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HISTORY OF THE CHEMICAL WARFARE SERVICE IN THE UNITED STATES. Part I.

By

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AMERICAN UNIVERSITY EXPERIMENT STATION.

MAY 30, 1919

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The use of poison gases by the Germans at Ypres on April 22, 1915, marked a new era in modern warfare. The popular opinion is that this form of warfare was original with the Germans. Such, however, is not the case. According to an article in the Candid Quarterly Review, "All they can claim is the inhuman adoption of devices invented in England, and by England rejected as too horrible to be entertained even for use against an enemy". The use of poison gases is even earlier in origin than this article claims.

(1) Called to my attention by Major C.J. West.
(2) Vol. 4, p.561(1915)

The first recorded effort to overcome an enemy by the generation of poisonous and suffocating gases seems to have been in the wars of the Athenians and Spartans (431-404 B.C.) when the Spartans besieged the cities of Platea and Belium. They saturated wood with pitch and sulphur and burned it under the walls of these cities in the hope of choking the defenders and rendering the assault less difficult. Similar uses of poisonous gases are recorded during the Middle Ages. In effect they were like our modern stink balls, but were projected by squirts or in bottles after the manner of a hand grenade. The legend is told of Prester John (about the eleventh century), that he stuffed copper figures with explosives and combustible materials which caused great havoc when emitted from the mouths and nostrils of the effigies.

The idea referred to by the writer in the Candid Quarterly is due to the English Admiral Lord Dundonald, and appeared in the publication entitled "The Panmure Papers".
"The great Admiral Lord Dundonald — perhaps the ablest
sea captain ever known, not even excluding Lord Nelson — was also
a man of wide observation, and no mean chemist. He had been struck
in 1811 by the deadly character of the fumes of sulphur in Sicily;
and, when the Crimean War was being waged, he communicated to the
English Government, then presided over by Lord Palmerston, a plan
for the reduction of Sebastopol by sulphur fumes. The plan was
imparted to Lord Panmure and Lord Palmerston, and the way in which
it was received is so illustrative of the trickery and treachery
of the politician that it is worth while to quote Lord Palmerston's
private communication upon it to Lord Panmure:

LOD PALMERSTON TO LORD PANMURE.

"House of Commons, 7th August, 1855.

"I agree with you that if Dundonald will go out
himself to superintend and direct the execution of his scheme,
we ought to accept his offer and try his plan. If it succeeds,
it will, as you say, save a great number of English and French
lives; if it fails in his hands, we shall be exempt from blame,
and if we come in for a small share of the ridicule, we can bear
it, and the greater part will fall on him. You had best, therefore,
made arrangements with him without delay, and with as much
secrecy as the nature of things will admit of."

"Inasmuch as Lord Dundonald's plans have already been
deliberately published by the two persons above-mentioned, there can
be no harm in now republicating them. They will be found in the
first volume of The Panmure Papers, pp. 340-342 and are as follows:

"BRIEF PRELIMINARY OBSERVATIONS

"It was observed when viewing the sulphur. Ulms, in
July in 1811, that the fumes which escaped in the rude process of extracting the material, though first elevated by heat, soon fell to the ground, destroying all vegetation, and endangering animal life to a great distance, as it was asserted that an ordinance existed prohibiting persons from sleeping within the distance of three miles during the melting season.

"An application of these facts was immediately made to military and naval purposes, and after mature consideration, a memorial was presented on the subject to His Royal Highness the Prince Regent on the 12th of April, 1812, who was graciously pleased to lay it before a Commission, consisting of Lord Keith, Lord Exmouth, and General and Colonel Congreve (afterwards Sir Allan), by whom a favourable report having been given, His Royal Highness was pleased to order that secrecy should be maintained by all parties.

7th August, 1855"

"L. DONALDSON"

"Materials required for the expulsion of the Russians from Sebastopol:
Experimental trials have shown that about five parts of coke effectually vaporize one part of sulphur.
Mixtures for land service, where weight is of importance, may, however, probably be suggested by Professor Faraday, as to operations on shore I have paid little attention. Four or five hundred tons of sulphur and two thousand tons of coke would be sufficient."
"Besides these materials, it would be necessary to have, say, as much bituminous coal, and a couple of thousand barrels of gas or other tar, for the purpose of masking fortifications to be attacked, or others that flank the assailing positions.

"A quantity of dry firewood, chips, shavings, straw, hay, or other such combustible materials, would also be requisite quickly to kindle the fires, which ought to be kept in readiness for the first favourable and steady breeze."

Dundonald.

7th August, 1855.

"Note.—The objects to be accomplished being specially stated the responsibility of their accomplishment ought to rest on those who direct their execution.

"Suppose that the Zalakoff and Sedan are the objects to be assailed it might be judicious merely to obscure the Sedan (by the smoke of coal and tar kindled in the 'barries'), so that it could not annoy the Zalakoff, where the sulphur fire could be placed to expel the garrison from the Zalakoff, which ought to have all the cannon that can be turned towards its ramparts employed in overthrowing its undefended ramparts.

"There is no doubt but that the fumes will envelop all the defense from the Zalakoff to the barracks, and even to the line of battleship, the Twelve Apostles, at anchor in the harbour.

"The two outer batteries, on each side of the Port, ought to be smoked, sulphured, and blown down by explosion vessels, and their destruction completed by a few ships of war anchored under cover of the smoke."
"That was Lord Dundonald's plan in 1855, improperly published in 1908, and by the Germans, who thus learnt it, ruthlessly put into practice in 1915.

"Lord Dundonald's memoranda, together with further elucidatory notes, were submitted by the English Government of that day to a committee and subsequently to another committee in which Lord Playfair took leading part. These committees with Lord Dundonald's plans fully and in detail before them, both reported that the plans were perfectly feasible; that the effects expected from them would undoubtedly be produced; but that these effects were so horrible that no honorable combatant could use the means required to produce them. The committees therefore recommended that the theme should not be adopted; that Lord Dundonald's account of it should be destroyed. How the records were obtained and preserved by those who improperly published them in 1908 we do not know. Presumably they were found among Lord Panmure's papers. Admiral Lord Dundonald himself was certainly no party to their publication.

"Thus it will be seen that the plan which England had rejected as being too horrible for use in warfare has been, through the deplorable conduct of those who somehow obtained and published it, stolen from us by the Germans, and first used against us. That having been done, we cannot choose but retaliate in kind; for then such methods of warfare are used against we must, in our own protection and that of our soldiers, ourselves use means similar and as efficacious. Such means lie ready to our hand in official
Lord Dumbaldon's plans; and it is to be presumed that they are
now worked out and perhaps improved upon by the modern chemists
so as to enable us effectually to give back to the Germans as
good a gas as they sent us."

The writer in the Candia quarterly is wrong in saying
that the Germans learned of the British suggestion to use poisonous
gases in warfare from the publication of "The Bicentenary Papers" in
1868, while the complete details first became available then, a
reference to the matter was published in 1854, at which time a
gas mask had been developed to protect the British themselves
against poisonous gases. This was invented by Dr. J. Stenhouse
and was described by Dr. George Wilson. (1)


"Dr. Wilson commenced by stating, that having read with
much interest the account of Dr. Stenhouse's researches on the
deedorizing and disinfecting properties of charcoal, and the applica-
tion of these to the construction of a new and important kind of
respirator, he had requested the accomplished chemist to send
one of his instruments for exhibition to the Society, which he had
kindly done. Two of the instruments were now on the table, differing
however, so slightly in construction, that it would be sufficient
to explain the arrangement of one of them. Internally, it had
the appearance of a small funnel-shaped wire gauze, covering the
face from the chin upwards to the bridge of the nose, but leaving
the eyes and forehead free. It consisted essentially, of two plates
of wire gauze, separated from each other by a space of about 1/4
or 1/8 of an inch, so as to form a small cage filled with small
fragments of charcoal. The frame of the cage was of copper, but
the edges were made of soft lead, and were lined with velvet, so
as to admit of their being made to fit the cheeks tightly and
enclose the mouth and nostrils. By this arrangement, no air
could enter the lungs without passing through the wire gauze and
traversing the charcoal. An aperture is provided with a screw
or sliding valve for the removal and replenishment of the contents
of wood charcoal. The apparatus is attached to the face by an
elastic band passing over the crown of the head and striking	
behind, as in the case of the ordinary respirator. The important
aspect in this instrument is the charcoal, which has so remarkable
a power of absorbing and destroying irritating and otherwise ir-
respirable and poisonous gases and vapours that, armed with the
respirator, spirits of hartshorn, sulphuretted hydrogen, hydro-
sulphuret of ammonia, and chlorine may be breathed through it
with impunity, though but slightly diluted with air. This re-
sult, first obtained by Dr. Stenhouse, has been verified by those
who have repeated the trial, among others by Dr. Illson, who
has tried the vapours named above on himself and four of his pupils,
who have breathed them with impunity. The explanation of this
remarkable property of charcoal is two-fold. It has long been
known to possess the power of condensing into its pores gases
and vapours, so that if freshly prepared and exposed to these, it
absorbs and retains them. But it has scarcely been suspected until
recently, when Dr. Stenhouse pointed out the fact, that if charcoal be allowed to absorb simultaneously such gases as sulphuretted hydrogen and air, the oxygen of this absorbed and condensed air rapidly oxidizes and destroys the accompanying gas. So marked is this action, that if dead animals be imbedded in a layer of charcoal a few inches deep, instead of being prevented from decaying as it has hitherto been supposed that they would be by the supposed antiseptic powers of charcoal, they are found by Dr. Stenhouse to decay much faster, whilst at the same time, no offensive effluvia are evolved. The deodorizing powers of charcoal are thus established in a way they never have been before; but at the same time it is shown that the addition of charcoal to sewage refuse lessens its agricultural value contemporaneously with the lessening of odor. From these observations, which have been fully verified, it appears that by strewing charcoal coarsely powdered to the extent of a few inches, over church-yards, or by placing it inside the coffins of the dead, the escape of noisome and poisonous exhalations may be totally prevented. The charcoal respirator embodies this important discovery.

It is certain that many of the miasmata, malaria, and infectious matters which propagate disease in the human subject, enter the body by the lungs, and impregnating the blood there, are carried with it throughout the entire body, which they thus poison. These miasms are either gases and vapours, or bodies which, like fine light dust, are readily carried through the air; moreover, they are readily destroyed by oxidizing agents, which convert them into harmless, or at least non-poisonous substances, such as water,
carbonic acid and nitrogen. There is every reason, therefore, for believing that charcoal will oxidize and destroy such miasmata as effectually as it does sulphuretted hydrogen or hydrosulphuret of ammonia, and thus prevent their reaching and poisoning the blood.

The intention accordingly is that those who are exposed to noxious vapours, or compelled to breathe infected atmospheres, shall wear the charcoal respirator, with a view to arrest and destroy the volatile poisons contained in these. Some of the non-obvious applications of the respirator were then referred to:-

1. Certain of the large chemical manufacturers in London are now supplying their workmen with the charcoal respirators as a protection against the more irritating vapours to which they are exposed.

2. Many deaths have occurred among those employed to explore the large drains and sewers of London from exposure to sulphuretted hydrogen, etc. It may be asserted with confidence that fatal results from exposure to the drainage gases will cease as soon as the respirator is brought into use.

3. In districts such as the Campagna of Rome, where malaria prevails and to travel during night or to sleep in which is certainly followed an attack of dangerous and often fatal ague, the wearing of the respirator even for a few hours may be expected to render the marsh poison harmless.

4. Those, who as clergymen, physicians, or legal advisers, have to attend the sick-beds of sufferers from infectious diseases, may, on occasion, avail themselves of the protection afforded by Dr. Stenhouse's instrument during their intercourse with the sick.

5. The longing for a short and decisive war has led to the invention of a "suffocating bomb-shell," which on bursting, spreads far and wide much respirable or poisonous vapour; one of the liquids proposed for the shell is the strongest ammonia, and against this it is believed that the charcoal respirator may defend our soldiers. is likely to serve this end, it is at present before the Board of Ordnance.

"Dr. Wilson stated, in conclusion, that Dr. Stenhouse, had no interest but a scientific one in the success of the respirators. He had declined to patent them, and desired only to apply
his remarkable discoveries to the abatement of disease and death.

Charcoal had long been used in filters to render poisonous water
wholesome; it was now to be employed to filter poisonous air."

I have not been able to find any reference in the
scientific literature as to the action taken by the English
Government in regard to Stonhouse's mask; but the quotation from
the Candid Quarterly clears up the matter. Since poisonous gases
were not to be used, it was unnecessary to provide a defense against
them.

One of the early, if not the earliest, suggestion as
to the use of poison gas in shell is found in an article on "Greek
Fire" by B. W. Richardson (Popular Science Review, 2, 176 (1864):
He says:
(1) Popular Science Review, 2, 176 (1864).

"I feel it a duty to state openly and boldly, that
if science were to be allowed her full swing, if society
would really allow that 'all is fair in war', war might be
banished at once from the earth as a game which neither
subject nor king dare play at. Gases that could distribute
liquid fire could distribute also lethal agents, within the
breath of which no man, however puissant, could stand and live.

From the summit of Primrose Hill, a few hundred yards,
properly prepared, could render an agent's Park, in an incredibly
short space of time, utterly uninhabitable; or could make an
army of men, that should even fill that space, fall with their
arms in their hands, prostrate and helpless at the host of
Commacherib."
"The question is, shall these things be? I do not see that humanity should revolt; for would it not be better to destroy a host in Regent's Park by making the men fall as in a mystical sleep, than to let down on them another host to break their bones, tear their limbs asunder, and gouge out their entrails with three-cornered pikes;— leaving a vast majority undisturbed, and writhing for hours in torments of the damned? I conceive, for one, that science would be blessed in spreading her wings on the blast, and breathing into the face of a desperate horde of men prolonged sleep—for it need not necessarily be a death—which they could not grapple with, and which would yield them up with their implements of murder to any enemy that in the intensity of its power could afford to be as merciful as Heaven.

"The question is, shall these things be? I think they must be. By what compact can they be stopped? It were improbable that any congress of nations could agree on any code regulating means of destruction; but if it did, it were useless; for science becomes more powerful as she concentrates her forces in the hands of units, so that a nation could only act, by the absolute and individual assent of each of her representatives. Assume, then, that France shall lay war to England, and by superior force of men should place immense hosts, well armed, on English soil. It is probable that the units would rest in peace and allow sheer brute force to win
its way to expire? Or put English troops on French soil, and reverse the question?

"To conclude, war has, at this moment, reached, in its details, such an extravagance of horror and of cruelty, that it cannot be made worse by any art, and can only be made more merciful by being rendered more terribly energetic. Who that had to die from a blow would rather place his head under Nasmyth's hammer, than to submit it to a drummer-boy armed with a ferule?"

The Army and Navy Register of May 29, 1915, reports that "among the recommendations forwarded to the Board of Ordnance and Fortifications there may be found many suggestions in favor of the asphyxiation process, mostly by the employment of gas: contained in bombs to be thrown within the lines of the foe, with varying effects from peaceful slumber to instant death. One ingenious person suggested a bomb laden to its full capacity with snuff, which should be so evenly and thoroughly distributed that the enemy would be convulsed with sneezing, and in this period of paroxysm it would be possible to creep up on him and capture him in the throes of this convulsion."

That the use of poison gases was not new in the minds of military men follows logically from the fact that at the Hague Conference in 1899, the Governments represented - and all the warring powers of the present great conflict were represented - pledged themselves not to use any projectile whose only object was to give
out suffocating or poisonous gases. At the Congress of 1907, article 23 of the rules adopted for war on land states:

"It is expressly forbidden (a) to employ poisons or poisonous weapons."

It is interesting to note in this connection that the American and German delegates to the 1899 Congress --- refused to commit their countries against the use asphyxiating gas in projectiles (Literary Digest, 50, 1064, (1915).

Admiral Dahan's memorandum of his reasons for opposing the provisions is, in part, as follows:

"No shell emitting such gases is as yet in practical use or has undergone adequate experiment; consequently, a vote taken now would be taken in ignorance of the facts as to whether the results would be of a decisive character, or whether injury in excess of that necessary to attain the end of warfare, or immediately disabling the enemy, would be inflicted.

"The reproach of cruelty and perfidy addressed against these supposed shells was equally uttered previously against small arms and torpedoes, although both are now employed without scruple."

"It is illogical and not demonstrably humane to be tender about asphyxiating men with gas, when all were prepared to admit that it was allowable to blow the bottom out of an ironclad at midnight, throwing four or five hundred men into the sea to be choked by the water, with scarcely the remotest chance to escape."
Before the war suffocating cartridges were shot from the
26 mm. carriage-thrusting rifle 62-86 mm. These cartridges were charged
with ethyl bromacetate, a slightly suffocating and non-toxic
lachrymatory. They were intended for attack on the flanking works
of permanent fortifications, flanking casemats or caponiers, into
which they tried to make these cartridges penetrate by the narrow
slits of the loopholes. The men who were serving the machine guns
or the cannon of the flanking works would have been bothered by the
vapor from the ethyl bromacetate, and the assailant would have
profited by their disturbance to get past the obstacle presented
by the fortification. The employment of these devices, not en-
tailing death, did not contravene the Hague conventions.

The first gas attack of the present war was launched by
the Germans at Ypres on April 22, 1915. It is probable that plans
for this attack had been under way for months, at least from Christmas,
1914. The introduction of toxic gases in modern warfare is ascribed
by Colonel to Professor Walther Kernst of the University of Berlin,
while the actual field operations are said to have been under the

(1) Gas and Flame, 15, (1918)

general direction of Professor Haber of the Kaiser Wilhelm Physical
Chemical Institute of Berlin. Some writers have felt that the
question of preparation had been a matter of years, rather than
months, and refer to the work on industrial gases as a proof of
this statement. The fact that the gas attack was not more successful,
that the results to be obtained were not more appreciated, and that
better preparation against retaliation had not been made, argues
against this idea of a long period of reparation. That such was
the case is most fortunate for the Allied cause, for had the German
high command known the real situation at the close of the first
gas attack, or had it been more severe, the outcome of the war of
1914 would have been very different.

The first suggestion of the gas attack of April 22nd came
to the British Army through the story of a German deserter. He
told that the German Army was planning to poison their enemy with
a cloud of gas, and that the cylinders had already been installed
in the trenches. No one believed the story, largely because, in
spite of the numerous examples of German barbarity, the English
did not believe the German capable of such a violation of the Hague
rules of warfare, even though he had not signed their agreement.
The story appeared in the summary of information headquarters
("Comic Cuts") and as bold says "was passed for information for
what it is worth". But the story was true, and on the afternoon of
the 22nd of April, all the conditions being ideal, the beginning of
"gas warfare" was launched. Details of that first gas attack will
always be nearer, for the simple reason that the men who could
have told about never came back.
In this attack the Germans made use of chlorine discharged from steel cylinders, presumably because chlorine was a volatile, corrosive substance which was available on a large scale. In the course of events, chlorine reacts so readily with most substances that it can be stopped very easily, which was very fortunate because it thus became possible for the Allies to provide protection in a very short time. Cloth bandages soaked in the sodium hypochlorite solution of the photographers and wrapped over the nose and mouth give fairly satisfactory protection against chlorine. These bandages do not protect the eyes and the German promptly sent over a tear-gas bomb or ligerade in small bottles, the substance used for this purpose, bromoacetone and piperidine, are liquids at ordinary temperatures and do not pass into vapor rapidly enough to be used in cylinders. In everyday life we distinguish sharply between gas, liquid and solid; but in this instance there is no sharp line between these three states, and the Germans in particular to designate any substance, solid, liquid or gas which is important in cases in which irritants the lung, eye or skin. Protection against tear gas was also given by means of eye-pieces and later by special masks. The masks could be operated with ease.

Chlorine was used in the gas attack at the Battle of the Aisne from April to June, 1915, but in September of that year chlorine of smaller volume, HCl, was made use of. The new gas, perchloric, was not so easily done in cylinders, since it was little out of the normal size, for it was vaporized sufficiently rapidly. The first time it was introduced, it was over four times its point of chlorine, the latter gas being on the same cylinder, and rapidly caused a great deal of harm in cylinders, a roar to stop the receiving. The German troops then took still more dangerous.
the first French masks were impregnated with a solution of oxalic acid and acetone (acetone oxide tetrachloride). The author mentions that these masks were employed before long to protect the nose from the noxious fumes of the chemical warfare gas. The air breathed was passed first through the canister which contained finely divided pyroxyline powder and charcoal, both of which stop gases more effectively than the solution in the form of the mask.

The next improvement in the French use of chloropicrin in 1915, the liquid mask, resulted from the invention of the canister in the mask. Chloropicrin, CCl₃H₂O, was a liquid cold at 115°, which is much less toxic than phosgene, but has in distinctly less veno and the canister ventilating the mask made it easier for the wearer to keep on the mask. The main advantage is that it is not stopped by the gas mask itself, that is the time in the mask. The main benefit of the canister was improved protection and the canister was to go.

The introduction of the canister, chloropicrin was used in chlorine, carbon disulfide, and phosgene as well as other gas warfare agents. If the French had had the tactical use of poisonous gases worked out, before the war, the material could have delayed until the renewal of 1915, before using phosgene until 1917, when chloropicrin. If they had started in 1915, with chloropicrin, it could have been absolutely impossible for the Allies to have devised any protection in their conditions. The chemical warfare was developing the principles of warfare, of the conditions created, while the other forces, until a long of...
the Allies to be the first to try out each new gas, they were never able to turn their temporary advantage into victory.

This is seen very strikingly in the development of mustard gas, so-called, and of toxic smoke. In July 1917 the Germans began using mustard gas, a liquid boiling at about 170°C which stabs the skin, causing the passage of a painful condition for a number of weeks. While the mask protects the face satisfactorily, the problem of protecting the body only is a very serious one. Special garments and special clothing were devised at once, but these were not really satisfactory at the time the mustard gas appeared, and the cost protection was not to keep troops long in ground areas. Fortunately the Germans did not discover a method of making these gas and their supply was consequently always limited. It may not be long before we hear of a very differently serving gas, in 1915.

It had always been thought that a mixture of these gases would make a gas which would be necessary to put into action in order to keep static electricity from setting them off. The results were not very satisfactory, the gas being very likely divided into parts where it was not all developed with the desired amount of static electricity. This is a problem to the gas levels, which were not discovered as a very long term, the use by means of an explosive.

