensitivities of different men.

(2) The canisters are placed as in (1), but it is only necessary to know if they will give perfect protection for a given length of time. The procedure is the same as in (1), except that the test is arbitrarily stopped at the end of the indicated time, and the number of canisters and the service times of the same noted.

(3) When the canisters are of such a type that they cannot be properly tested as in (1), or when it is desired to test the penetrability of the facepiece, the men wear the complete mask and enter the chamber. They remain until gas penetrates the canister or the facepiece, as the case may be, or until it is determined that the desired degree of protection is afforded. The service time is computed as in (1).

(4) Maximum-breathing-rate tests are made either by men in the chamber or by the men outside, in which they do vigorous work on a bicycle ergometer. In this test the average man will run his breathing rate up to 60 or 70 l. per min.

The concentration of the gas is followed throughout the test by aspirating samples and analyzing them.

**TYPE OF MASKS USED**—In general, the following procedure holds, although variations occur in special cases:

When men enter a gas chamber, the full facepiece is, of course, required. The type of facepiece will be determined by the nature of the gas. If the gas is most easily detected by odor or eye irritation, a modified Tissot mask is used. If it is most easily detected by throat irritation, a mouth-breathing mask is employed.

When men are outside the chamber, the choice is made in the same manner, except in the case of detection of the gas by throat irritation. In this case the mouthpiece is attached to two or three lengths of breathing tubes and a separate nose-clip is used. The facepiece is not needed and the men are much more comfortable without it.

**DISINFECTION OF MASKS**—Mouthpieces are disinfected after use by first holding them under a stream of running water and brushing out thoroughly with a test-tube brush; then the latter is dipped into a 2 per cent solution of lysol, and the inner parts of the mouthpiece are brushed out well; finally the mouthpiece and exhaling valve are dipped bodily into the lysol solution and allowed to dry without rinsing. Tissot masks are wiped out with a cloth moistened in alcohol, followed by another cloth moistened in 2 per cent lysol solution. The flexible tubes are given periodic rinsings with 95 per cent alcohol.

**APPLICABILITY OF MAN TESTS**—Man tests are applicable to all gases which can be detected by the subject of the test before he breathes a dangerous amount.

The man test laboratory described in this report provides facilities for getting information concerning the efficiency of canisters, facepieces, etc., within very short periods of time, without waiting for the construction of special apparatus required for machine tests. To get satisfactory results from machine tests, a delicate qualitative chemical test for the gas is essential. Man tests can be made when such a qualitative test is not known. Further, man tests can be made with higher concentrations of some gases than is practicable with machines. Evolution of excessive amounts of moisture when high concentrations of some gases are used causes much more trouble with machine tests than with man tests.

On the other hand, man tests are adversely affected by the varying sensitivities and lung capacities of the men, and the humidity of the air-gas mixture is not subject to as exact control as is the case with machine tests.

The development of the foregoing methods was started by the Bureau of Mines in April 1917, immediately after this country entered the war. At that time no information whatever was available on how to grade and test gas-mask absorbents, or the masks themselves. The early development work was done in the Bureau of Mines Gas Laboratory at the Pittsburgh Station, and, as a matter of fact, most of the methods were completely worked out before any information from abroad became available.

While many improvements were made after interchanging information with the British and French laboratories, the methods remained essentially the same as originally devised. Subsequent improvements were mainly along the lines of greater standardization, more rapid manipulation, and extending the tests to all sorts of new gases.