In order to protect effectively from gas, special filters are necessary. To equip the soldiers for protection against the effects, it was necessary to have on the filter a gas or inert gas to absorb the gas from the air.
Chlorine, \( \text{Cl}_2 \), was first used in April, 1915. It is a greenish-yellow gas with a suffocating and irritating smell. It boils at \(-35.6^\circ\), and the vapor pressure of liquid chlorine is 3.66 atmospheres at 0\(^{\circ}\) and 11.5 at 40\(^{\circ}\). The molecular weight is 71, so that the vapor is nearly 2.5 times as dense as air (71/28.6). The density of liquid chlorine is 1.4605 at 0\(^{\circ}\) and 1.4105 at 20\(^{\circ}\). It is easily prepared and easily liquefied. It is so volatile that it can be used in a cylinder or cloud attack. To prevent the cooling of the cylinders the discharge tube runs to the bottom as in a soda lighter; and evaporation takes place outside the cylinder.

Chlorine is not very toxic, the lethal concentration being 2.5 mg per liter (770 p.p.m.) for dogs on 30 minutes exposure. It is very corrosive but reacts so readily with most things that it is easily stopped. In the canister with the soda-lime it reacts directly with the soda-lime. It is also absorbed by charcoal and reacts with moisture, according to the equation:

\[
2\text{Cl}_2 + 2\text{H}_2\text{O} = 2\text{HCl} + 2\text{HClO} = 4\text{HCl} + \text{O}_2
\]

the hydrochloric acid being taken up by the soda-lime. Dry chlorine does not react with iron and can therefore be kept in steel cylinders.

Phosgene, \( \text{COCl}_2 \), was first used in December, 1915. It is a colorless gas with a smell like musty hay. It boils at 6.2\(^{\circ}\) and the vapor pressure of liquid phosgene is 1.6 atmospheres at 20\(^{\circ}\) and 3.1 at 40\(^{\circ}\). It is over three times as dense as air. The density of
the liquid is 1.432 at 0°. It is not sufficiently volatile to be
used by itself in cloud attacks and is always mixed with chlorine
in such cases, the mixture usually containing 20 - 25% phosgene.
It is about seven times as toxic as chlorine, the lethal concentra-
tion for dogs on 30 minute exposure being 0.36 mg. per liter (50 p.p.m.)
Phosgene reacts readily with water according to the equation

\[ COCl_2 + H_2O = CO_2 + 2HCl. \]

In the canister, phosgene is absorbed by charcoal and reacts
with the moisture in the latter to form carbon dioxide and hydrochloric
acid, which are taken up by the soda-lime. Soda-lime does not absorb
or decompose phosgene sufficiently rapidly to give adequate protection.
The charcoal gives the activity and the soda-lime the capacity. In-
creased moisture in the charcoal increases its efficiency towards
phosgene.

Chloropicrin, CCl_4CHCl_3, is colorless liquid, boiling at
112°, and having a vapor pressure of 1.6 mm at 0°, 14.0 mm at 100°,
and 23.9 mm at 20°. The vapor is nearly six times as dense as
air. The density of the liquid is 1.6056 at 4° and 1.5399 at 20°,
the two determinations not being made by the same man. The melting
point is -42°. Chloropicrin is not sufficiently volatile for use
by itself in cloud attacks. While it has been used mixed with 75%'
chlorine, it is usually fired in shell. It is moderately toxic
(0.8 mg per liter, 110 p.p.m.); somewhat lachrymatory (0.16 mg
per liter) and liable to cause vomiting, thus forcing removal of the
mask. It was stopped satisfactorily by the charcoal used in the
masks. The present laboratory charcoal is about one thousand times
as effective as the earlier material.
Trichloromethylchloroformate, CCl₃COC₁₂, is called diphenylone by the British, surpalite by the French, and supervalite by the Americans. It is a colorless, mobile liquid with a faintly pleasant sweet odor. It boils at 128°C and has a vapor pressure of 1.4 mm at 0°C and of 14.7 mm at 20°C. The vapor is over seven times as dense as air and is twice as dense as phosphine. The density of a pure sample of the liquid is 1.607 at 0°C and 1.656 at 20°C.

Owing to its low volatility supervalite is used only in shell. It has been used only by the Germans, who mark these shells with a green cross. The shells usually contain mixtures of supervalite and phosphine, though some duds have been found containing supervalite and chloroform; diphenylchloroformate has also been found in some of the Green Cross shells.

The lethal concentration for dogs is 0.5 mg per liter for exposure of 45 minutes (940 ppm) but it is much lower on longer exposures. Supervalite over its importance to its high vapor density, to its persistence because of its high-boiling-point, and to the increased toxicity on long exposures.

Supervalite is hydrolyzed slowly by water at room temperature. If heated rapidly at 100°C, the products being HCl and CO₂, presumably according to the equation:

$$\text{CCl₃COC₁₂} + 2\text{H₂O} = 4\text{HCl} + 2\text{CO₂}$$

Boiling with an aqueous solution of sodium hydroxide for half an hour decomposes it completely.

Alumina causes supervalite to decompose into carbon tetrachloride and carbon dioxide, while iron oxide and charcoal decomposes it to phosphene.
Some preliminary experiments seem to indicate that in a sealed tube at constant temperature, the decomposition of superpalite in presence of iron oxide does not run to an end, even though the reverse reaction does not take place. This raises the question whether the German really put a mixture of superpalite and phosgene into their shell or whether the extremely variable concentration of phosgene may be due to the catalytic decomposition by the steel shell. There are no experiments as yet to show that effect chloricrin has on this decomposition. In the canister, superpalite is decomposed by the charcoal to phosgene, which is then decomposed by moisture. Superpalite is also decomposed readily by soda-lime.

Bromacetone, CH₂BrCOCH₂ is a water-clear liquid which turns brown or black on standing owing to charring. It boils with decomposition at about 1200, and the vapor pressure of the crude product is 1.0 mm/ at 100 and 3.0 mm/ at 200. It is not quite five times as dense as air. The density of the liquid is given as 1.061 at 0° and 1.063 at 20°. It causes lachrymation at 0.0015 m/ per liter (4.21 p.p.m.) when pure and at 0.001 m/ per liter when containing 20% chloracetone (chloronite) although the chloracetone is a poorer lachrymator than bromacetone. Being fairly volatile and readily decomposed, it is classed as a non-persistent lachrymator. Bromacetone attacks steel and most other metals and must be used in shell lined with lead, glass or enamel.
Bromacetone is only slightly soluble in water; but readily miscible with alcohol and acetone. Traces of water stabilize the product somewhat and addition of chloracetone seems to have the same effect. There is some reason to believe that the instability is due to the presence of some impurity, but it is not known what impurity has this effect. Both the charcoal and the soda-lime in the canisters stop bromacetone.

The French have manufactured a mixture of about 80% bromacetone and 20% chloracetone, which they call Dystemite. In order to prevent the loss of half the bromine as hydrobromic acid, they add a mixture of sodium chlorate and sulphuric acid to oxidize the hydrobromic acid. The reaction is as follows:

\[
5\text{CH}_2\text{COCH}_3 + 4\text{Br} + \text{H}_2\text{SO}_4 + \text{NaClO}_3 = 4\text{CH}_2\text{BrCOCH}_3 + \text{CH}_2\text{OCHCOCH}_2 + \text{NaHSO}_4 + 3\text{H}_2\text{O}
\]

**Bromomethylketone**, is a mixture of CH₂BrCOCH₃ boiling at 155° and of CH₂COCHBrCH₃ boiling at 135°. It is made by brominating methylketone, CH₃COCH₂. It lacks water at 0.000 m/ per liter (1.3 p.p.m.) and is substituted bromomethane sole on account of shortage of acetone. The shell must be lined to prevent corrosion.

**Xylyl bromide**, CH₃CH₂CH₂Br, is a mixture of the ortho, meta, and para compounds, and boils at about 212°. It lacks water at 0.002 m/ per liter (0.25 p.p.m.) and is classed as a persistent lachrymator. Lined shell must be used. Both the charcoal and the soda-lime stop xylyl bromide. The mixture of the three xylenes, which is ordinarily called xylene, is heated, exposed to sunlight, and treated with bromine. Under these conditions the bromine sub-


stitutes in the methyl side-chain and not in the benzene ring. Care must be taken not to carry the bromination too far, as the di-bromide is of no value. Xylyl bromide is sometimes called tolyl bromide because the bromine substitution compound of toluene is called benzyl bromide.

**Mustard Gas**, so-called, \((\text{CH}_2\text{C}0\text{H}_2)\text{Ch}\) melts at 14.2\(^\circ\), when very pure, to a colorless, oily liquid which boils at 217\(^\circ\) at 750 mm. The name was given to it by the British soldiers and is an unfortunate one because the compound has nothing to do with what the chemist calls mustard oil. The vapor is a little less than six times as dense as air. The vapor pressure of a sample melting at 13.8\(^\circ\) was about 40 mm/ at 140\(^\circ\), 30 mm/ at 120\(^\circ\) - 125\(^\circ\), 20 mm/ at 110\(^\circ\), and 12 mm/ at 97\(^\circ\). Some British data are 44 mm/ at 123\(^\circ\) - 132\(^\circ\) and 10 mm/ at 100\(^\circ\). The density of the liquid referred to water at 0\(^\circ\) is 1.2730 at 15\(^\circ\), 1.2556 at 25\(^\circ\), and 1.2564 at 35\(^\circ\). Owing to the low vapor pressure the substance can be used only in shell and is very persistent. The Germans have marked their mustard gas shell with a yellow cross.

The lethal concentration is 0.06 mg/ per liter (7 p.p.m.). The liquid produces burns which appear 4 - 12 hours after exposure and heal very slowly. The vapor also causes burns but to much less extent. It attacks the eyes, causing conjunctivitis and temporary blindness. The percentage of death is rather low with mustard gas, only about 5\%, but this is not due to any low toxicity. It is because the number of casualties due to burns is very large. A comparison with phosgene, which does not burn the skin, is therefore quite improper.
Mustard gas is very slightly soluble in water, less than 0.1%. It is freely soluble in alcohol, ether, chloroform, tetrachloroethane, chlorobenzene, and trioxymethylene. It is miscible in all proportions with ligroin above 190° and with kerosene above 25.6°.

At a pressure of 760 mm 100 volumes of dichloroethyl sulphide dissolve 182 volumes of ethylene at 15° and 100 volumes at 35°. At 60° mustard gas dissolves about 3% of dry hydrochloric acid. At room temperature it dissolves about one percent of sulphur, the solubility becoming about 6% at 100°. Dichloroethyl sulphide is hydrolysed very slowly by cold water and quite rapidly by hot water to thiodiglycol, which is harmless.

\[(CH_2\text{CH})_2S - H_2O = (CH_2\text{CH}CH_2)_2S - 2\text{HCl}\]

Sulphur dichloride reacts rapidly with mustard gas, forming the tetrachlorosulphide. It is this property which makes sulphur dichloride such a valuable reagent in the laboratory for removing mustard gas. The reaction also takes place in carbon tetrachloride solution and more slowly the more dilute the solution. Sodium sulphide solution reacts, forming the ring compound \(5[(CH_2\text{CH})_2]_2S\).

Chloramine-T (p-toluene sodium sulphochloramide) and dichloramine-T (p-toluene sodium sulphochloramide) react vigorously with mustard gas, forming white, crystalline compounds which are not irritating.

Hydrocyanic acid, HCN, is a colourless, mobile liquid boiling at 26.5°. The vapor is slightly less dense than air. The toxic concentration for dogs is about 0.08 mg/liter (30 ppm) but dogs, as has been stated, are exceptionally sensitive to this gas.
There is no cumulative effect and neither the British nor the Germans have ever used hydrocyanic acid. The French have used a mixture called Vincomite; but there seems to be no evidence of its value.

Cyanogen Chloride, CNCl, is a colorless liquid, boiling at +12.6° and solidifying at about -7.4°. The vapor pressure is 444 mm Hg at 0° and 682 mm Hg at +10°. The density of the vapor is only a little more than double that of air. The density of the liquid is 1.23 at 0°. It is a good lacrymator (0.016 mg/liter) and is highly toxic, low concentrations causing cramps in the chest and higher concentrations causing symptoms similar to those of hydrocyanic acid. Like hydrocyanic acid there is no cumulative effect. The toxic concentration for dogs is 0.20 mg/liter (72 p.p.m.); but dogs are the most sensitive to cyanogen chloride of any of the animals.

When dry, cyanogen chloride does not attack iron or silver, but does attack copper. If moist, it attacks all these metals. Cyanogen chloride goes through the German mask very rapidly, which is the reason that the Allies hoped to use it on the Western Front. It is stopped by the American charcoal but not for very long.

Bromocyanogenide, C6H5CHBrCN, is a colorless solid melting at 23°. The crystals soon turn pink owing to a slight decomposition, which does not proceed far however. The commercial product melts at 10° - 22° and the crystals are varying shades of dark brown, often with a marked greenish tinge. The vapor pressure is given at 0.025 mm Hg at 0° and 0.250 mm Hg at 10°. The compound decomposes before the boiling point is reached, even in a high vacuum. The density of the solid is about 1.51 at 25°.
Brombenzylic cyanide is a very effective lachrymator. Most people can detect it at 0.021 parts per million and are lachrymated at 0.04 parts per million (0.00033 mg/liter).

The compound is insoluble in water, moderately soluble in cold alcohol, freely soluble in hot alcohol, and soluble in ether, glacial acetic acid, carbon bisulphide, and benzene. It is hydrolyzed very slowly by boiling water and by cold solutions of sodium hydroxide. A cold alcoholic solution of sodium hydroxide decomposes it rapidly, forming sodium bromide. It is oxidized slowly by potassium permanganate, bleaching powder, chromic acid mixture, etc. Brombenzylic cyanide attacks all metals except lead rapidly and does not corrode lead. It would probably have to be loaded in enamel-lined or glass-lined cell. The magnesium and brass contents are satisfactory in presence of brombenzylic cyanide. It does not react with mustard gas. The charcoal in the American canister stops it well; but the German charcoal appears to be ineffective.

Chloracetone-hydrone, C₇H₅COCH₂Cl, is a colorless crystalline solid, melting at 58°-59° and boiling at 145°-147°. The vapor pressure is about 0.003 mm Hg at 0°, 0.019 mm Hg at 25°, and 0.066 mm Hg at 270°. The density of the solid is 1.354 at 0° and 1.263 at 50°. The pure compound causes lachrymation in the majority of cases at 0.00031 mg/liter (0.046 p.p.m.), so that this substance is about equivalent to brombenzylic cyanide.
Chloracetophenone is insoluble in water but soluble in alcohol, benzene, ether, and chloroform. It is soluble to the extent of two percent in pure silicon tetrachloride but is insoluble in commercial silicon tetrachloride and titanium tetrachloride. It does not react with ammonium nitrate, trinitrotoluene or amatol when heated at 100° for twenty-four hours. It can be used in steel shell.

Ethylideneacetate, CH₃C=CH₂, is a colorless oil of extremely penetrating odor which turns brown in the air with liberation of iodine. It decomposes if boiled in the air. The vapor pressure is 250 mm at 143° and 0.87 mm at 30°. The density of the liquid is 1.8320 at 4°. The toxic concentration for dogs is about 1.6 mg per liter; but the substance is primarily a lachrymator. Nine people out of ten tested were lachrymated at 0.17 parts per million. Due to the scarcity of iodine this is not a very satisfactory substance for war purposes. It was made by the English at a time when the price of bromine was very high. It is made by treating an alcoholic solution of ethyl bromacetate of ethyl chloracetate with powdered potassium iodide.

Acrolein, CH₂CHCHO, is a colorless liquid with a pungent smell, boiling at 52.4°. The vapor is only about 1.5 times as dense as air, which is a distinct disadvantage. It is primarily a lachrymator, one part in a hundred thousand being intolerable. It is about as toxic as phosgene, however, and causes nausea and heart trouble, besides affecting the memory. Since it was not stopped all by the early German mask, great things were expected of this gas. It is not stable under ordinary conditions and polymerizes readily.
While one can stabilize the compound in the laboratory fairly well, it is a very difficult matter to do this on a commercial scale. The French have used it a little in hand grenades for clearing dugouts, because it was not necessary to store the material; but it would be a waste of time, with our present knowledge, to send arsenic across the sea.

**Arsine, AsH₃**, is a colorless and almost odorless gas, boiling at -65°C. It is less than three times as dense as air. The vapor pressures, as determined by Faraday in 1845, are 0.67 atmospheres at 0°C and 13.2 atmospheres at 15°C. The density of the liquid is given by Richter and Eyers as 1.47 at -10°C, 1.44 at 0°C, and 1.37 at 22°C. The toxic concentration for dogs is 0.04 mg/liter (200 p.p.m.)

Liquid arsine does not reduce litmus. The gas decomposes fairly readily in air. In a sealed glass tube, it decomposes into metallic arsenic and hydrogen; but this change does not take place when the tube contains liquid arsine. The decomposition seems therefore to be due to the catalytic action of the walls on arsine vapor.

It has not been used in gas warfare; but if the war had lasted, the allies might have experimented with liquid arsine in cylinders. There have always been rumors that the Japanese were going to use it and consequently protection against it had to be supplied. Neither charcoal nor soda-lime stop it satisfactorily; but it is stopped by soda-lime granules impregnated with sodium permanganate. The potassium salt is less soluble and must be used.
Diphenylchlorcarbim, \((C_6H_5)_2Cl\), is a solid melting at about 44° and boiling at about 330°. The vapor pressure is 25 mm at 233° and 7 mm at 180°. The density of the vapor is about nine times that of air. The density of the crystals is 1.4283 at 15°. It is used in shell in presence of high explosive which scatters it as a very fine powder or smoke in the air. The Germans marked these shells with a blue cross. The lethal concentration is about 0.1 mg per liter but the substance is used chiefly to cause sneezing and thus to force removal of the mask, and it is often called "sneeze gas." It can be detected at one part in a hundred million, produces nasal irritation at one part in fifty million, and is intolerable at one part in a million, attacking the eyes as well as the respiratory tract. It was first used by the Germans in July 1917. It is not soluble in water or ammonia but is readily soluble in alcohol, ether or benzene. It is hydrolyzed by water to \((C_6H_5)_2Cl\) and is oxidized by concentrated nitric acid to diphenyl carbonyl acid. Chlorine destroys the irritating effect of diphenylchlorcarbim, probably due to formation of \((C_6H_5)_2Cl\) or \((C_6H_5)_2Cl\) or \((C_6H_5)_2Cl\), though it is possible that the chlorine either causes the agglomeration of the smoke or causes it to be stopped by the constrictor. Phosgene has no such effect but phosgene containing one percent of chlorine does, and it is stated that a marked increase in the opacity of the cloud is noticed.

The vapor of diphenyl chlorcarbim is stopped by charcoal; but the suspended substance passes through either as solid or liquid and must be stopped by special paper filters or otherwise.
Phenyl dichlorarsine, $\text{C}_6\text{H}_5\text{AsCl}_2$, is a highly refractive liquid boiling at $25^\circ - 255^\circ$. The vapor pressure is 27 mm at $142^\circ$. The substance blisters the skin much more rapidly than mustard gas. A burn up to four days old would be judged 3 - 4 times as extensive as a mustard gas burn of the same age and equally as severe. The burns heal more rapidly than those from mustard gas, so that the usefulness of this liquid is not established. A 60% yield can be obtained by heating tri-phenylarsine and arsenic chloride in suitable proportions in an autoclave at $250^\circ$ for 14 hours.

$$ (\text{C}_6\text{H}_5)_3\text{As} + 2\text{AsCl}_3 = 3(\text{C}_6\text{H}_5)\text{AsCl}_2. $$

**Methyl dichlorarsine**, $\text{CH}_3\text{AsCl}_2$, is a colorless liquid with a powerful burning odor. It boils at $131.5^\circ$ and has a vapor pressure of about 2.2 mm at 0° and 19.3 mm at 35°. The vapor is between five and six times as dense as air. The density of the pure liquid is given by Lichter and J. prep. as 1.073 at 0° and 1.81 at 35°. The toxic concentration for dogs is $0.30 \text{ mg/liter}$ (78 p.p.m.) The substance begins to irritate the nose at a concentration of about 1.3 parts per million. As a liquid, methyl dichlorarsine is less irritant to the skin than phenyl dichlorarsine, but the vapor is much more irritating owing to the higher concentrations attainable. The advantage which this substance may have over mustard gas is that there may be a large number of casualties due to vapor burns. It also goes through clothing more rapidly than mustard gas.
In addition to the toxic gases, several substances have been used as irritant or concealing smoke.

**Phosphorus** is prepared by heating phosphate rock with sand and coke in an electric furnace.

\[
\text{Ca}_3(\text{PO}_4)_2 + 3 \text{SiO}_2 + 50 \text{CaO} + 3 \text{Ca}_3(\text{PO}_4)_2 \text{ plus } 500 \text{pounds } \text{CaPO}_4
\]

Phosphorus comes on the market either as white (yellow) or red phosphorus. Either form burns to phosphorus pentoxide and is then converted to phosphoric acid.

\[
4 \text{P} + 50 \text{O}_2 \rightarrow 2\text{P}_2\text{O}_5 + 6\text{H}_2\text{O} = 4\text{H}_3\text{PO}_4
\]

Since one pound of phosphorus takes up 1.36 pounds of oxygen and 0.9 pounds of water, it is not surprising that phosphorus is the best smoke producer per pound of material. In addition to its use as a smoke producer, it is used in incendiary shells and for coating or core bullets. For incendiary shells the English use a very fine powder or red phosphorus mixed with twice its weight of white phosphorus. Experiments in this country indicate that a better procedure is to use a conglomerate of lumps of red phosphorus embedded in white phosphorus because this mixture burns less rapidly.

**Tin tetrachloride**, SnCl\(_4\), is a liquid made by treating tin with chlorine. It boils at 114°, and fumes in moist air because it hydrolyzes to stannic hydroxide.

\[
\text{SnCl}_4 \text{ plus } 4\text{H}_2\text{O} = \text{Sn (OH)}_4 \text{ plus } 4\text{HCl}
\]

It makes a better and more irritating smoke for shell and hand grenades than either silicon tetrachloride or titanium tetrachloride. It goes through the charcoal and the soda-lime; but is stopped by the layers
of cotton wool in the canister. Since there is practically no tin produced in this country, silicon tetrachloride and titanium tetrachloride have been developed as substitutes.

**Silicon tetrachloride**, $\text{SiCl}_4$, is made from silicon or from impure silicon carbide by heating with chlorine in an electric furnace.

$$\text{Si} + 2 \text{Cl}_2 \rightarrow \text{SiCl}_4.$$  

It is a colorless liquid, boiling about $36^\circ$ and fuming in moist air owing to hydrolysis:

$$\text{SiCl}_4 + 2\text{H}_2\text{O} \rightarrow \text{Si(OH)}_4 + 4\text{HCl}.$$

It is not much good in shell but is better on moist cool days than on warm dry ones. A carbonic cylinder and a silicon tetrachloride cylinder with liquid carbon dioxide as propellant give a first-class smoke when the jet from the two cylinders impinges.

$$\text{SiCl}_4 + 4\text{H}_2\text{O} + 3\text{H}_2 = \text{Si} + 4\text{HCl} + 6\text{H}_2\text{O}.$$  

This is used in the Navy smoke signal to protect vessels from submarine attack and in portable cylinders to mask the advance of infantry. By adding a lubricator to silicon tetrachloride we get a mixture which works well in hand grenades foropping in trenches.

**Titanium tetrachloride**, $\text{TCl}_4$, is made from rutile, $\text{TiO}_2$, by mixing with carbon and heating in an electric furnace. Carbonitride is formed which is said to have the composition $\text{TiCl}_4\text{C}_4$; but the actual composition may vary from this to the carbide $\text{TiC}_2$. When these products are heated with chlorine, titanium tetrachloride is formed. This is a colorless, strongly refracting liquid which boils at about $136^\circ$, is stable in dry air, but forms in moist air. It is
said that the addition of water to form \(\text{TiCl}_4\cdot\text{H}_2\text{O}\) gives a good smoke and that the hydrolysis to \(\text{Ti}([\text{OH}]_4\) gives a poorer smoke. Tin tetrachloride is poorer than tin tetrachloride and silicon tetrachloride in hand grenades. In the smoke funnel it is better than tin but not so good as silicon. Since it costs more than silicon tetrachloride, it would really be used only in case of a shortage.
In February of 1917, war between the United States and Germany seemed inevitable, so the Director of the Bureau of Mines, Mr. Van H. Manning, called the attention of the War Department to the already existing technical organization in the Bureau for the study of poisonous gases in mines, and offered the facilities of the Bureau to the War Department for work on poisonous gases in warfare. A meeting was arranged between representatives of the Bureau and the War College, the latter organization being represented by Brigadier General Kuhn and Major L. F. Williamson. At this conference the War Department accepted enthusiastically the offer of the Bureau of Mines and agreed to support the work in every way possible.

Mr. George A. Surrell was summoned to Washington in April by Mr. Manning, and placed in charge of the work which was carried on with the funds of the Bureau of Mines until July 1, 1917, after which funds were supplied by the War Department and Navy Department.

At the first meeting called by Mr. Manning for the technical solution of some of the problems, there were present:

Mr. Van H. Manning, Director, Bureau of Mines
Mr. Geo. S. Dice, Chief Mining Engineer, Bureau of Mines
Mr. J. W. Paul, Mining Engineer, Bureau of Mines
Mr. A. C. Fieldner, Chief Chemist, Pittsburgh Station, Bureau of Mines
Dr. Yandell Henderson, Consulting Physiologist, Bureau of Mines
Prof. N. E. Gibbs, Mechanical Engineer, Bureau of Mines
Major L. F. Williamson, War College
Mr. G. A. Surrell

In April the following committee on gases used in warfare was formed by the National Research Council for the purpose of cooperating with the Bureau of Mines:
Two months later the committee was re-formed, owing to the
removal of certain army medical officers, as follows:

Mr. Van M. Muck, Chairman, Director, U.S. Bureau of Mines.

Maj. L. P. Williamson, Medical Department, U.S.

Lt. T. S. Wilkinson, Bureau of Ordnance, U.S.

Medical Director, J.S. Gatewood, Medical Department, U.S.

Dr. M. T. Seger, Professor of Organic Chemistry, Columbia University.


Mr. Van M. Muck, Chairman, Director, U.S. Bureau of Mines.

Dr. Geo. E. Hale, ex-officio, Science and Research Division National Research Council.


Maj. A. J. Atkinson, Corp. Medical Corps, U.S.

Dr. M. T. Seger, National Research Council.

Major Bradley Dewey, Chief, Medical Service, Surgeon School's Office.

Lieutenant Commander W. M. Wick, U.S.N.

Lieutenant Joseph M. Philip, F.A. Surgeon, Bureau of Medicine and Surgery, U.S.N.

Capt. W. J., Ad. R.C., Bureau of Ordnance, U.S.

Lieutenant T. S. Wilkinson, Bureau of Ordnance, U.S.

Dr. M. T. Seger, National Research Council.

Dr. H. C. Muck, U.S.N.

Dr. C. B. H. Ahlburg, Chief, Bureau of Chemistry, U.S.

Lieutenant Commander W. M. Wick, U.S.N.

Dr. C. B. H. Ahlburg, Chief, Bureau of Chemistry, U.S.

Capt. W. J., Ad. R.C., Bureau of Ordnance, U.S.

After the literature on gas warfare received in this country from
France and England had been digested, definite lines of research were begun.

The work was started at the Bureau of Mines Laboratory at Pittsburgh. One of the first men added to Mr. Sull's staff was Bradley Dewey, research chemist for the American

Standard Tin Plate Company at Pittsburgh. It was soon deemed advisable to...
Mr. Bovee was commissioned a major in the ordnance corps, and was placed in charge of the 3rd defense service, his duty being to manufacture and all appurtenances, while Mr. Burrell remained in charge of the research work. The research staff grew so rapidly that sufficient laboratories were not available either in Washington or in any one place. Before long, research work was being carried out at the bureau of mines, Pittsburgh, Pa.; the National Carbon Company, Cleveland, Ohio; the Forest products laboratory, Madison, Wisconsin; the university of Illinois; the research laboratory of the American sheet & tin plate company, Pittsburgh, Pa.; the bureau of chemistry laboratory, Washington, D.C.; the Yale medical school laboratory, New Haven, Conn.; the Massachusetts institute of technology, Cambridge, Mass.; the Mellon Institute, Pittsburgh, Pa.; and elsewhere. In the summer of 1917, the university of Washington generously offered its buildings and laboratories for free. A good deal of reconstruction was necessary in order to complete the buildings and grounds. These laboratories became available in the autumn. Later the Catholic University of America placed part of its medical laboratory at the disposal of the bureau of mines for research work.

The procurement of toxic gases for the filling of shells was assigned to the trench warfare section of the ordnance department, Captain (afterwards Lt. Colonel) E.J.J. Hagedale in charge. In June 1917, General Grozier, then Chief of the ordnance department, approved the general proposition, submitted by Captain Hagedale, of building a suitable plant for filling shells with toxic gas.
While the regular artillery handle the shell, special troops are required for cylinder attacks, Stokes mortars, Alvin's cranes, etc. General Pershing accordingly advised by cable the organization and training of such troops and recommended that they be placed, as in the English army, under the jurisdiction of the Engineer Corps. On August 15, 1917, the General Staff authorized one regiment of gas and flame troops, which was designated the 30th Engineers. Major (afterwards Colonel) A. J. Atkinson was placed in charge. At first it was intended that this regiment should consist largely of chemists; but it was pointed out by the British officers in this country that this was not necessary; so consequently many of the chemists were transferred so that their special training might be utilized more effectively.

In September 1917 a re defense field training section had been started in the Sanitary Corps under Captain (afterwards Major) J. H. Atkinson, Jr. It was soon found that neither the Training Section nor the divisional Sanitary Officers should be under the Medical Department and in January 1918, the organization was transferred to the Engineer Corps.

In the spring of 1917 Professor E. A. Vulett of Princeton University was sent abroad by the Bureau of Mines and the National Research Council to study the question of war chemistry. It was soon seen that visible steps must be taken to develop the science and Professor Vulett returned to this country with a letter, dated August 14, 1917, in which General Ferdinand said: "Dr. Vulett has been of the greatest assistance to this office in the study of the organization of a War Service and it is desired to utilize in our organization his very expert technical knowledge. It is recommended that all chemical or War Service of the United States be united under one head. He shall be an officer of the United States Army with
suitable rank, staff, etc. In order to obtain questions the medical service of the United States should be guided by the experience of the service in France, as this service is being developed here and improvements occurring should be adopted here at early date. An auxiliary service for the medical expeditionary force should be organized and ready to frame at once.

It is desired that the experience and knowledge of W. Meikle be utilized in creating this organization and that he be sent as Director or it with appropriate rank under your instruction. There is enclosed herewith a copy of the medical organization which has been adopted for the Medical Expeditionary Force, under W. Meikle in a position to explain in detail, the reasons for its adoption.

On September 3rd, 1917, an order was issued constituting a department of the Medical Expeditionary Force in France to be known as the Medical Service under Lieutenant Colonel (afterwards Brigadier-General) M. M. Kriie. This is the organization referred to in General Pershing's letter and the entire organization is in the full near chart, which also calls for some kind of administrative centre in the United States. On September 21, 1917, the Surgeon General, therefore, submitted a memorandum for the Chief of Staff in which he urged the militarization of the Russian Initory under the Bureau of Mines at the Massachusetts University. The belief was also expressed that a distinct gas service should be established.
operating under one head, the Director of Gas Service, who should report directly to the Chief of Staff and who should have at his assistants, an Assistant Director of Gas Service in the Ordnance Department in immediate charge of the production of gas shells, projectiles and other offensive apparatus, and an Assistant Director of Gas Service in charge of the Chemical Service Section. The Bureau of Mines objected to this plan on the ground that greater flexibility for research could be obtained under civilian management than under military control. The recommendation was disapproved by the Secretary of War; but on October 16, 1917, the Secretary of War authorized the establishment of a Gas Service in which an officer of Engineers not above the rank of Colonel, and recommended by the Chief of Engineers, should be appointed Director of Gas Service and ordered to report to the Chief of Staff for duty. As assistants he was to have one Ordnance Officer not above the rank of Lieutenant Colonel, recommended by the Chief of Ordnance, one Medical Officer not above the rank of Lieutenant Colonel, recommended by the Surgeon General, and one officer of the Chemical Service Section of the National army, recommended by the Director of Gas Service. There was authorized the raising and maintaining, by voluntary enlistment or draft of a Chemical Service Section of the National army with the following personnel:

Commissioned:
2 lieutenant colonels
4 majors
6 captains
10 first lieutenants
25 second lieutenants
47 total commissioned

Enlisted:
10 sergeants, 1st class
15 sergeants
20 corporals
50 privates, 1st class, and privates.
56 total enlisted.
On November 1st, 1917, there was created the first administrative unit of the chemical service in the United States, which holds also the distinction of being the first recognition of chemistry as a separate branch of the military service in any country or any war. Colonel Claude L. Potter of the Engineers was appointed Director of the Service and Prof. W. R. Walker of the Massachusetts Institute of Technology, who had been for a short time in charge of the Helium Production of the Bureau of Mines, was commissioned Lieutenant Colonel and made Assistant Director of the Service and Chief of the Chemical Service Section. Major S. J. M. Hald, Chemical Adviser to the British Mission and Captain H. C. Dingley acted as liaison officers for offense and defense problems.

On September 26th, 1917, General Pershing had said:

"Send at once Chemical Laboratory complete with equipment and personnel, including physiological and pathological sections, for extensive investigation of gases and powders. Arrangements to make for physiological chief from medical personnel now in Europe. The laboratory is to be auxiliary to the one in the United States and is for local emergency investigations to meet the constant changes in gases and powders used by the enemy and by ourselves."

Mr. J. E. Bacon of the Mellon Institute of Pittsburgh was commissioned Lieutenant Colonel to organize and take charge of the laboratory in France and Prof. C. H. Long of the University of California and Dr. J. A. H. H. of the Mellon Institute were commissioned Majors on his staff. Men were secured, a complete laboratory equipment was purchased, and Lieutenant Colonel Bacon sailed for France about January 1st, 1918. Much of the equipment went astray in France and did not become available until May, 1918.

All the men in the French laboratory were in uniform and belonged to the same organization, the Chemical Service Section. At the American University Experiment Station of the Bureau of Mines most of the research
men were civilians and those who were in uniform were distributed among
the Ordnance Department, the Sanitary Corps, the Corps of Engineers, the
Signal Corps, the Aviation Section, and the Chemical Service Section. It
seemed wiser to transfer all the chemists in uniform to the Chemical
Service Section and to make provision that the civilian chemists might be
taken into the Chemical Service Section if they so desired. Accordingly
a request was made for an enlargement of the Chemical Service Section so
that the personnel should be as follows:

<table>
<thead>
<tr>
<th>Commissioned</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Lieutenant Colonels</td>
<td>2</td>
</tr>
<tr>
<td>Major</td>
<td>40</td>
</tr>
<tr>
<td>Captains</td>
<td>30</td>
</tr>
<tr>
<td>First Lieutenants</td>
<td>50</td>
</tr>
<tr>
<td>Second Lieutenants</td>
<td>125</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td>227</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Enlisted</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Sergeants, 1st class</td>
<td>50</td>
</tr>
<tr>
<td>Sergeants</td>
<td>75</td>
</tr>
<tr>
<td>Corporals</td>
<td>100</td>
</tr>
<tr>
<td>Private, 1st class</td>
<td>100</td>
</tr>
<tr>
<td><strong>Total Enlisted</strong></td>
<td>525</td>
</tr>
</tbody>
</table>

This personnel was authorized on February 15th, 1919.

In the meanwhile Colonel Potter had retired from the position of
Director of Can Service and had been replaced by Lieutenant Commander M. H.
Ward, who resigned his commission in the Navy and was appointed civilian
Director of Can Service. Owing to ill health Mr. Ward did no active work
...all the duties of the office fell to Lt. Colonel Walker until March 1898 when he was transferred to the Ordnance Department, made a Colonel, and given charge of the shell filling plant at Pikeville, Maryland. To succeed him Prof. J. T. Fogart, of the University of Colorado was commissioned Lt. Colonel and appointed assistant director of the Service and Chief of the Chemical Service Section.

The personnel authorization of February 15, 1913, was not adequate for long and in April Lieutenant Colonel Fogart requested an enlargement of the personnel to the following amount, which was authorized on May 6th:

**Commissioned**

<table>
<thead>
<tr>
<th>Rank</th>
<th>Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lieutenants Colonel</td>
<td>2</td>
</tr>
<tr>
<td>Majors</td>
<td>20</td>
</tr>
<tr>
<td>Captains</td>
<td>35</td>
</tr>
<tr>
<td>First Lieutenants</td>
<td>111</td>
</tr>
<tr>
<td>Second Lieutenants</td>
<td>235</td>
</tr>
<tr>
<td>Total Commissioned</td>
<td>395</td>
</tr>
</tbody>
</table>

**Enlisted**

<table>
<thead>
<tr>
<th>Rank</th>
<th>Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sergeants, 1st class</td>
<td>80</td>
</tr>
<tr>
<td>Sergeants</td>
<td>120</td>
</tr>
<tr>
<td>Corporals</td>
<td>170</td>
</tr>
<tr>
<td>Privates, first class</td>
<td>250</td>
</tr>
<tr>
<td>Privates</td>
<td>300</td>
</tr>
<tr>
<td>Total enlisted</td>
<td>920</td>
</tr>
</tbody>
</table>

Lieutenant Colonel Fogart also recommended on April 17, 1913, that the Chemical Corps, Chemical Service Section, be made a separate organization or corps like the Ordnance or Medical Department, but this was disapproved.

Cobalt blue and golden yellow, the colors of the American Chemical Society, were adopted about this time as the colors for the hat cords of the enlisted men, instead of the colors of the engineers which had been worn hitherto.

With the increasing demands of gas warfare, the danger of a possible shortage of graduate chemists seemed threatened. Therefore, on May 8
2018, at the urgent request of the Chemical Service Section, the adjutant general ordered that all enlisted men who were graduate chemists should be assigned to duty where their special knowledge and training could be fully utilized and that they should not be sent overseas unless they were to be employed on chemical duties. It was also ordered that, whenever chemists were needed by one of the Bureaus or Staff Corps, requests should be made of the Chief of the Chemical Service Section for recommendation of men having the qualifications necessary for the particular class of work in question. If men having chemical qualifications were wanted only for a short period of time they would be attached temporarily to the Bureau or Staff Corps, but when the duty was of a permanent nature instructions would be issued covering their transfer. Whenever the chemists thus attached or transferred were no longer needed for purely chemical duties a report was to be made to the Chief of the Chemical Service Section in order that they might be assigned to chemical duties at other places.

In the beginning of May, over a year after the United States entered the war, practically every branch of the United States Army had some connection with Gas Warfare. The Medical Corps directed the Gas Defense Production. Gas Defense Production was in the hands of the Ordnance Department. Alarm devices, etc., were made by the Signal Corps. The Gas and Flame troops formed the 30th Regiment of engineers and the Field Training Section of the Gas Defense Training had been transferred from the Sanitary Corps to the Engineer Corps. Research work was being carried on by the Bureau of Mines, a branch of the civilian department of the Interior. Besides this a Chemical Service Section had been formed primarily to deal with overseas work. While the Director of this Service was expected to coordinate all the activities of the various departments dealing with
chemical warfare, he had no authority to control policy, research, or production.

In order to improve these conditions, Major General W. L. Gist, a distinguished Engineer Officer who had built the Gatun locks of the Panama Canal and who had commanded the 2nd Division in France, was appointed Director of Gas Service on May 11, 1918, and was instructed to formulate a plan for better coordination of the several departments dealing with gas warfare. On May 20, 1918, General Gist sent to the Chief of Staff a request for the following transfers:

1st: The Gas Defense Service of the Medical Department.

2nd: The Edgewood Arsenal and all operations there and elsewhere that concern the manufacture of gas warfare material, and the loading of shell with gas material.

3rd: All proving ground operations that are connected with gas warfare.

4th: The manufacture of all signal and alarm devices for gas warfare.

On July 13, 1918, this transfer was authorized and General Gist took over from the Ordnance Department the whole staff of the Edgewood Arsenal under Col. Miller, and from the Sanitary Corps the Gas Defense Service under Lieut. Col. Dewey. Later the Gas Defense Training Section and the Gas and Flame Troops were transferred from the Corps of Engineers to the Chemical Warfare Service. On July 21, 1918, the Medical Section of the Gas Defense Service was transferred from the Sanitary Corps to the Chemical Warfare Service, remaining in charge of Colonel W. J. Lyster who was given a temporary detail under General Gist. This division was charged with the necessary research work in the medical field pertaining to protective devices and to Warwick gases and other toxic substances; including prophylactic and curative measures in connection with the experimentation, producing, and handling of these; also with the inspection of the organization for protection of employees against Warwick and other toxic substances at plants handling these substances, and the making of appropriate reports and recommendations thereon, with a view to developing and standardizing an effective system.
On June 25th President Wilson ordered that the American University Experiment Station be transferred from the Bureau of Mines to the War Department and placed under the Director of Gas Service. In connection with this transfer Secretary Baker wrote President Wilson as follows:
June 25th, 1918.

My dear Mr. President:

In connection with the proposed transfer of the chemical section at American University from the Bureau of Mines to the newly constituted consolidated Gas Service of the War Department, which you are considering, I am specially concerned to have you know how much the War Department appreciates the splendid services which have been rendered to the country and to the Army by the Department of the Interior, and especially by the Bureau of Mines under the direction of Dr. Manning. In the early days of preparation and organization, Dr. Manning's contact with scientific men throughout the country was indispensably valuable. He was able to summon from the universities and the technical laboratories of the country men of the highest quality and to inspire them with enthusiastic zeal in attacking new and difficult problems which had to be solved with the utmost speed. I do not see how the work could have been better done than he did it, and the present suggestion that the section now pass under the direction and control of the War Department grows out of the fact that the whole subject of gas warfare has assumed a fresh pressure and intensity, and the director of it must have the widest control so as to be able to use the resources at his command in the most effective way possible. The proposal does not involve the disruption of the fine group of scientific men Dr. Manning has brought together, but merely their transfer to General Sibert's direction.

Respectfully yours,

Newton D. Baker.

The President.

Upon receipt of this letter President Wilson wrote as follows to Director Manning in recognition of his services and that of the bureau under him:

THE WHITE HOUSE

Washington

26 June, 1918.

My dear Dr. Manning:

I have had before me for some days the question presented by the Secretary of War involving the transfer of the chemical section established by you at the American University from the Bureau of Mines to the newly organized Division of Gas Warfare in which the War Department is now concentrating all the various facilities for offensive and defensive gas operation. I am satisfied that a more efficient organization can be effected by having these various activities under one direction and control, and my hesitation about acting in the matter has grown only out of a reluctance to take away from the Bureau of Mines a piece of work which thus far it has so effectively performed. The Secretary of War has assured me of his own recognition of the splendid work you have been able to do, and I am taking the liberty of enclosing a letter which I have received from him, in order that you may see how fully the War Department recognizes the value of the service.
I am to-day signing the order directing the transfer. I want, however, to express to you my own appreciation of the fine and helpful piece of work which you have done, and to say that this sort of team work by the bureaus outside of the direct war-making agency is one of the cheering and gratifying evidences of the way our official forces are inspired by the presence of a great national task.

Cordially yours,

WOODROW WILSON.

Dr. Van. H. Manning,
Chief, Bureau of Mines,
Department of the Interior.

In a memorandum to the Chief of Staff on July 5th, 1916, General Sibert proposed to divide the activities of the Chemical Warfare Service into an office organization consisting of a Director, an Assistant Director, a Technical Advisory Staff, a Liaison Service and an Administrative Bureau with the following divisions: Overseas; Research; Development; Proving; Gas Defense Production; Gas Offense Production; and Gas and Flame Troops. He requested the following personnel:

1 major general
2 brigadier generals
14 colonels
51 lieutenant colonels
180 majors
464 captains
857 1st lieutenants
732 2nd lieutenants

Total Commissioned

91 master engineers, senior grade
152 master engineers, junior grade
13 regimental sergeants major
24 regimental supply sergeants
33 battalion sergeants major
35 battalion supply sergeants
206 1st sergeants
363 sergeants, 1st class
86 mess sergeants
186 supply sergeants
947 corporals
1912 corporals
As a consequence of the new personnel Colonel Fries, who was in charge of the overseas Gas Service was made Brigadier General and Lt. Colonel H. F. Bacon of the French Laboratory was promoted to Colonel. Colonel Walker was placed in charge of the new Gas Defense Production Division; Lieut. Colonel Dewey was promoted to Colonel and placed in charge of the Gas Defense Production Division; Mr. Burrell was commissioned Colonel and placed in charge of the Research Division; Mr. J. M. Dorsey was commissioned a Colonel and placed in charge of the Development Division; Captain E. S. Bacon was made a Major (later Lt. Colonel) and placed in charge of the Proving Division and Lieut. Colonel Bogert was promoted to Colonel. In August 1918 Colonel H. S. Newcomer was transferred from the Engineer Corps, promoted to Brigadier General, and made assistant director.

On August 1st, 1918, after Colonel J. H. Lewin came back from France, he was put in charge of the Training Division.

On General Hibert's staff were Colonel W. J. Lyster, F.C. representing the Medical Department and in charge of the Medical Division; while Lt. Col. C. J. Damm represented the Ordnance Department, and Lt. Col. J. J. H. said the British Military Ordinance. During Colonel Mand's absence in England, Major J. H. Bratman acted for him.

Effective coordination of the different activities was obtained by weekly meetings of the Executive Committee, consisting of Major General Hibert, Brigadier General Newcomer, Colonel Lyster, Lt. Col. Tharpen, Lt.
The Relations Section, under Col. Bogert, saw to it that men who were essential to chemical industries vital to the war were kept at their posts because they would be of far greater value to the country there than at the front, and a transfer would have curtailed and delayed the output of munitions. This section also arranged that a certain number of enlisted chemists were sent back to the universities on indefinite furlough to assist in teaching. This was very important because the chemical staffs of the colleges had been badly crippled by the demand for chemists. If the chemical departments had had to give up important parts of their work, there would have been no continuous supply of chemists and the Government would have been handicapped badly because the war demands for chemists were increasing rapidly. After the armistice was signed, this section acted for a while as an employment bureau securing positions for the lists of those who had been demobilized and discharged.

To meet the needs of the rapidly expanding military establishment abroad, the Adjutant General directed, on September 16, 1918, that the additional Car and Plane regiments be organized. Several well-known field officers were given commissions in the field to bring the operations of the general public which did not understand that these officers were selected for their ability as leaders and not for actual or future ability as tacticians.

On October 20, 1913, a final increase in personnel was authorized as follows:

"(a) Under authority conferred by the act of Congress "authorizing the President to increase temporarily the military establishment of the United States," approved May 18, 1917, and the act of Congress approved July 9, 1918 (Pub. 35), the Adjutant General is authorized for the period of the existing emergency to enlist, enroll, recruit, and maintain a regiment of the line service, exclusive of non-combatant regiments, to have been or may be hereafter authorized, and of such personnel as may be authorized by Tables of Organization for the Headquarters of Divisions, Army Corps, and Field Service, shall consist of:

1 Major General 168 Master Engineers, E.G.
2 Brigadier Generals 323 Master Engineers, J.C.
12 Colonels 34 Regimental Sergeants Major
55 Lieut. Colonels 35 Regimental Supply Sergeants
275 Majors 41 Battalion Sergeants Major
646 Captains 42 Battalion Supply Sergeants
941 First Lieutenants 156 1st Sergeants
1139 Second Lieutenants 1242 Sergeants, 1st Class
3051 Total Commissioned 125 Base Sergeants
166 Supply Sergeants 2 Stable Sergeants
2074 Sergeants
4064 Corporals
152 Ensigns
576 Cooks
386 Mechanics
1450 Wagoners
1 Horseshoer
1 Saddler
6284 Privates, 1st Class
11841 Privates
29090 Total Enlisted

"And the President further directs that for each Division that has been or may be authorized, there shall be Chemical Warfare Service personnel for the Division headquarters, as is prescribed by the Tables of Organization. That for each 4 combat divisions or major fraction thereof there shall be Chemical Warfare personnel, as prescribed by Tables of Organization, for any Corps headquarters; and that for each 20 combat divisions or major fraction thereof, there shall be Chemical Warfare Service personnel, as prescribed by Tables of Organization, for an Army Headquarters."

Owing to the signing of the Armistice on November 11, 1918, the actual personnel was not reached.
<table>
<thead>
<tr>
<th>Position</th>
<th>Name</th>
<th>Initials</th>
<th>Rank</th>
</tr>
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<tbody>
<tr>
<td>Medical Director</td>
<td>John</td>
<td>J.O.</td>
<td>Major</td>
</tr>
<tr>
<td>Assistant Director</td>
<td>Jane</td>
<td>J.A.</td>
<td>Captain</td>
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<tr>
<td>Teaching Division</td>
<td>Robert</td>
<td>R.B.</td>
<td>Sergeant</td>
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<tr>
<td>Research Division</td>
<td>Michael</td>
<td>M.C.</td>
<td>Staff</td>
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<td>Staff</td>
<td>Staff</td>
<td>S.S.</td>
<td>Sergeant</td>
</tr>
<tr>
<td>Board of Directors</td>
<td>Bob</td>
<td>B.B.</td>
<td>Captain</td>
</tr>
<tr>
<td>Chemical Warfare</td>
<td>Tom</td>
<td>T.C.</td>
<td>Captain</td>
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<tr>
<td>Staff</td>
<td>Staff</td>
<td>S.S.</td>
<td>Sergeant</td>
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RESEARCH DIVISION

In the preceding chapter it has been shown that the first war work was started in the Bureau of Mines under Mr. Van N. Manning with Mr. C. A. Burrell in immediate charge, and that various changes took place whereby the organization under Mr. Burrell finally became the Research Division of the Geological Survey Service, which had been transferred from the Department of the Interior to the War Department. The first five months the research work was done in affiliated laboratories scattered over the country; but in September 1917 the building of the American University in Washington became available and that laboratory also the research Division became known as the American University Experiment Station of the Bureau of Mines. The organization at that time was as follows:

Mr. C. A. Burrell, Director of the Bureau of Mines
Mr. C. A. Burrell, Assistant to the Director, in charge of the investigations
Mr. W. F. W. Lovett, in charge of scientific research
Mr. A. R. Stoll, in charge of chemical research
Mr. W. H. Jordan, in charge of geological research
Mr. A. S. H. Trimble, in charge of mining research
Mr. W. H. Jordan, in charge of administrative matters

Name

John Hopkins University
Princeton University
National Carbon Co., Cleveland
B. U. Park, Cleveland
Carnegie University
Yale University
Cornell University
Carnegie Institute of Technology

Address

E. A. Smith
W. F. W. Lovett
C. A. Burrell
W. H. Jordan
J. R. Smith
J. A. Norton
J. A. Norton
J. C. M. Fox
R. W. Withrow

Institution

Cornell University
As time went on there were many changes. Dr. John Johnston was placed in charge of Ordnance Programs, a position corresponding to that of J. H. Lewis in defense matters. When Johnston resigned to go to the National Research Council, L. P. Kosler was called from Harvard to fill the vacancy. The work on Chemical Research became more than one man could handle and was divided into two parts, Ordnance Chemical Research under N. F. Tollefson and Defense Chemical Research under M. I. Linder and defense chemical research under L. L. Doherty. A major position in charge of Pathological Research, was created in the new department at New Haven. The Pathological Research was later split into two parts, one under Linder and the other under Dr. M. M. Tollefson. The scientific work continued on pathological problems, with the transfer from the Bureau of Mines to the Department of Health. Linder, Tollefson, Underhill, Linder, Underhill, and Underhill were transferred to the Medical Division. The Technical Division was split into two parts under M. M. Tollefson and W. L. Doherty. Later, it was consolidated under W. L. Doherty, then split again (afterward, L. J. Linder) into two parts. In the new year, the work was coordinated with that of H. B. Brey under the Division of Research, and the Chemical Division, under L. L. Doherty, placed in charge of the Development Division.
Briggs at Yale University under T. B. Johnson, at Clark University under C. A. Kraus, at Worcester Polytechnic Institute under J. E. Jennings, and at Harvard University under Reid Hunt, while the branch laboratory started at the University of Wisconsin had been transferred to the Medical Division.

Dr. Hallikin left the Institute of Technology to become a Major in the Chemical Warfare Service with duties at Headquarters.

The Catholic University of America placed a large portion of its new chemical laboratory at the disposal of the Research Division and Columbia University provided facilities for a number of experiments on a semi-large scale. Even after the transfer to the American University, the Pittsburgh laboratories of the Bureau of Mines were used for investigations.

In order to keep in touch with the country at large Secretary Lane appointed an advisory board consisting of E. H. Nichols/Chairman, E. C. Franklin, C. H. Hopkins, C. M. Parsons, Ira Remsen, T. M. Richards, and F. P. Venable. This board was continued by General Libbert but was never called together by him.

At the time the armistice was signed, the organization of the Research Division was as follows:

- Colonel G. A. Burrell, Chief of Research Division
- Dr. E. K. Lewis, In charge of Defense Problems
- Dr. E. F. Kohler, In charge of Offense Problems
- Dr. Reid Hunt, Advisor on Pharmacological Problems
- Lt. Col. J. D. Bancroft, In charge of Medical Work and Catalytic Research
- Dr. E. L. Jones, In charge of Offense Chemical Research
- Major L. C. Fieldner, In charge of Gas Mask Research
- Major C. L. Lichter, In charge of Pyrotechnic Research
- Dr. A. S. Loevenhart, In charge of Pharmacological Research
Major R. C. Tolman, In charge of Dispersoid Research
Major J. S. Rowland, In charge of Small-Scale Manufacturing
Major E. B. Fogler, In charge of Mechanical Research & Development
Captain G. A. Rankin, In charge of Explosives Research
Major Richmond Levering, In charge of Administrative Section.

The maximum staff on was about 1900, consisting of 1200 technical men and 700 service assistants. The latter included stenographers, clerks, accountants, purchasing agents, machinists, instrument makers, etc.
The chief functions of the Research Division are:

1. To make and test compounds which may be of value in gas warfare, determining the properties of these substances and the conditions under which they may be effective in warfare.

2. To develop satisfactory methods of making such compounds as seem promising.

3. To develop the best methods of utilizing these compounds.

4. To develop materials which shall absorb or destroy the gas agents, studying their properties and determining the conditions under which they may be effective.

5. To develop satisfactory methods of making such agents, etc., as may seem promising.

6. To do the research work in connection with masks, canisters, protective clothing, etc.

7. To develop incendiaries, smoke, signal, etc. and the best methods of utilizing them.

8. To co-operate with the manufacturing divisions in regard to difficulties arising during manufacture.

9. To co-operate with other branches of the government, civil and military, in regard to war problems.

10. To collect and make available to the Director of Chemical Warfare Service all information in regard to the chemistry of gas warfare.
In one sense the research division is the most important of all because without the research work nothing could be done. In another sense it is the least important of all, because it never delivers anything tangible. Its results are turned over to the production divisions which actually make the guns or the mine. A somewhat similar distinction occurs between the production divisions of the army and the fighting divisions. The soldier cannot fight unless he is fed, clothed, and armed to some extent; but it is the soldier at the front who gets the actual fighting and drives the enemy back.

As has been stated the term "war gas" is a flexible one and includes solids, liquids, and gases. It is only necessary that the substance should have sufficiently marked characteristics to make it useful in weather. It may be poisonous, like phosgene; it may produce heat, like bromoethyl cyanide; it may cause nausea, like chloropicrin; it may cause sneezing, like diphenylchlorarsine; it may cause burns, like mustard gas; it may have a foul smell, though otherwise harmless, like isobutyl mercaptan; or it may produce a smoke, like phosphorus. Of course several of these properties may be combined in a single substance. To be of real value a compound must have some one of these properties to a marked extent. When gas are employed for thirty minutes the concentration which kills is 0.0 mg per liter for chloropicrin, 0.35 mg per liter for phosgene, 0.05 mg per liter for mustard gas. In other words a gas which requires a concentration of 1.0 mg per liter to kill a dog on a thirty minute exposure is scarcely worth considering as a toxic gas unless it has some other striking characteristic such as .
the mask readily. Most men are lachrymated by bromoacetone at 0.0013 mg per liter, by allyl bromide at 0.002 mg per liter, and by bromoethyl cyanide at 0.00033 mg per liter. A substance which requires a concentration of 0.003 mg per liter to cause lachrymation would probably not receive further consideration.

Another determining factor in the use of any gas is the availability of raw materials. It would be useless to consider iodine compounds as toxic gases because it would be impossible to get enough iodine to make a supply of material which would count. The required use of ethyl isocyanate for lachrymator at a time when the price of bromine was very high; but lachrymators not at much lower concentrations than toxic gases and are also not used to often. Curare is a deadly poison but the available annual supply in this country probably does not exceed one gram and the unit for toxic gases is a thousand of tons.

A good method of manufacture should be at hand. If the substance is good enough, it will be made by any method, no matter what. The same when the Germans made mustard gas by the cyanide method instead; but this is not true in most cases. The Allies probably have used trichloroisocyanuric acid, superphosphate, if a good method of manufacture had been developed; but the waste of chlorine was so great it to overwhelm the distinct merits of the compound.

A substance must be stable or fairly stable. If it polymerizes too rapidly, hydrolyzes too rapidly, is too inflammable, or not to pieces on detonation, it cannot be used effectively. Under ordinary circumstances acrilin polymerizes too quickly to be made in the United States because the shell are likely to be loaded in this country 3 - 6 months before they are
fired. Our limits is to stability against polymerization must therefore be more rigid than those of the British and French who use their material, if necessary, within a month or two after loading. As a matter of fact the French have used aeroloid in small quantites; while it is possible to stabilize aeroloid when made in small quantities, it is uncertain whether this could be done satisfactorily in large-scale manufacture.

Aromatic thiocyanate hydrolyses too rapidly to have any value and hydrogen sulphide is so inflammable that it is dangerous to use in cloud attacks, as the English found to their cost. Chlorpierine decomposes to some extent when fired in small but not enough to be serious. On the other hand tetrachloro-dinitro ethane, an otherwise promising smoke, is destroyed pretty completely when fired in a three inch shell.

It may be worth while to outline the general procedure when a new toxic substance is to be considered by the research division. The substance in question may have been used by the Germans or the Allies; it may have been suggested from outside; or the unit may have thought of it from a search of the literature, from analogy, or from pure inspiration.

The Office of Research Section takes the substance. If it is easily it is sent to the Physicochemical Section, where methods of dispersion, it is worked out. When this has been done, or at once if the compound is a liquid or vapor, it is sent to the Toxicological Section to be tested for toxicity, irritant power, etc. If the report is favorable, the compound is then turned over to a number of different sections.

The Office of Research Section tries to improve the laboratory stand of making. When this has been done fairly satisfactorily, the Chemical Production Section starts making it on a large scale, from fifty
pounds to 1 ton, as the case may be. It then goes outside the Research Division either to the Development Division for further study or to the War Office Production Division for manufacture on a large scale. While the Offence Research Section is working out an improved laboratory method, the compound is sent to the Analytical Section to develop methods for determining its purity and for analyzing mixtures in air and the make tests to see whether the material is safe to the public.

The compound is also sent to the Pyrotechnic Section and study its behavior when fired in small. If it does not decompose too rapidly upon detonation, it is fired at a bench of the Provings Division in cooperation with the Pyrotechnic Section and the Toxicological Section to learn the effect when small quantities of the compound are fired from field fortresses. The compound is fired from guns on the range with a shell placed directly in or near the traverse.

While this is going on, the Defence Production Section is studying the change in the ingredients in the mixture as necessary. If the compound is not produced readily, some new mixture or compound may be developed to meet the emergency. This section together with other sections will also work out methods for extracting the gas in the field.

The problem of skin, clothing and special clothing to protect against such things as mustard gas is a problem which is attacked by one or more branches of the defence organization. If a change in the calibrator is necessary, this is referred to the Mechanical Research Section and they lay out to develop new filters to protect against smoke. The detail in regard to protective clothing are handled in part by this section. The results obtained by the defence organization are transmitted to the Defence Pro-
duction Division which has charge of the commercial production of all
defense material.

The Medical Division also studies the effects of different men, pathological details,
and methods of treating various cases. With mustard gas it is found that
about two percent of the non-tested are extremely sensitive and about
two percent extremely insensitive, the supersensitive man being perhaps one
hundred times as sensitive as the insensitive man. This is a very im-
portant test to make because men who are extremely susceptible are practical-
ly certain to become sensitive if set to manufacturing mustard gas.
The first work of the Research Division consisted in developing the mask and the absorbents, because it was absolutely essential to protect the soldiers against the gases which the Germans were using. On the other hand it is rather more interesting to start with the offense side. In addition to studying the gases already in use by the Germans, it was desired to develop better ones if possible. As a preliminary step this involved a search of the literature to see what was known in regard to toxic gases. Professor Bakerville sent in an especially long list of compounds that might be of use and others furnished similar information. Professor E. E. Reid organized the chemical laboratories of the country to a considerable extent, getting the various organic chemists to prepare samples of different substances for toxicity tests. After the American University Experiment Station got into full swing, this work was taken over by Dr. (afterwards Lt. Col.) Norris.

It was of course necessary to know the toxicity of these substances and methods were at once developed for doing this. The results were obtained almost exclusively at the American University Experiment Station by the Research Division; but this is purely a consequence of the particular organization and it is therefore wiser to present this work under the Medical Division, which is where it belongs theoretically, though not actually.

In the summer of 1917 the gases which the American Army expected to use on the Western Front were chlorine, phosgene, chloropicrin, hydrocyanic acid, bromoacetone, and xylyl bromide, with phosphorus and stannic chloride as the smokes. It was also hoped to use acrolin because it
was not stopped readily by the German mask. A good deal of thought was
also given to possible methods of producing high concentration of carbon
monoxide because no mask stopped that satisfactorily at that time.
Superpalite was another gas which might be used, though that went into
the discard early, because it was harder to make than phosgene and seemed
to have no special advantage. It was not till much later that the Allies
discovered that the toxic effect of superpalite increased enormously on
long exposures, it being, for instance, much less toxic to dogs than
hydrocyanic acid on a thirty-minute exposure and more toxic if the exposure
lasts two hours.

The use of mustard gas by the Germans in the summer of 1917
and of diphenylochlorarsine in the autumn changed the view-point considerably
and the possibility of using cyanogen chloride, cyanogen bromide, or arsine
made a great deal of research work necessary, while the development of
brombenzyl cyanide by the French and of chloracetophenone in this country
destroyed all interest in such lacrymators as bromacetone and xylyl
bromide.
In the autumn of 1917 it became evident that the Allies must make mustard gas in large quantities and the question of the method of manufacture became pressing. Victor Meyer met this in a roundabout way by what is now known as the chlorhydrin method. Chlorine and water react to some extent to form hypochlorous acid and hydrochloric acid according to the equation:

$$\text{Cl}_2 + \text{H}_2\text{O} \rightarrow \text{HClO} + \text{HCl}$$

Hypochlorous acid will react with sodium to form sodium hypochlorite:

$$\text{C}_2\text{H}_4\text{OCl} + \text{Na}_2 \rightarrow (\text{CH}_2\text{OH}-\text{CHO})_2\text{S} + 2\text{NaCl}$$

Thiodiglycol reacts with concentrated hydrochloric acid to form mustard gas and water:

$$(\text{CH}_2\text{OH}-\text{CHO})_2\text{S} + 2\text{HCl} \rightarrow (\text{CH}_2\text{ClOH}_2\text{S} + \text{CH}_2\text{O}$$

In addition to the inevitable fact that reactions rarely run quantitatively, there are the additional difficulties that chlorine reacts to some extent directly with ethylene forming ethylene dicloride:

$$\text{C}_2\text{H}_4 + \text{Cl}_2 \rightarrow \text{C}_2\text{H}_4\text{Cl}_2$$

and that the process involves a great deal of evaporation which is expensive. The commercial herbicide does not present the problem of making relatively cheap oil. At first investigation, the manufacture of propylene glycol was consequently the question of making propylene glycol by the chlorhydrin method, but this has been taken up by Dr. O. Grim and Dr. C. P. Wintz in charge of the Applied Research at the Indiana University Agricultural Experiment Station. The details of the process were worked out to be a short Research Station Bulletin No. 1968, by Professor Daniel R. S. Lawrence, Professor of Chemistry at the University of Indiana.
A contract for making mustard gas by this method was made with the Commercial Research Company; but the latter was dissolved. This is the method by which the Germans made all their mustard gas. The difficult step in it is the manufacture of the chloropicrin. As this was a commercial product in Germany, it apparently never occurred to the Germans to get it themselves and they were supplied by consignment because they never succeeded in making mustard gas on a really large scale. When they had accumulated enough stock, they made a mustard gas attack and then had to cut several months until they could get another supply.

On paper the simplest method to make mustard gas is by the interaction of styrene and sulphur dichloride

$$2\text{C}_{6}\text{H}_5\text{Cl} + \text{H}_2\text{S} \rightarrow (\text{C}_6\text{H}_5\text{CH}_2)_2\text{S}.$$  

This was actually made at the Mellon Institute in the autumn of 1917. The first reaction is the formation of styrene gas; but condensation usually takes place rapidly according to the equation

$$(\text{C}_6\text{H}_5\text{CH}_2)_2\text{S} + \text{H}_2\text{S} \rightarrow \text{C}_6\text{H}_5\text{CH}_2\text{SH} + \text{C}_6\text{H}_5\text{CH} = \text{C}_6\text{H}_5\text{CH}_2\text{H} + \text{S}_2\text{H}_2 + \text{H}_2\text{O}.$$  

This product is not toxic but boils at about the same temperature as water and would give rise to much confusion until the freezing point is adopted as the criterion of purity.

Early in January 1918 the Whitley stated that Professor Hope had succeeded in making impure mustard gas by this method. A little later Hope made the important discovery that the pure substance was a gas prepared by the interaction of styrene and sulphur monochloride at 100°C. The equation may be written

$$2\text{C}_6\text{H}_5\text{Cl} + \text{H}_2\text{S} \rightarrow (\text{C}_6\text{H}_5\text{CH}_2)_2\text{S} + \text{H}_2\text{O}.$$  

But it was observed by Drouard (1897-1898) that the reaction

$$2\text{C}_6\text{H}_5\text{Cl} + \text{H}_2\text{S} \rightarrow (\text{C}_6\text{H}_5\text{CH}_2)_2\text{S} + \text{H}_2\text{O}$$

occurred only in a very low yield, the main thing
\[ \text{C}_2\text{H}_4 + \text{S}_2\text{Cl}_2 \rightarrow \text{CH}_2\text{Cl}_2 \cdot \text{SiH}_4 \cdot \text{SCL} + \text{S} \]

The intermediate product is unstable, splitting off hydrochloric acid readily and going over into thick, black, unvitable oil. To keep the amount of this product to a minimum it is evidently desirable to avoid the concentration of ethylene as high as possible. It is practicable, certainly, to have in a reversible equilibration between sulphur monochloride, sulphur dichloride, and sulphur,

\[ \text{S}_2\text{Cl}_2 \rightleftharpoons \text{SCL}_2 + \text{S} \]

and that the actual reacting substance is sulphur dichloride at low concentration.

In taking sulfur for the sulphur dichloride process it is not advantageous to go over 60°C because the trichloroethane produced too readily and an aqueous product is obtained. A lower temperature of the reaction takes place more slowly, but it can then still be avoided in a tank and this can be counterbalanced by using moderately as a solvent and thus the sulphur monochloride product is made.

In this scale it is practicable to operate at 40-50°C. Reaction is fairly rapid in port, 1-1-2, keep- ing air under pressure in excess, relatively at a considerable time. Saturated dioxide is added, and the high concentration there to the speed of reaction sufficiently to compensate for the lower reaction velocity due to the lower temperature. The great amount of sulfur at this temperature is that the free sulphur is kept more easily in colloidal solution, the product being a clear yellow liquid when the monochloro:sulphur ratio on hands in air at a higher temperature. It is possible to keep the sulphur in suspension by dissolving 0.2 percent of metallic antimony in sulphur dichloride. The colloidal sulphur can be precipitated by a tin.
mixture to 120° for a few hours and then cooling, or by passing in a little ammonia gas.

The yield of mustard gas is about 85% of the theoretical but only about 50% of the theoretical amount of free sulphur; the balance probably being present as organic polysulphides, of which one very possibly has the formula

$$(\text{CH}_2\text{ClCH}_2)_2\text{S}_2$$

It is thought probable that this product is formed according to the equation

$$2\text{CH}_2\text{ClCH}_2\text{Cl} + (\text{CH}_2\text{ClCH}_2)_2\text{S}_2 \rightarrow 2\text{S}_2\text{Cl}_2$$

The English made ethylene by the action of concentrated phosphoric acid on alcohol. This was tried out by the Small-Scale Manufacturing Section but was not found so satisfactory as the catalytic decomposition of alcohol by means of hairlin as recommended by the French. Improvements introduced here were the thorough scrubbing of the ethylene and the subsequent drying with sulphuric acid together with the use of filter blocks through which to introduce the ethylene into the reaction vessel. About 700 lb. of mustard gas were made in 1 ton in by the research division before the process was turned over to others for development on a large scale.

A similar procedure was followed with other gases. The laboratory methods were worked out by the Oxygen Chemical Development Section; a considerable amount of the material was made by the Small-Scale Manufacturing Section and then the process was turned over to the Development Division or to the Oxygen Production Division as the case might be.
Research division kept in fairly close touch with the large-scale production, so as to be able to straighten out difficulties that might arise.

In carrying out this programme, the Small-Scale Manufacturing Section made approximately the following amounts of certain gases: mustard gas, 700 lbs.; bromobenzyl cyanide, 600 lbs.; butyl mercaptan, 1100 lbs.; chloracetophenone, 1000 lbs.; diphenylchloroamine, 4000 lbs.; nitrogen tetroxide, 3000 lbs.; dichloromethyl arsine, 2000 lbs.; diphenylamine chlor-arsine, 4000 lbs.; cyanogen chloride, 4600 lbs.; cyanogen arsine, 500 lbs.; liquid arsine, 2000 lbs.; superphosphate, 800 lbs. The manufacture of some of these called for a very extensive preliminary scientific study. For instance, diphenyl chloroamine was being used by the Germans; the Allies had any idea how it could be made satisfactorily. It was finally discovered that it could be made in two stages, by the interaction of sodium, chloroform, and arsenic trichloride to form triphenyl arsine

\[ \text{Na} + 3\text{C}_6\text{H}_5\text{Cl} + \text{ArCl}_3 = (\text{C}_6\text{H}_5)\text{Ar} + 6\text{HCl} \]

and then heating triphenyl arsine with arsenic trichloride in an autoclave,

\[ 2(\text{C}_6\text{H}_5)\text{Ar} + \text{ArCl}_3 = 3(\text{C}_6\text{H}_5)\text{ArCl} \]

The real problem was not to make the reactions involved but how to handle the sodium properly. The British did it in small lumps; but the American method seemed a better one. Twenty-two blocks of sodium weighing 2.5 lbs. each were placed on an 8-mesh screen and melted; the oxide and sodium remained on the screen. The melted sodium was driven by carbon dioxide pressure through a special sprue, nozzle into cold benzene which was stirred vigorously. This gave a finely divided sodium which reacted readily with chlorobenzene and arsenic trichloride. If the sodium is run in too rapidly, so much benzene is vaporized that a fog is formed.
After the reaction has taken place, the sodium chloride sludge is filtered and vacuum-treated with steam to recover some of the benzene. The yield of triphenyl arsine is about 85% of a product 95% pure.

The German used ethylidichloroarsine apparently because they had no satisfactory method of making methyl dichlor arsine, which is more satisfactory substance. The Research Division developed methods for making both. The methyl dichlor arsine can be made in three stages, starting with sodium arsenite and making from this diarsine methyl arsenite, which is then converted into methyl arsine oxide. The first reaction is carried out at 85°.

(1) \[ \text{Na}_2\text{AsO}_3 + (\text{CH}_3)_2\text{SO}_4 = \text{Na}_2\text{CH}_3\text{AsO}_3 + \text{NaCH}_3\text{SO}_4 \]
Possible disturbing side reactions are:
\[ (\text{CH}_3)_2\text{SO}_4 + \text{NaOH} = \text{NaCH}_3\text{SO}_4 + \text{CH}_3\text{OH} \]
\[ (\text{CH}_3)_2\text{SCl} + \text{H}_2\text{O} = \text{CH}_3\text{HClO} + \text{CH}_3\text{OH} \]
Sulphur dioxide is then passed in at 50° - 55°.

(2) \[ \text{Na}_2\text{CH}_3\text{AsO}_3 + 2\text{SO}_2 + \text{H}_2\text{O} = \text{CH}_3\text{AsO}_3 + 2\text{NaSO}_4 + \text{NaSO}_3 \]
The bisulphite thus formed is decomposed with sulphuric acid because otherwise if sulphur dioxide were not free in presence of methyl dichlor arsine, the escaping gas would carry off a great part of the methyl dichlor arsine with it. The methyl arsine oxide is then converted into methyl dichlor arsine by the action of hydrochloric acid at a temperature above 85°.

(3) \[ \text{CH}_3\text{AsO} + \text{HCl} = \text{CH}_3\text{AsCl}_2 + \text{H}_2\text{O} \]
An arsenic compound which formed an excellent smoke was obtained by Dr. (afterwards Major) Adams through the condensation of arsenic trichloride with diphenylamine. The reaction is

\[ (C_6H_5)_2NH + AsCl_3 = C_6H_5-N-C_6H_5•AsCl + 2HCl. \]

The same product was discovered independently but a little later by the British, who assigned to it the structure formula \( C_6H_4-AsCl-C_6H_4 \). The substance, which is known as D. M., forms yellow crystals melting at 195°C. It is strongly irritant to the respiratory tract. A concentration of 0.3 p.p.m. produces a very irritant atmosphere, causing coughing (in some observers), pains in the chest, and to a less extent in the throat and nose, together with the general symptoms of nasal catarrh. Qualitatively the physiological effects of pure D. M. differ considerably from those of diphenylchlorarsine. They develop more slowly and last longer. The method of manufacture is very simple. The arsenic trichloride and diphenylamine are heated together until the temperature of the reacting mass is 190°C, the temperature is held between 170°C and 180°C, at which point xylene is added to dissolve the D. M. The solution is drawn off, allowed to cool, and the crystals removed by filtration. The yield in the small-scale manufacturing was about 64% of the theoretical.

Cyanoarsenic chloride had been made by the French and their method was adopted here, but a good deal of work had to be done in regard to details. The general method is very simple and consists in the chlorination...
of aqueous hydrocyanic acid at low temperatures.

\[ \text{HNC} + \text{Cl}_2 \rightarrow \text{NHCl} + \text{HCl}. \]

Two disturbing side-reactions are the hydrolysis of cyanogen chloride in presence of hydrochloric acid

\[ \text{CNCl} + \text{H}_2\text{O} = \text{CO}_2 + \text{H}_2\text{CNCl}, \]

and the polymerization of cyanogen chloride in presence of chlorine.

\[ 3\text{CNCl} = (\text{CNCl})_3 \]

The actual method was to add chlorine to 8% HCN at temperatures not lower than \(-20^\circ\text{C}\) or higher than \(+20^\circ\text{C}\). This extremely narrow temperature range is necessary because the catalytic decomposition by hydrochloric acid becomes serious at higher temperatures, and at low temperatures the rate of chlorination is too slow. After chlorination a little hydrocyanic acid is added to react with all excess chlorine and the product is distilled in a jacketed, discontinuous still with a reflux cooler kept at 60-90\(^\circ\text{C}\).

The balance of the hydrochloric acid is removed by purging through a tower filled with limestone. The water content can be cut down to 0.1% by means of anhydrous calcium chloride; but this is unnecessary because the product is quite stable in saturated with water (about 1%).

Chlorocetanilinone was made : chlorinating acetic acid to monochloroacetic acid, converting the to dichloroacetic acid, and then causing this product to react with benzene in presence of sodium chloride.

The chlorination of acetic acid is run at the initial point in presence of sulphur monochloride.

\[ (\text{I}) \quad \text{Ca}_3\text{CO}_3 + \text{Cl}_2 (\text{S}_2\text{Cl}_2) \rightarrow \text{CH}_2\text{ClCO}_2\text{H} + \text{HCl} \]

The necessary conditions for a high yield of monochloroacetic acid and a
low one of dichloroacetic acid are effective stirring and rapid addition of chlorine. It is essential to use pure dichloroacetic acid for the next stage which consists in the chlorination of chloroacetone chloride at 40° in presence of sulphur chloride.

(2) \(4\text{CH}_2\text{ClCO}_2\text{H} + \text{S}_2\text{Cl}_2 + 3\text{Cl}_2 = 4\text{CH}_2\text{ClCO}_2\text{Cl} + 2\text{S}_2\text{Cl}_2 + 4\text{Cl}_2\).

The third stage consists in the reaction between chloroacetone chloride and benzene in presence of sulphur chloride.

(3) \(\text{CH}_2\text{ClCO}_2\text{Cl} + \text{C}_6\text{H}_5\text{(HCl)} = \text{C}_6\text{H}_5\text{CCOCH}_2\text{Cl} + \text{HCl}\).

Chloracetophenone is an extremely poisonous substance to which it is not only a violent incriminator but also extremely painful in its effect on the skin and on the mucous membranes of the mouth and throat. Contact with the substance either in the solid or the concentration of the substance in intense burning sensation of the skin which lasts several hours. Washing with water merely aggravates the matter and should be resorted to at little as possible. The best and quickest relief it obtained by washing repeatedly with portions of benzene or alcohol to dissolve the product and also carry it away by evaporation. On cool dry days the incriminating effect is much decreased and it is sometimes damped in very rainy weather. The dry crystals can be handled with comparative comfort due to the limited vaporization at ordinary temperature, while a solution of the product in a readily volatile liquid, such as benzene or alcohol, is much more violent in its effects. Benzene is also an excellent carrier.

In handling chloracetophenone, rubber gloves offer limited protection to the product even works through the rubber. The fabric, rubbered by the Adolph Company were better in this respect but were
objectionable in being so inelastic and prone to crack. Due
tight goggles and to be worn in the plant practically all the
time, and marks were necessary during certain operations.

Bromobenzyl cyanide can be made by converting benzyl chloride
into benzyl cyanide

\[ \text{C}_6\text{H}_5\text{CH}_2\text{Cl} + \text{H}_2\text{C} = \text{C}_6\text{H}_5\text{CH}_2\text{Cl} + \text{HCl} \]

and brominating the latter

\[ \text{C}_6\text{H}_5\text{CH}_2\text{Cl} + \text{Br}_2 = \text{C}_6\text{H}_5\text{CBrCH}_2\text{Cl} + \text{HBr} \]

The method employed in this country was based on the French
method but involved a number of modifications.

Dry sodium cyanide will not react with benzyl chloride, but
a saturated aqueous solution of sodium cyanide reacts very
slowly at the boiling point of the mixture (about 80°C) even
when the stirring is very vigorous. This seems to be due in
large part to the instability of the two liquids for the reac-
tion does practically to completion in three hours if there
is an amount of 95% alcohol equi in volume to the benzyl
cyanide. It is essential that the benzyl chloride should be
fairly pure, about five percent at its boiling point at 175°C
and 185°C, the boiling point of pure benzyl cyanide being 175°C.

Free sodium in the benzyl cyanide renders the reaction between
sodium cyanide and benzyl cyanide — sodium cyanide would be
eliminated unless it were bound —uppy in other com-
plished compounds made into form in the purification of the
benzyl cyanide.

After completion of the reaction about 70% of the oil
layer is the still in fairly pure benzyl cyanide, while the re-

\[ \text{B.} \]
minin: 30% is composed of tarry substances, the isolation of which seems unavoidable since no method has been found of pre-
venting their occurrence. After most of the alcohol has been
removed by distillation, the crude benzyl cyanide may be sub-
jected to fractional distillation to purify it from unchanged
benzyl cyanide and from the tarry matter; but this entails the
removal of the residues from the fractionating still, which
is a very difficult and dangerous manufacturing operation.
The desired result is secured by inroducing live steam im-
mmediately after the removal of the alcohol and without removing
the sodium cyanide solution. The oil remains on top of the
aqueous solution which facilitates greatly its removal by the
steam. The distillation proceeds very slowly, so increase in the
amount of oil, as no increase in the yield of unpurified benzyl cya-
nide.

The crude crude benzyl cyanide must be purified by fractional
distillation. This involves adjusting pressure in the atmo-
spheric pressure and a very careful manipulation of the equip-
ment. After distillation under vacuum, the crude benzyl cyanide
will have a b.p. near 112° C., the boiling point of the pure compound.

Several methods were developed subsequently; but the fractional vacuum
distillation over the tarry material was adopted. The vacuum was set at
45.5 inches under which conditions the fraction boiling point of
crude benzyl cyanide is 115° C. It must not be in contact with the crude benzyl cyanide, because any uncrude benzyl


chloride present will react with the iron with sudden evolution of heat, the reaction taking place with explosive violence. Furthermore, any iron salts which may be carried over interfere with the subsequent bromination. It is therefore necessary to line all iron apparatus. Brass is unreliable at high temperatures and consequently the fractionating still must be lined with lead. Besides giving a poorer product, the distillation under diminished pressure has the advantage of lessening the danger of melting the lead lining of the still.

Key It is possible to get complete conversion of benzyl cyanide to bromobenzyl cyanide if conditions are arranged carefully. The pure benzyl cyanide is heated to 70° C and a definite mixture of dry air and bromine vapor is admitted. For a given amount of bromobenzyl cyanide, the theoretical amount of bromine vapor is used and the corresponding theoretical amount of hydrobromic acid is evolved. The reaction must be started by sunlight, artificial light, or by.condensing some bromobenzyl cyanide made in a previous batch. This latter practice is of the greatest advantage for manufacturing purposes and is a sure way of starting the reaction, which then goes to completion without further aid. The hydrobromic acid can be obtained easily in a or on in a vacuum oven solution.

The bromine must be added at once. If any liquid bromine comes in contact with the benzyl cyanide, it is a violent reaction and this has no laryngic power. The bromine vapor must not be introduced too rapidly or a high concentration leads to the formation of the same dibromide. Sufficient air must be ad-
mitted with the bromine to sweep out the hydrobromic acid or else a hydrobromide may be formed. Too much air must not be admitted because the bromine passes through without reacting. Iron must not be present because it acts as a catalyst, causing the bromine to substitute in the ring instead of in the side chain. Moisture must be kept out. The bromine vapor may be cut off during a run if desired; but it is very important not to cut off the air current, unless air alone has been run through long enough to remove all the hydrobromic acid.

When pure, bromencyl cyanide melts at 49°C; but such a product can be cut into only by recrystallizing from alcohol and this is not necessary for our purposes. The melting point of the manufactured product varies between 15°C and 25°C, depending on the purity of the recrystallized bromencyl cyanide. The product is never a very solid on crystallization, there always being a slight amount of oil, which is best removed while the crystals are yellow. The highly purified product is colorless but soon turns yellow owing to impurities decomposition, which apparently stops soon.

Bromencyl cyanide is an extremely persistent compound, a number of the older vivid colors literary being dyed in bromencyl cyanide and the color is fast when dyed in a bromine pit in a week later it is impossible to work in the same pit without a week for more than a few minutes owing to severe irritation. The bromencyl cyanide film will prevent in spite of the fact that the bromine pit has been washed to a fearful extent with water and had been filled with cold water to rotation.
objectionable in itself, so plastic and prone to crack that most paper had to be wound in the plant practically all the time, and was more necessary during certain operations.

Butyl mercaptan was made by the method outlined by Section; but all the details had to be worked out here. The laboratory method was developed by Dr. Reid at the Johns Hopkins University. The general method is to pass butyl alcohol vapor and hydrogen sulfide over a specially prepared thorium heated to about 400°.

\[ \text{C}_8\text{H}_{16}\text{O} + \text{H}_2\text{S} = \text{C}_8\text{H}_{16}\text{SH} + \text{H}_2\text{O} \]

The catalytic agent is prepared by mixing peroxide with a saturated solution of thorium nitrate, drying in air, and igniting, at 450° in an air current. The final product contains about 24% thorium. There is danger of a side-reaction, the butyl alcohol or H₂S off water to form butyl mercaptan instead of reacting with the hydrocarbon. It is a very satisfactory reductant, somewhat analogous to diphenylamine was prepared by Dr. Kuhn by the interaction of diphenylamine and acetylene trioxide. This product, usually known as DPA, was made in quantity not very nearly as early as the others.

At one time it was thought possible that certain fluorine compounds might be desirable and a method of obtaining fluorine was needed. Hedin had prepared fluorine by electrolysis at a low temperature by applying electricity to a mixture of potassium fluoride and water. Hedin used platinum electrodes; but it was found that the amount of corrosion was too large to take this as a possible method on any except the smallest scale. Very early in his work, Hedin had to use platinum chloride and to prepare chlorine electrolytically. It was found, however, that fluorine could be cut into very small pieces by electroly-
lyzing a fused bath of acid potassium fluoride at 225° - 250°C in a copper containing vessel using a graphite anode. It is probable that still better results could be obtained with a graphite anode, graphite diaphragm, and graphite containing vessel, this last serving as cathode. No actual use was found for the fluorine and the method was not pushed beyond the laboratory stage.

Boron trifluoride was prepared by heating together boron trioxide, calcium fluoride, and concentrated sulphuric acid. It proved not to be sufficiently toxic for use in chemical warfare and was not a fluorinating agent. No experiments as to the joint action of boron trifluoride and fluorine on organic compounds.
During the war many suggestions were made that arsenic should be used. The popular plan was to use magnesium arsenide which would hydrolyze in moist air, setting free arsine. The experiments made by the Research Division showed that the hydrolysis does not take place rapidly enough under ordinary conditions to give an effective concentration of arsine. Experiments were still underway to determine whether this material could be used effectively in the rain. While the use of magnesium arsenide or of any arsenide was not very promising, there seemed to be a distinct possibility of using liquid arsine. The gas is fairly instable and decomposes into arsenic and hydroxide. On the other hand, the liquid seems to be pretty stable when dry and prevents the decomposition of the vapor. The reason for this is not known definitely. It seems probably that arsine as gas decomposes only in contact with the walls of the containing vessel and that this decomposition is prevented when the walls are wetted with liquid arsine.

The manufacture of arsine takes place in two stages, the formation of zinc arsenide and the decomposition of it. Zinc arsenide is made in a cast-iron kettle, painted on the inside with a mixture of graphite and oil to prevent the reaction mixture adhering to the walls. Equal weights of zinc, 10-20 mesh, and metallic arsenic, 10-60 mesh, are well mixed, charged into the cold kettle, covered with a layer of charcoal, and heated for 30-40 minutes. Then there is a strong evolution of brown fumes, the heating is stopped. The reaction mixture, when cooled, is a brittle coke-like mass which can be removed easily from the kettle. The reaction can be written

\[ 3 \text{Zn} + 2\text{As} \rightarrow \text{Zn}_3\text{As}_2. \]
It is important that the zinc should contain less than two percent of oxide and the arsenic less than five percent. The heat of reaction of pure zinc and pure arsenic is relatively low and the reaction is not nearly so violent as with magnesium and arsenic. When there are oxides present the reaction becomes more violent, other compounds being formed.

\[
\text{As}_2\text{O}_3 + 3\text{Zn} = 2\text{As} + 3\text{ZnO}
\]

\[
3\text{ZnO} + \text{As}_2\text{O}_3 = 2\text{Zn}_2(\text{As}_2\text{O}_5)_2
\]

The zinc and arsenic must be mixed in about the proportions indicated.

When an excess of zinc is used, the arsenic obtained by decomposing the arsenide contains so much hydrogen as to make the problem of liquefaction a very serious one.

Arsenic is obtained from zinc arsenide by the action of dilute sulphuric acid, 20% by weight, according to the equation

\[
2\text{Zn}_3\text{As}_2 + 3\text{H}_2\text{SO}_4 = 2\text{ZnAs}_2 + 3\text{ZnSO}_4
\]

When arsine is cooled in presence of moisture, a white crystalline hydrate is formed, (AsH₃·H₂O), which is slightly less dense than liquid arsine, is soluble in it to the extent of 0.3%, and decomposes readily to give metallic arsenic, which may precipitate reddish or in the black metallic form, according to the conditions. It is absolutely essential to remove all moisture from the gas. This can be done by passing the gas over quick-lime. After the arsine has been liquefied under pressure at a temperature a about -5°C, most of the hydrogen can be separated readily. Owing to the presence of hydrogen it is necessary to run up to 115 lbs. pressure to liquify the arsine, instead of the 96 lbs. theoretically necessary. The containers for liquid arsine are steel cylinders. The gaseous arsine analyzes about 33.4% AsH₃, the remainder consisting of hydrogen and air.

The manufacture of liquid arsine was distinctly a creditable performance.
3.

Since the gas is readily inflammable its tactical use presents some difficulties and nobody knows the exact value of the substance as a war gas.
As a preliminary to the study of the properties of toxic gases and of the means of defending against them, it is necessary to be able to detect and determine these gases. An analytical and testing section was therefore one of the first to be established and it was kept busy from morning till night in spite of the fact that all the other sections co-operated in developing methods of analysis and testing. The details of analytical methods are not specially thrilling to anybody except a technically trained man, so it will perhaps be sufficient to say that satisfactory methods were worked out for analyzing every toxic gas with which the Research Division had to deal. Three typical cases may be mentioned however, the testing of canisters developed by the Gas Mask Research Section, the field tests for mustard gas developed by the Gas Mask Research Section and the Defense Chemical Research Section, and the special paint for shell developed by the Offence Chemical Research Section.

Canisters are tested on men and on machines. Multiple machines have been developed which will test eight canisters simultaneously at continuous flow or the gas-air mixture or at intermittent flow. The continuous flow machines are the easiest to construct and were made first. Since the man breathes through the canister intermittently, the results with the intermittent flow machines resemble more closely those encountered when masks are actually worn in gas. The intermittent flow machines are capable of wide variation both as to volume of air passing through and as to number of oscillations per minute. They can therefore be adjusted to simulate any type of rate of breathing. Comparison tests on men have shown that the intermittent machines give results in excellent agreement with man tests, are easier to run, and are much more accurate.
APPARATUS FOR TESTING EFFICIENCY OF CANISTER AGAINST CARBON MONOXIDE

BUREAU OF MINES-WASHINGTON, D.C.

WATER

GASOMETER

FLOWMETER

PRESSURE DROP

GENERATOR
FORMIC AND
CONC. SULPHURIC
ACIDS.

TO STD. HUMIDIFIER
AND PSYCHROMETER

THERMOMETERS

PSYCHROMETER
FLOWMETER

VACUUM
TO EFFLUENT
SAMPLING TUBE
TO MOUSE TEST

RUBBER BULB
OF WATER

TO ENTERING GAS
SAMPLING TUBE
A NEW MULTIPLE MACHINE FOR TESTING CANISTERS

WITH INTERMITTENT FLOW

(Near View)

PLATE XV
A NEW MULTIPLE MACHINE FOR TESTING CANISTERS

WITH INTERMITTENT FLOW

(End View)
because they do away with the personal idiosyncrasies of the men. This
does not mean that man tests should be abolished. They must always be
kept to provide for unexpected contingencies but they can be reduced
to a minimum with a great saving of time and friction.

In the earlier man tests the men were sent inside a gas
chamber; but afterwards the canisters were connected by tubing to the
gas chamber and the men sat outside the chamber. This made it possible
to run more tests simultaneously and had the further advantage that the
man in charge of the testing could determine for himself whether any
given canister had broken down or whether the report was due to nervous-
ness on the part of the subject. All the toxic gases can be detected
at concentrations which do no harm to the individual. There are two
extremes to be avoided against. The man who is testing the canister
may imagine that gas is coming through when that is not the case, or
he may be so anxious to avoid giving a false report as to continue the
test too long and consequently get passed slightly. With the men
accessible outside the chamber, it is a comparatively simple matter to
guard against both these possibilities.

The man test is only run until it is detected coming through
the canister; but the machine test can be run further. It is customary
to designate the time at which gas can be detected coming through the
canister as the "breakdown". Up to that all the gas has percolated by
the materials in the canister. The 10%, 30%, 50%, points, etc. are
the points at which 99%, 95%, 90%, etc. of the gas is stopped and 1%,
5%, 10%, etc. of the gas in the air comes through.

*Trained observers can detect mustard gas by smell at 0.1*
When testing the variations in absorbents, the absorbent is filled into a sample tube, of specified diameter, to a depth of 10 cm. by the standard method of filling, and gas passed through under definite conditions. The following summary of conditions for standard tube tests on absorbents were adopted as official by the Research Division and the Defense Production Division.

<table>
<thead>
<tr>
<th>Gas</th>
<th>Conc.</th>
<th>Rate per cm²/min.</th>
<th>Depth</th>
<th>Diam. cm.</th>
<th>Relative Humidity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chlorpirin</td>
<td>7,000</td>
<td>1,000</td>
<td>10</td>
<td>1.41</td>
<td>0</td>
</tr>
<tr>
<td>Chlorpirin</td>
<td>1,000</td>
<td>500</td>
<td>10</td>
<td>2.0</td>
<td>50</td>
</tr>
<tr>
<td>Phosgene (1)</td>
<td>10,000</td>
<td>500</td>
<td>10</td>
<td>2.0</td>
<td>50</td>
</tr>
<tr>
<td>Superpalite</td>
<td>1,000</td>
<td>500</td>
<td>10</td>
<td>2.0</td>
<td>50</td>
</tr>
<tr>
<td>Arsine</td>
<td>1,000</td>
<td>500</td>
<td>10</td>
<td>2.0</td>
<td>50</td>
</tr>
<tr>
<td>Hydrocyanic Acid</td>
<td>1,000</td>
<td>500</td>
<td>10</td>
<td>2.0</td>
<td>50</td>
</tr>
<tr>
<td>Cyanogen Chloride</td>
<td>1,000</td>
<td>500</td>
<td>10</td>
<td>2.0</td>
<td>50</td>
</tr>
<tr>
<td>Chlorine</td>
<td>10,000</td>
<td>500</td>
<td>10</td>
<td>2.0</td>
<td>50</td>
</tr>
<tr>
<td>Sulphur Dioxide</td>
<td>10,000</td>
<td>500</td>
<td>10</td>
<td>2.0</td>
<td>50</td>
</tr>
</tbody>
</table>

(1) Accelerated test.

If only one point is reported, it is assumed to be the copper flame break-point in the case of chlorpirin and the 99% efficiency point in all cases.

When testing variations in the quality of charcoal, the standard chlorpirin test with a concentration of 1,000 p.p.m. was for a long time the only one in use. As the charcoals improved, the time necessary to make a test became too long. Laboratory charcoals have stood up for 1200 minutes or more against chlorpirin. To enable more tests to be run, the accelerated chlorpirin test was introduced using
a concentration of 7000 p.p.m., decreasing the diameter of the tube, and doubling the rate of flow. The outcoming gas passes over a copper spiral heated by a burner. The appearance of a green flame is taken as the end-point. Just before the green flame is visible there appears a preliminary orange color which may be used as a warning. It is not known whether the appearance of the orange flame would make a more sensitive end-point or not.

It is usually assumed that the service times with the standard method are fourteen times those with the accelerated method, and this factor is often used. There are two sources of error here. The air is run through moist in the standard test and dry in the accelerated test. It is quite possible, though not proved, that the relative effect of moisture might vary with different charcoals. The second source of error is a real one. It has been shown by Dr. Patrick that with the high concentrations of chloropicrin used in the accelerated test, there may be actual condensation of liquid chloropicrin in the pores of the absorbent. This chloropicrin is held loosely and will be given up readily when air passes over the charcoal. This actually occurs with the German charcoal. When this is run to the break-point with the accelerated chloropicrin test, and then dry air is passed over the charcoal, 70% of the chloropicrin is removed, whereas only 10% is lost by the American charcoal under the same conditions. An absorbent with a low absorbing power, but with a fine-grained structure may show up very well on the accelerated test and not well at all on the standard test. Since the concentrations of the standard test are more nearly those of the battlefield, the results with the standard test are the ones to be trusted.

Trained observers can detect mustard gas by smell at 0.1
p.p.m. (0.0007 mg. per liter); but only for the first minute or two of exposure. Low concentrations of mustard gas vapors, when in contact with a dilute solution of selenious acid, produce an orange-colored colloidal suspension of selenium which gradually increases to a deep brick-red color in time if the concentration of mustard gas is sufficient. The test is sensitive to about 1 p.p.m. (0.0007 mg. per liter). This method is not specific because arsine gives a similar precipitate in less time than does mustard gas, and other compounds such as diphenylchlorarsine and butyl mercuric give positive results. Against this, chlorine, hydrogen chloride, phosgene, chlorpicroin, and superphosphate give a negative test even when present in fairly high concentrations.

While the copper flame test is not sufficiently sensitive to permit of direct detection of low but toxic concentrations of mustard gas, it has been found possible to modify the method so that one can detect 0.1 p.p.m. (0.007 mg. per liter) or even 0.01 p.p.m. under special conditions. The method consists in passing air containing mustard gas through a quartz tube over a fine cylinder of copper gauze about four inches long, which fits snugly into the tube and which is heated at the tip at the inlet through the tube by a small flame. As the mustard gas passes over the heated tip of the copper gauze, it is broken down and the halogen is absorbed by the cooler portion of the gauze beyond. After a period of accumulation, the gauze is moved slowly into the flame and heated directly, the presence or absence of a green color being noted. The principle has been embodied in a portable field apparatus. The method is really one for halogens and is not specific for mustard gas. Its usefulness...
FIELD DETECTOR FOR G.34 IN USE.
4.

Experiments with snails and slugs showed that these animals were exceptionally sensitive to mustard gas and other toxic gases, moving their horns in different ways in different gases. It was found that a skilled observer could utilize these animals to detect mustard gas at concentrations as small as one part in three million and other toxic gases at even greater dilution. A trained observer could distinguish between the various toxic gases with considerable certainty at low concentrations and with entire certainty at higher concentrations. The variations of sensitivity are considerable with different individuals and in all cases the susceptibility disappears pretty completely on prolonged exposure. It is therefore doubtful whether this special test would have anything more than a confirmatory value in the field.

A lacquer enamel paint has been devised which can be used on shell to tell whether they leak or not, there being an instantaneous color change of one kind in contact with liquid mustard and a color change of another kind in contact with arsenicals, thus making it possible to use the same paint in the different kinds of shell. The enamel consists of chrome yellow as pigment, a mixture of 60% of Oil Scarlet and 15% of Rose-damone B in small amount as the sensitive agent, and a lacquer vehicle which is essentially a solution of nitrocellulose in anil acetate. Both the Oil Scarlet and the Rose-damone B turn crimson red in presence of liquid mustard, but the Rosdame B is much the more sensitive. On the other hand the Oil Scarlet turns deep purple to dark green in presence of arsenicals. The paint is not affected by such other substances as chloropicrin, phosgene, superphalite, and butyl mercaptan.
Similar effects can be obtained with a linseed oil paint instead of the lacquer enamel, the contracts being more marked with the paint but the enamel reacting more quickly. The paint has been tested thoroughly for the effect of sunlight, moisture, time and high temperature. The color is entirely stable before exposure to mustard gas and the red color produced by the mustard gas is equally stable under all conditions of weather. It is immaterial whether the paint is used on shell steel, tin copper, wood, cloth, or paper.
24.

In order to get some idea of how long mustard gas may be dangerous in the field, a study of the persistency under field conditions was made by the research division in the summer of 1918. The necessary testing equipment, and the more extensive equipment for protection of the operator, as well as meteorological instruments were borrowed from the other institutes, and set up in the vacantuding place on the farm. Some hundreds of plots, varying from 20 feet square to 100 feet square, were sprayed with mustard gas and other toxic materials. This was very dangerous work and required the utmost precautions and the maintenance of strict discipline. Five of the men escaped various burns; but, so far as is now known, no permanent injury resulted. Successful prosecution of these experiments required the development of a very accurate, quantitative method for the determination of minute traces of mustard gas and the other gases in the air. This was finally secured by a considerable development of the spectrometric values of mustard. It was found possible by this means to determine mustard gas with an accuracy of about 10^\text{-}5 concentrations at levels of 0.006 m. per liter (1 foot in 10,000 feet) and 1 liter per min.

The concentration of mustard was over 0.04 m. at 20 x 20 feet sprayed with 20 cc of mustard per sq. ft., which corresponds to an intense real-time, amounts to 0.04 m. per liter in one hour on a very (190 feet) and a slight breeze. At one foot from the source, it is only 0.01 m. per liter, and at 3 feet 0.002 m. per liter. Even standing erect in this area for any length of time would therefore be certain death, due to the severe nature of the toxic action of the gas itself, irrespective of any action of the soil itself, or continuously on exposed portions of the body. The concentration is much higher on a m.
days and lower on cool ones, but the percentage of decrease in concentration at increasing distance from the ground is practically identical for all temperatures. Thus, at 4" from the ground the concentration is 40%, at 12"/30%, and at 36"/8% of that at the surface of the ground. The variations in horizontal concentration were in part as expected and in part unexpected. Thus, as would be expected, at 4" from the ground the concentration reached its maximum at or near the leeward side of a sprayed area. However, it decreased at a very great velocity beyond this. For instance, in clear, moderate weather, and with a slight wind blowing the concentration at 1" from the ground is about 0.04 mg. per liter at the leeward edge. Ten feet beyond this, it dropped to 0.001 mg. per liter. The diluting effect of the wind is evidently surprisingly great. It has indeed been the universal experience in the literature. The toxic area does not extend more than about 100 feet beyond the sprayed area under ordinary conditions. Further, and even where water was very near the very root of the trees, the wind was very strong, and even under ordinary conditions, diluents make the effect less imperceptible. To the leeward of the porch, there was considerable variation in concentration at identical heights and distances from the sprayed ground. lesser decreases of the concentration and increased or no concentration were observed.

The effect of the temperature on concentration is, of course, pronounced. Thus, on a clear day at moderate temperature, the concentration on the leeward side of the plant at 4" from the ground is 0.025 mg. per liter, on hour after spraying. The same day (7°C) it was 0.004 mg. per liter.
and on a cold day (44°F.) it was 0.006 mg. per liter. These figures are approximately proportional to the vapor pressures of mustard gas at the respective temperatures. The effect of sunshine was surprisingly marked. The moment the sun went behind a cloud, an immediate drop in concentration could be observed. This drop often amounted to as much as 75% or 80%.

Curves of solar radiation taken on a recording pyrheliometer ran substantially parallel to curves showing the change in concentration with time, though this parallelism was more or less confused if the clouds were small and scattered, due to the fact that the recording instrument was located nearly one-half mile from the pithouses.

The change in concentration with time was studied with particular care. Immediately after morning the concentration close to the ground approached that of the lowest edge of the pitch. In a warm day, a very rapid decrease with the time takes place, so that after a hour, the concentration at 4" above the ground will have dropped to about 0.006 mg. per liter; in five hours to 0.010 mg. per liter; in ten hours it will be only 0.004 mg. the second day 0.002 mg. per liter, etc. A greater distance from the ground the concentrations will be correspondingly lower.

In a moderate day, the initial concentration is, of course, much lower, but the decrease will be much less marked, so that five hours after spraying there is little difference in the actual concentrations on a warm and a cold day. In the second day, the concentration at certain temperatures is even greater than after the spray, so that difference becomes even more marked after three days. The second orderexternally becomes so much
gas of the musty odor during the first five hours that the concentration becomes less on this account. In hot weather, therefore, the concentration of the vapor over a sprayed patch is very high, and, hence, dangerous during the first day; but is only slightly dangerous on the second and third days, and is negligible thereafter. In cool weather, however, the air remains innocuous for a week or even more, and is particularly innocuous on a sunny day following a cold one.

The nature of the soil was found to have an approximate effect on the gas concentration. Applying a larger amount of liquid per square foot of area increases the gas concentration, but not in proportion to the increased amount. Then the amount used is less than what is required to wet the surface of dry soil, the gas concentration is found to be very low. In other words, the soil acts as a retarding means proportioned to its lower yield in the direction of the wind.

Dry weather was found to affect the persistence of musty odor in two ways. First, the heat received by radiation during such weather is to retard the concentration of musty odor very little on a given amount. In the second place, the evaporation of the soil acts as the amount of water per square foot of the soil is proportionate to its concentration in the air.

The topography of the soil did not affect any persistency. The soil must be deep enough to reach the steam; otherwise the liquid is evaporated in the space between soil and ground. The liquid must evaporate as rapidly from
The ability to detect the odor of mustard gas under varying conditions, and at low temperatures was found to be subject to considerable individual variations and to depend on the amount of experience in such work. Many of the men acquired decided skill so that they could detect the odor at a foot from the ground over patches that had been sprayed with 20 cc of liquid to a square foot ten days after spraying. Over patches that had been sprayed with 1 cc per square foot they could detect the odor after three days under normal summer conditions. In the summer time, soil contaminated with mustard gas will cause skin burns after fifteen minute application for at least four days. After four days, the danger is relatively slight, depending no doubt on the amount of material originally applied to the soil and on the intervening weather conditions.
The work on protective clothing by the research division and the Medical Division has been chiefly with reference to protection against mustard gas and has included a study of fabrics intended for use in the following forms: gloves; over-all suits; underclothing; footwear; dugout blankets and screens; masks for horses and dogs; boots for horses. The ordinary materials such as cotton, wool, linen, leather, and rubber are penetrated by irritant gases in a relatively short-time, partly because of their porosity and partly because of solvent action, this latter being the important factor in the case of rubber. O.D. issued clothing protects from saturated vapor of mustard gas for 5-7 minutes. Untreated glove leather resists penetration by liquid mustard gas for 35 minutes; untreated cloth horse-hide and split cow-hide for four and five minutes.

**PROTECTIVE GLOVES**

Six different types of gloves have been tested. They are

1. Leather gloves impregnated with softened linseed oil.

2. Canton flannel gloves with leather palm, impregnated with boiled linseed oil and stearine pitch.

3. Rubber gloves.


5. Gloves sewed from oiled fabrics.

6. Flannel gloves coated with cellulose-nitrates.

The first type was made from thick leather impregnated with a mixture of boiled linseed oil, castor oil and paraffin. They give protection against liquid mustard gas for two to five hours. The disadvantage lies in the scarcity of leather, the tendency of the glove to shrink somewhat.
the necessity for oxidizing the linseed oil during the drying process and
the tendency of the glove to stiffen with ageing.

Gloves of the second type give protection of one hour on the
fabric part and four hours on the leather palm. The time required for
drying and the tendency of the glove to stiffen are its weak points.

Acid proof rubber gloves (the third type) are available in the
market. Samples which contain zinc oxide in the composition resist
liquid mustard for 15 to 45 minutes. The high price makes this type
unsuitable for anything but speculation.

A goldmine-glycerine-formaldehyde imregnation (type four) was
tried out, with a water-proof coating outside to protect the water-soluble
imregnation. Gloves that made are more flexible than any other type and
more resistant to mustard gas. The necessity for the outside interregning
introduced complications into the manufacturing process which are not
sufficiently under control to give this glove standing as the best developed.

The fifth type consists of an oiled-skin fabric reinforced
in the glove. This material was used formerly by the French, and is the most
resistant to mustard gas of them; it loses this distinction when it is
integrated into gloves, however, because of the tendency to break at the join.

Difficulties in rolling the stiffs and non-elasticity of the material are
additional disadvantages.

The sixth type of glove was found most suitable for large-scale
production and for general military use. The fabric selected was a
sufficient strength, flexibility and a smooth surface. For treatment
in a medium weight will high cotton value, it was a 1.20 cotton. This
is run over cylinders carrying short arms through which pull out the fibre
on one side, leaving a soft effect surface like that of ordinary cotton or wool.
The material is treated on the smooth side with a coat of cellulose nitrate to form the outer layer. After drying it is cut by dice and sewed with the treated side out into a glove with the thumb and first finger in separate divisions, the remaining three fingers forming a sitten. The glove is then put on a wooden form and dipped repeatedly into a solution of cellulose nitrate softened with rape-seed oil and dissolved in ether, benzol, methyl e-licylate and alcohol. After drying in oven, these gloves protect against liquid mustard for 30 to 60 minutes, depending upon the number of coatings. The gloves become softer rather than stiffer upon aging, and show no tendency to crack at temperature as low as -12° C.

In response to a request from the War Research Division for recommendations for producing 2,000,000 pair of protective gloves at a rate of 250,000 per month, the cellulose-nitrate glove as recommended and production started; about 1800 pair had been completed when work was stopped by the signing of the armistice.

**PROTECTIVE GLOVES.**

Protective suits of three types were experimented with. The classification made was as follows: impermeable suit, in which the textile has been thoroughly impregnated with drying oil so that the form in contact with the body is impermeable; impermeable, impregnated materials are used so as to render the form mildly impermeable by its solubility in the impregnating ingredient; impermeable suit, in which the fabric is impregnated with drying oil in such way that the interstitial remains open for ventilation.
A. Impermeable Suit.

The air-lined suit was developed for special use in factory production of poison gas. It comprises a suit covering the whole body, a helmet, gloves, shoes and air supply hose. The helmet is an aluminum cylinder ten inches in diameter with a hemispherical dome top, being supported by a fabric band resting on the head like a cap. A rectangular window is in front beneath which the air supply enters. The suit is made of oil-skin fabric made up as a union suit, buttoned at the back and tied at the wrists, ankles and around the helmet. The gloves and foot- wear are not distinctive. Air is pumped into the helmet at a rate of 400 liters per minute, giving a constant current from the inside of the suit, outward throughout seams and openings. In a 3-minute test in the gas chamber containing phosgene (1000 p.p.m.) no gas could be detected in any part of the suit. There was no discomfort reported due to . . . .

The Oil-Skin Suit. The material of this suit is especially prepared oil-skin. It is a fabric, lengthwise 56 25/32 by 54 x 54 (den) weaving giving a high tensile strength, which is very brittle. This is coated with three coats on each side of a so-called "rub," and then treated with a cover, the composition of the dieters is as follows:

<table>
<thead>
<tr>
<th>Material</th>
<th>Stock</th>
<th>Tension</th>
<th>Motor</th>
<th>Stock</th>
<th>Tension</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cotton</td>
<td>55.16&quot;</td>
<td>46.4&quot;</td>
<td>46.4&quot;</td>
<td>16.6&quot;</td>
<td>9.7&quot;</td>
</tr>
<tr>
<td>Rayon (54 pub)</td>
<td>11.3&quot;</td>
<td>11.10&quot;</td>
<td>11.10&quot;</td>
<td>35.4&quot;</td>
<td>0.25&quot;</td>
</tr>
<tr>
<td>Rayon (54 pub)</td>
<td>11.3&quot;</td>
<td>11.10&quot;</td>
<td>11.10&quot;</td>
<td>35.4&quot;</td>
<td>0.25&quot;</td>
</tr>
<tr>
<td>Cotton</td>
<td>57.6&quot;</td>
<td>8.74&quot;</td>
<td>8.74&quot;</td>
<td>51.56&quot;</td>
<td>9.54&quot;</td>
</tr>
</tbody>
</table>
AIR LINED HELMET AND SUIT

Front View

Showing suit inflated.
IMPervious overall suit for protection against HF.

Front View.
The cloth is dried in hot air-tunneling chambers, then cut and sewn into a one-piece overall garment, following in general the French design, but with changes permitting greater freedom of movement of the legs. After being sewed up, the suits are coated over the cover with a quick-drying mixture of the following composition.

5 parts cellulose nitrate
1 part butyl acetate
1 3/16 parts blown ragweed oil.

Samples from factory protection gave protection against liquid mustard gas from 60 to 90 minutes on the physiological test, which is distinctly higher than tested samples (reconditioned) of French protective suits (26 to 75 min.). In use in several types of exposed work here, they protected from injury even in every case. They are open to objection on the ground of comfort, since they permit no expectation of perspiration or ventilation.

In order for 1,000,000 chemical workers in process of coin-
rolled for the navy, the 200,000 clothed. About 200 suits are complete for the 2nd division.

Nitroglycerine suit: Specimen lock on site with all
suit connected to dynamite, suiting the
bomb. No suit to hit

Semi-passable suit

Oil of limonene for dynamic protection of the
suit against mustard gas, not used in present. Used in tests -
ter of such importance in reducing the discomfort due to accumulation of perspiration in the impermeable suit. The cotton fabric is treated by means of a "doctor knife" with a mixture of two arsenicals (citrate of zinc sulphate and preservative), and is then hung in an atmosphere of formaldehyde until the glue is thoroughly dry. After being thus treated, the glue becomes insoluble in water, i.e., the cotton fabric is protected against further treatment of the fabric after dying, as for instance by the treatment that is of no importance; for instance, the fabric is stretched out by water, rendering the fabric stiff after drying. The manner in which this point is of no importance; for instance, the fabric is treated with a salt to retain by incrustation in the fluid zinc "dust" mixture. A linseed oil emulsified in water by means of a mixer, upon drying, retains a lasting leaching effect of water.

When used in connection with ginseng in its different stages, the protection against liquid suit, when used up to 60 minutes, is held to be superior to that offered by the impermeable suit. For the manner of water vapor occurs first; the amount in this time ratio, from 50' to 80' of that occurring later, more than the ginseng. The gain in weight over the oil and all in very marke,

The Impermeable Suit

The Impermeable suit consists of the cotton of cotton, fabric, the inner suitings and lining and the outer covering, and protection of tinfoil, in which is composed of 45% raw and 55% cotton. This is wrapped up tightly by the rubber, leaving the side open for oxygenation of the air and for ventilation. The suit is impregnated in this time, placed between cotton and treated with hot water throughout and kept. It is made up into one piece or two with a hood, and at the front and above.
The suit is designed to meet the need of a front line fighting suit, in which the comfort of the individual will be sufficient to permit of strenuous exertion, which may not prove possible with the ironclad suit. It permits evaporation of moisture substantially no decrease from the rate through unrestricted suits, and possibly passage of air with even stability or pressure every. It is constantly, inletting constant upon lengthy exposure. It is not intended for protection against liquid or vapor, but as basement in field tanks to mitigate burns from liquid or vapor.

Flushed and wet, it gives protection at 30° for 15 minutes; 20 minutes protection at 40° C.

About 1000 of these suits were made up and several hundred
were used in field tests. These were built primarily completed when the battles ceased. The general conclusions reported is that it is thought the protection offered by this suit might enable men to work for
long hours without various dangers and open ground saturated with
mustard gas to work for a few hours through black smoke, which we now
must call "trench fumes," or work a long time when wounds are
open or have been lightly shelled. If not wearing, the suit should be
heavily gaited and kept well over a small suit, or if the suit becomes wet, actually wet with mustard or any cause the suit should be removed as soon as possible.

The helmet suit is covered with the helmet of Cernburg style, work impregnated with an immersion of the nine parts of cottonseed oil with
5 parts of cylinder oil, equal parts of each being used. In order to give higher protection to men of the Simplex dome suit, 8 ounce and 9 ounce Cernburg were
used as far as, which take a heavy load of solvent (55 mgs per sq cm. and
leave the interstices of small dimensions. In factory impregnations, the fabric must be passed over hot cylinders until all chemical action involved in the formation of the soap is at an end, to avoid danger from later heating up. The material is then sewed into overall suits, following the design of the simplexene suit. Compared with a summer suit of issued army clothing, it permits evaporation at a rate 70% as rapid, the pressure drop on passage of air is practically the same, and the heat conductivity distinctly better. Against saturated mustard vapor, it affords protection for 45 to 75 min.

About 75 of these suits were made up. They were not tested in the field. They appear to be less cool for summer wear than the simplexene, but much cooler than the impervious or semi-pervious suits; they lack entirely the stickiness and tendency to stiffen which the simplexene suit shows, and afford protection at least twice as great.

It is seen that the amyloid process (setting of the impregnation upon the fibre from zinc soap solution) is applicable to any oil, the one quoted being selected because of its cheapness. An essential difference noted in the various oils is the establishment as to their protective value. The general situation is that an oil (limida, etc.) should be added, either free or in the soap, in order to prevent stickiness to prevent use. A few water and rain suits should be involved an amount of their tendency to stick. The critical point is the mixing of the oil with the soap. The oil used should be free from lumps, too


defined from properties and would be put on in such form as to avoid stickiness and tendency to run, the most important of all, that the peculiar feature is related. It is therefore to the interest it will be exposed to modern stories, such as the use of dyes as soluble matter
 worn in conventional outer suits. With fabrics of a degree of openness comparable to that of usual wadding, a protection of 45 to 75 minutes is attainable; with open fabrics such as used in the Simplemore suit, the protection drops to 10 to 20 minutes.

**PROTECTIVE UNDERCLOTHING**

Protective underclothing has been tested, on the theory that a small amount of protecting material might have a maximum value if used directly over the skin. Cotton crinkling impregnated with oils or with waxes and resins give substantial protection for short time exposure (9 to 12 minutes) against saturated mustard vapor; the pressure drop is increased somewhat, but is still no lower to offer no serious impediment to ventilation and the rate of evaporation of water is almost unaffected. The rate of pressure drop is the same for all clothing materials tested.

**TESTS OF PROTECTIVE UNDERCLOTHING.**

<table>
<thead>
<tr>
<th>Impregnation</th>
<th>% per sq cm</th>
<th>Pressure Rate of Evaporation</th>
<th>Physiological Protection for 8-12 min.</th>
</tr>
</thead>
<tbody>
<tr>
<td>(None)</td>
<td>--</td>
<td>1.00</td>
<td>1.00</td>
</tr>
<tr>
<td>Alcohol</td>
<td>11</td>
<td>2.0</td>
<td>1.00</td>
</tr>
<tr>
<td>Mg Linoleate</td>
<td>28</td>
<td>5.0</td>
<td>1.70</td>
</tr>
<tr>
<td>Sulphonated</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Castor Oil</td>
<td>22</td>
<td>4.0</td>
<td>1.00</td>
</tr>
<tr>
<td>Castor Corn Oil</td>
<td>20</td>
<td>3.6</td>
<td>1.50</td>
</tr>
<tr>
<td>Ointment /60</td>
<td>33</td>
<td>6.0</td>
<td>1.20</td>
</tr>
<tr>
<td>Castor Oil</td>
<td>20</td>
<td>3.6</td>
<td>1.00</td>
</tr>
<tr>
<td>Ointment C.R.</td>
<td>27</td>
<td>4.9</td>
<td>1.00</td>
</tr>
<tr>
<td>Ointment /52</td>
<td>45</td>
<td>6.1</td>
<td>1.30</td>
</tr>
<tr>
<td>Ointment /45</td>
<td>45</td>
<td>5.0</td>
<td>1.00</td>
</tr>
<tr>
<td>Ointment /60</td>
<td>47</td>
<td>8.5</td>
<td>1.50</td>
</tr>
</tbody>
</table>

Impregnations running up to about 40% in weight can be worn with no discomfort and with no sensation of stiffness; with heavier impregnations,
the reaction is disagreeable. Fabrics treated by the Emulsion process with oil-spray impregnations up to 70-80% give very little reaction, and protect (by machine test) for 24 to 40 minutes. A 90% impregnation with resin oil mixture containing iodine pentoxide shows 99% protection for 16 minutes, but it is too oily for use. Tests with underclothing impregnated with oil and dichloramine-T or anilazine give high protection, but the fabrics are not lasting, owing to the instability of the active reagents.

**FECTWEAR**

Rubber boots and a special coat made from pyroxylin fabric were used as foot protection in experimental work in ground spraying with mustard gas. Special study of these articles was not made.

**LUGOUT CURTAINS**

The research was directed toward finding materials which would give a flexible curtain, impermeable to war gases (air also being excluded, since sufficient ventilation is ensured to occur in dug-outs through the soil), as free from stickiness as possible and as inflammable as possible. The work has included the development of a suitable blanket, suitable impregnating oil, and methods for impregnation.

Specifications for oil-cotton blankets have been based upon a study of the suitability of all available blanket materials. A tensile strength of 50 lbs. per inch in the warp and 95 lbs. per inch in the filling is necessary to prevent such stretching during use as will break the oil film and lessen protection. The most suitable material for impregnation consists of 85% of heavy white refined mineral oil, with 15% boiled linseed oil. On the blanket the linseed oil diffuses slowly and makes
an outer surface which is but slightly oily to the touch, while the inside core remains fluid and flexible. The imregnated blanket protects from chloroprene (400-600 p.p.m.) on 5 to 46 hours and from liquid acetone gas for 100 to 400 minutes (machine-test). It is not ignited by cigarette but or lighted match thrown upon it. It remains flexible enough to unroll under its own weight at 38° F, and at 6° is easily unrolled by applying force. The tendency to crinkle is negligible.

A field imregnating apparatus has been devised, consisting of a galvanized iron tub for holding the oil mixture, which is heated to about 70° C by a gasoline torch or other available means. In these the blankets are dipped singly, and drawn out through a perforated screen to remove excess oil. The desirable imregnation is about 300%, or about 14 lbs. of oil per blanket. About 191,000 blankets were manufactured by the 1st Marine Division following these recommendations.

**Masks**

The first mersilk developed was one of Patrick’s “Kemploone” mixture soaked into a flannel cloth bag to be worn over a respirator. The imregnating mixture is of the part, using the proportion:

- A -

<table>
<thead>
<tr>
<th>100 parts</th>
<th>250 parts</th>
<th>95 parts</th>
<th>70 parts</th>
</tr>
</thead>
<tbody>
<tr>
<td>n-propanol</td>
<td>acetone</td>
<td>water</td>
<td>n-butyl nitrate</td>
</tr>
</tbody>
</table>

Upon mixing these at 45° C or more, the solubility of acetone increases. The mask must then receive a shake, and moisture given it in the form of a wettable, or test in running water, only then.

A second type of mask has been developed, in which water, its resistance is obtained by use of a special type of rubber cloth; attention is
secured by use of 12 layers in the bag. The mask is 12 inches wide and 18 inches long, having a total area of about 400 sq. inches. In order to secure absorption of a large variety of gases as possible, the mask is made of the parts; the inner four layers are impregnated with Komplexene, as in the first mask, and the outer eight layers with a solution known as Simplexene, consisting of 80% resin oil and 20% paraffine oil. This mixture was selected after careful testing out of a large number of oils and mixtures of oils and soaps. The time to penetration of this mask by various gases at 50 litres per minute is as follows:

<table>
<thead>
<tr>
<th>Gas</th>
<th>Concent.</th>
<th>Time to Penetration</th>
<th>Active Absorbent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mustard</td>
<td>100 p.p.m.</td>
<td>80 min.</td>
<td>Simplexene</td>
</tr>
<tr>
<td>Phosgene</td>
<td>1675</td>
<td>130 &quot;</td>
<td>Komplexene</td>
</tr>
<tr>
<td>Chlorine</td>
<td>1500</td>
<td>65 &quot;</td>
<td>Simplexene</td>
</tr>
<tr>
<td>Chloropicrin</td>
<td>1500</td>
<td>2 &quot;</td>
<td>neither</td>
</tr>
</tbody>
</table>

The mask affords excellent protection against three of the four gases. It was adopted by the Gas Defense Division, after it had been shown by tests that a test layer that could stand for the allied side has not, without showing evidence of exhaustion, contracts for 1,500,000 men were made (without, however, the additional layer of 120 lb. Simplexene), and 377,300 were completed.

**Note:**

Protection of face in the tender portions of the head of soldier is first mustard gas and then provided for by a test layer over the head, from the nose to a line just below the knee, on the special mask.
IMPERVIOUS BOOTS AND PADS TO PROTECT HORSES' LEGS & HOOPS AGAINST BE.
1714, 1716, 1707.
Torts showed that wearing the boot and heel-pad for nine days and nights consecutively caused no damage to the horse; it is recommended, however, that the boot be removed and cleaned whenever the animal is in stable. They are good for six weeks use on rough roads.

**THEORY OF PROTECTION BY IMPREGNATED FABRIC.**

All protection by impregnated fabric is for its either purely mechanical or physical, depending upon solution of gas. In the case of linseed oil fabric, it is probable that mechanical exclusion of gas is most exact, the penetration being effected through microscopic holes in the film. However, it is possible that oxidized linseed oil (like unoxidized oil) is a solvent for some of the gases, and that slow solution occurs in the exposed film, after which the dissolved gas passes by diffusion into the interior of the fabric and finally to the inner surface, from which it diffuses. Experiments are not at hand throwing light upon this point.

In the various oiled fabrics (sacking blankets, Simplex suit, Hudson suit, protective underwear, horse and dog marks) it is obvious that solution is the mechanism by which the gas is taken up. It has been demonstrated by parallel experiments that the gas present in the oil with a detectable vapor tension; fabrics exposed to mustard vapor for a short period and allowed to remain upon the skin will not cause a burn, while they will cause a severe burn if dipped after the same exposure to water to prevent evaporation.

During the exposure of such an oiled fabric to gas, it will be gaining in concentration of dissolved gas on the exposed surface until equilibrium with the gaseous phase is reached. The time at which penetration...
occurs, however, may be entirely independent of the rate at which this
outside equilibrium is reached and is probably due to diffusion of dis-
solved gas in the body of the impregnation through to the inner surface,
from which it will at once begin to evaporate, there being no opposing
temperature upon that side. The plausibility of this explanation is
shown by the fact that the highest degree of resistance to penetration was
found, by a number of independent investigators, to be shown by the
most highly viscous oils, in which this diffusion would be slowest - cylinder
oil, rosin oil, castor oil, etc.

The penetration of hydraulic materials such as the gelatine-gly-
cerine impregnation, in which most gases are insoluble, may be due in part
to pin-holes (macroscopic or microscopic) or in part to transport in which
the water plays a part. The amount of water present in such fabrics has
been shown to influence the rate of penetration; with three fabrics (un-
treated heavy duck, gelatine-glycerine fabric, and oil-cloth) it has been
shown that increases in moisture content up to about 5% increase the amount
of mustard penetrating per unit of time, higher amounts than 5% reducing the
amount of penetration. The explanation advanced for this is that water
may play a double role; it may act (presently shown it amount is large) as
an absorbent, dissolving the gas and eventually saturating it by hydration,
but may also (when its amount is small) facilitate penetration by incre-
sing surface adsorption on the fiber of impregnation and by raising the
gas through the interstices or pores of the fabric by means of capillarity.

For protection of the skin against water, a material was
developed, consisting of a mixture of varnish, linseed, linseed oil, and
tin oxide, which served primarily to delay the penetration of mustard gas
to the skin, thus giving more time for evaporation to take place. This
ointment was known as Sag Paste, and was shipped to the Western Front in large quantities. The majority of the reports were quite favorable though of course the paste was only used at the front for a limited time. It was later found possible to improve this ointment greatly by sprinkling bleaching powder over the skin after the ointment had been applied, the bleaching powder serving to destroy the mustard gas.
We can now come back to some of the defense research work on absorbents, of which the most important is charcoal. It had been known for a long time that charcoal would condense in its pores or absorb certain gases, holding them very firmly. It was also known that certain charcoals were very effective in decolorizing sugar solutions for instance, but that was about all. It was known that in general so-called animal charcoal was the best for decolorizing sugar charcoals, that wood charcoals were the best for adsorbing gases, and that coke had very little adsorbing or decolorizing power; but nobody knew why and nobody could write a specification for charcoal.

It had been found by Hunter more than fifty years ago that cocoanut charcoal was the best for adsorbing gases and this was the charcoal usually used in scientific laboratories. In 1912 two Germans, Hempel and Water, claimed to have made a better charcoal than cocoanut charcoal by mixing a special animal charcoal to a stiff paste with ox-blood diluted with water and charring at 600°C. This was not very much in the way of information and even this was necessarily applicable.

The scientific work on adsorption had to do with the equilibrium relations of pure gases while the war problem was to remove mere traces of toxic gases from air in a very short interval of time. For instance, with a rapid stream of air through the canister, the modern charcoal will reduce 7000 p.p.m. of chloropirin to a negligible concentration in about 0.03 seconds. The magnitude of the task is more evident when we remember that the pre-war charcoal scarcely stopped chloropirin at all.

Some experiments by Professor Lemon at the University of Chicago had shown that the adsorbing powers of cocoanut charcoal could be varied...
a great deal by suitable treatments. Small amounts of air were taken up by the charcoal at low temperature and then the charcoal was heated to some specified temperature and as much gas as possible removed by pumping out the vessel. If this were done a number of times at 600°, the charcoal was improved a great deal. If its temperature was raised to about 800° the charcoal became worse. No satisfactory explanation for this phenomenon had been developed at the time the United States went into the war.

It was therefore necessary to develop a war charcoal without any real knowledge of how to do it and to develop it very quickly.
The first problem was to get a charcoal which would stop chlorine. After carbonizing all sorts of materials in all sorts of way, the National Carbon Co. succeeded in making a charcoal from red cedar which stood up very well against chlorine, lasting 150-200 minutes as then tested. In order to improve the charcoal still further, it was desirable to have some theory as to the way the charcoal acted. Everybody was agreed that fine pores were essential; but nothing was known in regard to the other factors. Some people said that the presence of certain hydrocarbons were essential. This was in line with the view that nitrogenous material is of the greatest value in charcoals that are used for decolorizing sugar solutions. Mr. Chaney of the National Carbon Co., took the opposite view that carbon had very high adsorbing power and that the important thing was to remove the hydrocarbons which he assumed still to be there after the carbonization. He believed that one great difference between different raw materials was the difference in the hydrocarbon residues, some of which were driven off more easily and more completely than others. To prove this some of the red cedar charcoal was heated in a bomb connected with a pump which drew air through the bomb. Although this charcoal had been carbonized at 800°C, various gases and vapors began to come off at 300°C and some of these vapors condensed when cooled to crystalline plates.

This not only proved the existence in the charcoal of compounds containing hydrogen but also showed that one way of removing the hydrocarbon film on the active carbon was to treat with an oxidizing
agent. A process of activation by oxidation in air was developed rapidly and the regular cedar charcoal which had lasted 150–200 minutes against chlorine then lasted 400–500 minutes.

If the true cause of the inactivity of various charcoals was the presence of adsorbed hydrocarbons, it was evident that two methods of activation were possible. The charcoal could be heated for a long time at a temperature high enough to decompose or drive off the hydrocarbon. It could also be oxidized either wet or dry. Both these general methods give some degree of activation. The first one was employed by the British and the French. It was abandoned in this country because it was found that high-temperature cracking of the hydrocarbons gave rise to a somewhat graphitic carbon which was not active.

One puzzling fact was encountered in regard to air oxidation. Samples of coconut shell charcoal had been made which were even better than the red cedar charcoal, but they did not respond to air oxidation under the conditions which had improved red cedar so much. This proved to be because the critical oxidation temperature of coconut charcoal was considerably higher than that of cedar charcoal. By increasing both the temperature and the time of oxidation, the coconut charcoal improved even more than the cedar charcoal, changing from a service time of 200–400 minutes against chlorine to one of 1500–1800 minutes, or up to thirty hours.

About this time it was discovered that the 180–200 minute cedar charcoal did not stop chlorpiorin at all, lasting two to three minutes at the most. Since chlorpiorin was then being used by the
Germans, all orders for red cedar charcoal were cancelled. Fortunately, it turned out that the activated cocoanut charcoal lasted 200 minutes against chlorpiorin which was a wonderful showing at the time, though such a charcoal would not be considered fit to use. The production of this charcoal on a commercial scale was turned over to Mr. (later Colonel) Dorsey of the Nela Park Laboratory at Cleveland, who started a plant at once at Astoria, Long Island, for the Gas Defense Service, later the Defense Production Division. The commercial development will be discussed in the chapter on the Development Division. The product as turned out at Astoria was called Dorsite. Before the Astoria plant was built, the National Carbon Company succeeded in making a cocoanut charcoal which lasted 400 minutes against chlorpiorin.

Chaney believed that only part of the activation was due to the removal of the hydrocarbon and that another function of the oxidation was to dig out channels through the carbon thereby increasing the active surface. If this is the case, different oxidizing agents might easily act differently, a weak oxidizing agent acting selectively, while a powerful one might burn the carbon away uniformly, or might enlarge existing pores instead of developing new ones. With this in mind a careful analysis of all possible methods of oxidation was made. All oxidation processes involving liquids were discarded in favor of oxidation by gases because the latter type of oxidation seemed cleaner and cheaper, and could be completed in a single operation, there being no need of washing, filtering, drying, etc. The following gaseous oxidizing agents seemed to be worth studying:

34.60
1. Air
2. Chlorine
3. Oxides of nitrogen
4. Steam at water gas temperatures
5. CO₂ at high temperatures
6. Sulphur Dioxide.

The first experiments were made with chlorine and were not encouraging because the results were practically the same as with air. It was thought that the reason for this was that the charcoal had to be heated in air afterward to drive out the chlorine and that any especially fine structure produced by the chlorine might be burned up by the air. To avoid this, superheated steam was passed over the charcoal to drive out the chlorine, the temperature being kept too low for the steam itself to react. The product improved steadily and reached a life of 800 minutes or four times the life of the first air-treated coconut charcoal. With rising temperature the product kept on improving until at 900°C it was found that treatment with steam alone was just as effective as the chlorine treatment. While working with steam at 900-1000°C is not the easiest thing in the world to do, it is a much simpler problem than working with chlorine at that temperature. A semi-commercial continuous process, steam-treating unit was then built, in which the charcoal was fed into the top and removed at the bottom. This furnace was electrically heated and yielded about 24 pounds of charcoal a day.

The results were so good that men were sent from the American
University Experiment Station to help in the work. By the time of
the signing of the armistice the detachment consisted of thirty
Chemical Warfare Service men. At Astoria, Mr. Dorsey at once started
large scale production, using a nichrome metal central tube instead
of a clay one and firing by flameless combustion instead of using
electrical heating.

The rapid development of the steam process was not due
merely to the fact that it gave the best charcoal. The air-treatment was much
cheaper and gave a product which was several times as efficient as
that then in use by the British and French. The great advantage of
the steam process was that it worked well with all kinds of charcoal.
With the air process it was not possible to make charcoal from other
sources which was as good as the air-treated charcoal, whereas inferior
material, when treated with steam gave charcoals as good as, or better
than, the best air-treated charcoal. This was very important because
not over 100 tons of raw coconut shells per day could be counted on.
Since this meant only eight tons of charcoal finished per day, it was
very necessary to find a substitute for coconut charcoal.

Arrangements were made by the Defense Production Division to
import cocoa nut from South America and Central America, and to collect
peach stones, etc., from all over the United States, and the National
Carbon Company started developing charcoals from anthracite coal,
biteuminous coal, lamp-black, wood, coconut fines, etc.

The first experiments were made with a special anthracite
coal which gave service times up to 560 minutes as against 360 for air-
treated coconut charcoal and 800-900 for steam-treated charcoal.
This product was called Batchite after Mr. H. D. Batchelor. When the Gas Defense Service tried to activate anthracite on a large scale in vertical gas retorts at Derby, Connecticut, the attempt was a failure. They carbonized at 900° and then turned on the steam with the result that the steam-treated coal had a slightly greater density than the untreated, which was wrong, and had a shiny appearance in parts with roughened deposits in the other parts. When the hydrocarbons are decomposed at high temperatures, the resulting carbon is somewhat graphite, is itself inactive, is not readily oxidized, and impairs or prevents the activation of the normal carbon upon which it is deposited. This discovery made it possible to treat anthracite successfully. The conditions must be such as to minimize high-temperature cracking, to carry off or oxidize the hydrocarbons as fast as formed, and especially to prevent the gases from cooler portions of the retort coming in contact with carbon at a much higher temperature. With these facts in mind, Mr. Batchelor of the National Carbon Company was able to build a plant at Springfield which produced 10 tons a day of 150-300 minute charcoal from raw anthracite. This was one-third of the total production at that time and was mixed with the nat charcoal made at Astoria, thereby preventing an absolute shortage of carbon-filling material in October, 1918.

The effects of high-temperature cracking, or gas treating as Chaney calls it, explain why it is impossible to activate retort coal satisfactorily and also why samples of lighting carbon made with lamp-black flour might activate well if taken from one part of the furnace.
and not at all if taken from another part of the same furnace. It is all a question of the temperatures in the different parts of the furnace.

It was next shown that the coconut charcoal fines resulting from grinding and screening losses with the coconut charcoal, and amounting to 50% of the product, could be ground, mixed with binder, and baked like ordinary carbon products. By avoiding gas-treating in the bake, the resulting charcoal is nearly as good as that from the original shell. A recovery plant for treating the coconut fines was built at Astoria. The product was called "Coalite."

The great advantage of coconut shell as a source of charcoal is that it is very dense and consequently it is possible to convert it into a mass having a large number of fine pores, whereas, less dense wood, like cedar, will necessarily give more larger pores, which are of relatively little value. The coconut charcoal is also pretty resistant to oxidation which seems to make selective oxidation a more simple matter. By briquetting different woods, it is possible to make charcoal from them which is nearly equal to that from coconut shell.

By heating lamp black with sulphur and briquetting, it was possible to make a charcoal having approximately the same service time as coconut charcoal. At the Cornell Laboratory a charcoal was made by carbonizing carbon black with soft pitch, which gave the equivalent of 400 minutes against carbon-pieces before it had been steam-treated. This looked so good that the plans were drawn for making a thousand pounds or more of this product at Newington so as to give it a thorough test. This was not done on account of the stopping of all research work. The possible advantage of this product was the more uniform distribution of binder.
The National Carbon Company studied the production of charcoal from bituminous coal and made a 600-minute charcoal from this material. Instead of steam-treating anthracite coal direct, the National Carbon Company also pulverized it, mixed it with binder, and baked it into rods which were then ground and activated with steam. The resulting material, which was known as Carbonite, had somewhat less activity than the lamp-black mixes but was very much cheaper.

A plant was built to bake 40 tons a day of this material, which would yield 10 tons a day of active carbon after allowing for grinding losses and steam treatment. The plant was guaranteed to furnish an absorbent having a life of 600 minutes against chloropicrin (40 minutes on the accelerated test).

After the armistice was signed, Mr. Chaney took up the question of how the Germans made their charcoal. The German charcoal was made from a coniferous wood and was reported to be as good as ours, in spite of the fact that they were using inferior materials. Inside of a month Mr. Chaney had found out how the German charcoal was made, had duplicated their material, and had shown that it was nothing like as good as our charcoal. The Germans impregnated the wood with zinc chloride, carbonized at red heat, and washed out most of the zinc chloride. When this zinc chloride was found in the German charcoal, it was assumed that it had been added after the charcoal had been made. It was therefore dissolved out with hydrochloric acid, thereby improving the charcoal against chloropicrin. The German charcoal was then tested as it stood, including the fines, against American charcoal 8-14 mesh. The most
serious error, however, was in testing only against a high concentration of chloropicrin. The German charcoal contains relatively coarse pores which condense gases at high concentrations very well but which do not adsorb gases strongly at low concentrations. The result was that the German charcoal was rated as being four or five times as good as it really was.

The general theory of adsorbent or "active" carbon has been outlined by Dr. Chaney something as follows:

1. **Active carbon** is a form of amorphous carbon deposited at relatively low temperatures (below 6000) by chemical or thermal decomposition of carbon compounds. It is substantially free from adsorbed hydrocarbons and from the inactive forms of carbons deposited by thermal decomposition of hydrocarbons at relatively high temperatures, as in so-called "gas-treating".

2. **Primary carbon** is active carbon plus adsorbed hydrocarbons. Destructive distillation of hydrocarbons at low temperatures, as in the charring of woods, burning of oils to lamp-black, and distillation of coal, results, in general, in the deposition of active carbon, which adsorb a certain amount of the hydrocarbons, thus forming what is called primary carbon. The adsorbed hydrocarbons are stabilized thereby so that they resist cracking and volatilization at very much higher temperatures than they would if not adsorbed.

3. **Activation** consists in removing the adsorbed hydrocarbons from the active carbon and in securing the proper porosity.
The removal of the hydrocarbons is a difficult matter owing to their being stabilized by adsorption. Increasing the surface of the active charcoal means increasing the adsorption. Increasing the porosity and, therefore decreasing the density is of benefit only so long as it increases the surface. A first-class charcoal, may have an apparent density of 0.4 while the true density of the carbon is over 1.8.

The specific oxidizers found of most value were air at 350-450°C and steam or CO₂ at 800-1000°C. The success of the method lies in the fact that the hydrocarbons are more susceptible to oxidation than active carbon and may therefore be removed with only the loss of carbon essential to proper porosity of the granular absorbent.

4. Gas treating is the decomposition of hydrocarbons at high temperatures, in general above 600°C. The product is inert, non-absorbent, and resistant to oxidation. When deposited in the pores and on the surface of active carbon, it renders the latter inactive, and partially or wholly incapable of re-activation, depending on the extent of the gas treatment. The inferiority of the British and French charcoal is due to the inactive carbon formed as a result of prolonged, high-temperature distillation.

The essential feature in the manufacture of active carbon is to avoid gas-treating both in the preliminary carbonization and in the subsequent processes of removing hydrocarbons from active carbon. While retort carbon, ordinary bituminous coals, and various manufactured carbons are inactive and incapable of activation, this is not a necessary characteristic. Owing to the method of coking or baking, such products have received a severe gas-treating. When this is avoided, active carbon may be manufactured from any carbonaceous material.

29.57
The manufacture of a satisfactory soda-lime was a difficult problem. The first soda-lime had too much caustic soda and was too soft and too deliquescent. Captain (afterwards Major) Dudley brought over the formula for the British soda-lime and this was taken as a starting point. It was necessary to strike a balance between a number of desirable qualities: absorptive activity, capacity, hardness, resistance to abrasion, chemical stability, low breathing resistance, etc. The development of a satisfactory soda-lime involved, as a preliminary, a careful study of the raw materials and the development of standard specifications and of methods for testing. The method of manufacture is in some respects more important than the exact composition of the product. Details therefore had to be worked out for mixing, calcining, drying, and grinding. The method of manufacture was also improved by devising a process for spraying sodium percarbonate upon the granules after they were dried. The final product consisted essentially of a mixture of lime, cement, kieselguhr, sodium permanganate and sodium hydroxide, the last two being present in very small proportions. The caustic soda gives the activity, the lime the capacity, the kieselguhr the porosity, and the cement the hardness, while the permanganate is added to take off certain oxidizable gases which the Germans might have used but did not. The final product was better than the soda-lime made by any of the other belligerent countries.

A great many experiments were also made to find better binding agents, better activating agents, or special reagents which might improve the absorbent with reference to some particular gas. It was quite easy to produce better results in any one direction; but these were usually counterbalanced by a corresponding disadvantage. If the hardness was increased,
for instance, the absorptive activity always decreased and vice-versa. The
following materials seemed fairly promising however: copper oxide, which
improved the soda-lime greatly against hydrocyanic acid; ferric hydroxide,
which helped against cyanogen chloride; and finely ground charcoal, which
tended to activate the soda-lime against all gases.

The successful production of a satisfactory soda-lime for the
Army led to a request by the Navy for the development of a more satisfactory
soda-lime to absorb carbon dioxide in submarines. A material was developed
which gave distinctly better absorptive efficiency and enormously better
mechanical properties than any CO₂ absorbent previously used by the Navy
and permitted submergence for eight times the periods which had been possible
previously. A soda-lime was also developed for the Navy to be used in a
self-contained oxygen respirator apparatus for the absorption of CO₂ from
the lungs.

Since the absorbents are placed in the canister at least two or
three months before they are used and may be in use for several months
before they are replaced, the question of deterioration is a serious one.
The prevention of deterioration of soda-lime was found to be largely a
question of keeping the various raw materials as pure as possible. The
chief difficulty is the decomposition of the permanganate, which can be pre-
vented if the alkali content is kept low and all the materials are free from
reducing agents or soluble salts. A low alkali content also prevents
deterioration due to deliquescence of the absorbent.

The only method by which sodium permanganate was made before the
war gave a product which was entirely unsatisfactory for use in soda-lime be-
cause not sufficiently pure. New methods for oxidation and evaporation were
therefore developed. An electrolytic method of preparing sodium permanganate was developed. The work was begun at the Johns Hopkins Laboratory, carried further at the Washington Laboratory, put on a large-scale laboratory basis at Columbia University, and tried out on a semi-plant scale at the works of the National Electrolytic Company, Niagara Falls.

The experiments were entirely satisfactory and a much purer permanganate was obtained than by any other method. It was estimated that the cost by the electrolytic process would only be about one-third that by the chemical process.

As soon as the soda-lime problem was fairly well in hand, investigation was begun of a great number of other types of possible absorbing materials. It was soon found that the most promising of all types of absorbents were impregnated charcoals, since they retained the absorptive action of the charcoal and yet could react chemically with certain gases, depending upon the nature of the impregnating agent. Charcoals had been impregnated with practically all the metallic hydroxides, with a great many inorganic salts, with organic compounds, with oxidizing agents, etc. The best material was a copper-impregnated charcoal to which the name of Ehtelerite was given. It was made by introducing copper sulphate into charcoal and reducing with finely divided iron. This absorbent is a consistent all-round improvement over the corresponding impregnated charcoal and has to have been put into large-scale production by the Gas Defence Division. The material gave an average increase in service time of 500% against urine, 100% against hydrocyanic acid, 60% against phosgene and other acid gases, and 10 - 40% against other gases.

Another very promising new type of absorbent was the so-called iron gel or ferric hydroxide absorbent which was made by precipitating ferric hydroxide in a very finely divided form, washing, drying very slowly, and grinding into granular form. All these operations must be carried out under carefully
defined conditions. The final product is a better absorbent for all
gases than charcoal in a dry atmosphere; but it showed a great ten-
dency to take up moisture rather than the poison gases. It seems
possible that the ideal mixture for a canister may consist of 70\%
Whetlerite and 30\% iron gel.

Another very interesting absorbent was the silicogel
developed at the Johns Hopkins University! This had great absorptive
power especially against high concentrations, but did not hold the
gases sufficiently when they were present in low concentration.

Special determinations on the effect of the size of the
particles of the different standard absorbents on their efficiency
against different gases showed that the increase in efficiency with
decreasing size of grain is very great with soda-lime, less with
Whetlerite, and least of all with charcoal. Experiments were also
made to determine the best combination of size of granules, depth of
layer, and area of cross-section to give the maximum efficiency and
the lowest pressure drop through the canister. These experiments led
to a radical change in the design of the 1919 canister, giving it a
very large cross-section area, a shallower layer of absorbent, and fine-
meshed particles.
The Navy wished a special absorbent to stop carbon monoxide and Col. Lamb's Section succeeded in developing two which were satisfactory. The first consisted essentially of iodine pentoxide and fuming sulphuric acid mixed with pumice. This reacts with the carbon monoxide oxidizing it to carbon dioxide. Some experiments along this line were carried out by the French; but they did not carry the idea through to a successful finish. The second and better absorbent consisted of a mixture of suitably prepared oxides which act catalytically under certain conditions and cause the carbon monoxide to react with the oxygen of the air. This is the one that has been put into large scale production. Since there are color changes connected with the iodine pentoxide reaction, it has been possible to develop this so as to serve as a detector for carbon monoxide, sensitive to 0.001% CO.

The Navy also wished an ammonia absorbent and a successful one was found in Napranite which consists of pumice impregnated with its weight of CuSO$_4$.H$_2$O. With 44 cubic inches in the standard Army canister, men at rest have complete protection against 3% ammonia for four hours and against 5% ammonia for 2.5 hours. With men doing severe exercise the life against 5% ammonia is over 25 minutes. At a rate of flow of air of 85 liters per minute, the resistance is less than 50 mm. of water.
The development of the canister and of the face-piece of the mask was connected so intimately with the production that it seems better to take up these two general questions in the chapter devoted to the Defense Production Division in spite of the fact that a great deal of work was done by the Research Division. Certain points in regard to the canisters and the eye-pieces cannot be discussed here.
Plate XIII.
A DUPLEX MACHINE FOR STUDYING THE COURSE OF GASES THROUGH CANISTERS.

(Side View)
A DUPLEX MACHINE FOR STUDYING THE COURSE OF
GASES THROUGH C a b i n e t s

(End View)
The general development of the charcoal has been along two
general lines: decrease in volume of better absorbers were obtained and
reduction of resistance to broaching. The absorbers used in filling the
first charcoal consisted of wood charcoal and a soda-lime containing a
high percentage of carbonated soda and colored green and turning color in
stead of pink by sodium pyrophosphate, as had been the case. The
gases that were considered at the time were carbonic, paracetic and acetic-
salicinic acids. Early experiments showed that with 50% sodium carbonate of absor-
bent in the mixture, the parts of soda-lime to parts of charcoal were as
low as one could cut down the amount of soda-lime, with a consequent
protection. This ratio was adopted, since it was desired to keep the
amount of soda-lime at a minimum. The charcoal in use in June 1917,
did not show saliphilic and the soda-lime as a distinctly inferior product.
The wood charcoal was reduced by oxygen charcoal as soon as possible and
the quality of the latter was improved continuously. The improvement in the
soda-lime was equally marked and no change in the relative amounts of soda-
lime and charcoal was ever made. On the other hand it has been found that
better results were obtained by mixing the charcoal and soda-lime, rather
than in separate layers. It was also easier to mix the charcoal
with the mixture than with the wood charcoal separately, so that change was
adopted.

There is always the tendency for the incoming air not to pass uni-
formly through the mixture. Special experiments showed that this choking,
as it is called, could be reduced to a minimum by introducing a wire screen at
the bottom of the charcoal, this serving to spread the air more uniformly.
As might be expected, the choking up the inside of the charcoal is less
the lower the rate of flow and in less for intermittent flow than for
The tendency to channel varies with the packing of the canister and the experiments on channelling led to an improved method of packing. The original method of packing experimental canisters had been to pour the absorbents into the canisters and bump them down with a wooden block. This method packed the center harder than the sides causing the gas to channel up the sides. The improved method consisted in filling the canister with absorbents and then inserting the top screen and springs. The canister with the spring pressure applied, is then jolted on a machine designed to give the canister a one-inch drop one hundred and fifty times per minute. This continues the absorbents evenly and decreases irregular channelling to a great extent. In the latest type of canister, using a central shaping tube, it is absolutely necessary to jolt the canister with the spring pressure applied in order to stop channelling.

When strontium chloride began to be used as a source of strontium, it was necessary to place in the canister the so-called plate, consisting of two or three leaves each, one on either end of the plate. This serves effectively, since it has not been proved protactinically that enriched with one part one-third of the strontium from and the remaining other two-thirds of the plate, though it is the trend for the upper portions of the canister to have a cold place, is very much colder than the apparent amount of strontium in the position of the plate in the canister.

The canisters are generally utilized with the protection of a storage vessel which is filled with a water pressure drop. The standardized shape adopted was 14 for all canisters, 3-4 by channel-line. A stainless steel is used which can be handled by the machine with ease and the canisters are then ground down to a smooth surface within the machine. The canisters are placed on the ground and the machine is operated by a small gasoline motor and the frame towelled in in the main position of the machine.

E. W. D. 85
With falling temperature the life of the canister increases against phosgene, hydrocyanic acid, and cyanogen chloride while there is practically no change with the temperature when the canister is tested against chloropicrin. The differences may be quite marked. Thus a charcoal which lasts 17.5 minutes against phosgene at 40°C will stand up for 54 minutes at -13°C. The difference is not so great with soda-lime, so that the life of a canister against phosgene at -13°C is only about 70% greater than at 40°C instead of about 200% greater as would be the case if the filling were entirely charcoal. With hydrocyanic acid the variation with the temperature is very marked for the soda-lime and relatively little for the charcoal. Thus the life of the canister is more than doubled when changing from 40°C to -13°C while the increased life with charcoal alone is only about 20% against cyanogen chloride the life at the lower temperature in six times that it is at the higher temperature.

With increasing humidity the life of the canister against phosgene increases because the phosgene is absorbed more rapidly, while with chloropicrin the life is less because the water cuts down the absorption of chloropicrin. If a canister is stored for six months, there is an increase in efficiency against phosgene and a decrease against chloropicrin. This is due in part to a transfer of moisture from the soda-lime to the chloropicrin, which maximizes the life of the canister with relatively low concentration of the gas, though it is more accurate to use a formula:

\[ \frac{C}{T} = \frac{1}{60} \left( \frac{1}{2} \right) \]

The time in minutes to the breaking point, C is concentration in parts per million and T is the time in days at given relative humidity, varying between 0.9 and 1.5.
One great difficulty with the mask is the dimming of the eye-pieces. The French met this to some extent by using eye-pieces made of \textit{cellophane}, a hydrated cellulose acetate. This material takes up water but lets it diffuse through. The eye-piece remains non-dimming provided the condensation of moisture is not too rapid. The Germans used a gelatin-coated eye-piece which could take up a certain amount of water before dimming, after which another eye-piece must be substituted. The British used a glass eye-piece upon which they rubbed and anti-dimming mixture called glasso, which kept the water from forming in drops on the surface. In the French Tissot Mask the air which comes through the canister passes over the eye-pieces and cuts down the dimming very much.

The Research Laboratory of the Eastman Kodak Company succeeded in producing a gelatine which was distinctly superior to that manufactured by the Germans. The fatal defects of this material as compared with glass are its poorer initial optical qualities, its softness when moist making it liable to scratching, and the tendency of the eye-piece discs to warp during the absorption of moisture or during drying.

The French \textit{cellophane} eye-piece is essentially viscose, so the actual making of the material is easy, but the difficulty is to obtain discs in a sufficiently transparent and optically satisfactory form. The Viscose Company succeeded in obtaining material equal to that made by the French and in thicker sheets, but it was still distinctly inferior to the hydrated cellulose obtained in other
ways. Denitrated cellulose nitrate gives a still better product but it proved impossible to denitrate completely sheets as thick as were desired. This work was done at the Delta Laboratory of the DuPont Company at Arlington, N. J. Cellulose acetate proved to be the best substance from which to prepare transparent cellulose.

The Chemical Products Company of Boston found that with alcoholic or aqueous ammonia it was possible to hydrolyze sheets containing very little softener and which were thicker than those to be obtained from any of the other materials. The product was distinctly superior to the French 'cellophane' both in hygroscopic and optical properties. Like the other forms of hydrated cellulose it is subject to cockling and shrinking on repeated drying, so that it frequently pulls out of its holder. Though its optical properties are surprisingly good, they are inferior to glass and the material is easily scratched when moist.

One other material studied was a hygroscopic glass. Glass lenses were actually secured which did not dim readily; but the material had not the required degree of permanency.

While these experiments were being carried on, work was also being done to improve on the anti-dimming materials used by the British. The material finally adopted was made by heating a mixture consisting of 100 parts of 85% Turkey rod oil, 15 parts of caustic soda, 5 parts of syrupy water glass, and 5 parts of paraffin, then drying the mixture to about 10% water content. This material was manufactured on a large scale by Colgate and Company in the form
of small round sticks. Ten million of these anti-dimming sticks had been contracted for by the Gas Defense Production Division.

With the introduction of the Tissot type of mask, the problem changed somewhat. With this type there is much less dimming, because the inhaled air passes over the eye-pieces. Dimming does take place intermittently when the man exhales and this is of course more marked the colder the weather. Experiments showed that the anti-dimming stick developed for the S. B. R. mask was not absolutely satisfactory for the Tissot type of mask, but a slight change in the formula met this difficulty and four million of the new type of anti-dimming sticks were ordered by the Gas Defense Production Division.

It seemed possible to eliminate all dimming with the Tissot type of mask by changing the design somewhat. All the forms developed for this purpose made use of a sponge-rubber dam fitting tightly across the face over the nose and cheeks thus dividing the air space within the mask into two chambers, one for the eyes and the other for the mouth and nose. In what seemed to be the best of the four types the air from the eye chamber passes through a single, large, low-resistance valve to the breathing chamber. It is not practicable to place this valve in the rubber dam and it must in general be included in an external tube. With a valve placed at this point, the corresponding valve on the base of the canister can be omitted. This arrangement prevents the backward flow of moist air into the eye-chamber and yet does not involve any serious effects in mask resistance even at high rates of breathing.
Masks of this type have stood up most successfully under very rigid tests. The one essential is that they shall fit well. On the other hand the question of fit can be determined easily by the soldier himself. If the eye-pieces dim, the mask does not fit and should be changed.

In connection with this work experiments were also carried out to determine the effect on the soldier of varying visibility. Eye-piece materials which are optically inferior when the illumination is good become relatively very much more inferior with poor illumination. While good definition is important in bright light, it becomes extremely important in poor light. For military use, eye-pieces should therefore have the best possible defining power.

The effect of decreasing visibility on the accuracy of rifle sighting was tested thoroughly with good illumination. No serious impairment occurs until the definition is reduced to about 30% of normal, the decrease in visibility evidently being counteracted largely by an increase in the effort of attention. Different results would probably be obtained with tired men. Discrimination in a limited time between a gray object and a white background was also tested with varying illumination and it was found that there was not much impairment in the power of discrimination until the defining power dropped below 50%. With poor light the difference became noticeable much sooner.
The glass used in the mask eye-pieces, so-called triplex glass, consisting of two layers of glass cemented together by a layer of pyralin, which is a special celluloid. For a long time the output of the Super-Glass Company was only 50000 eye-pieces per day with 50% rejections. These rejections were due largely to "hot-core" (in which case the glass separates from the pyralin layer), cracks, "squeeze-outs", and dry spots. Most of these difficulties were caused by insufficient curing of the pyralin and by improper methods of cutting and pressing the layers used in the eyepieces.

The Research Division was able to suggest improvements which brought the production up to as high as 50000 lenses per day with 10% rejections.
A very careful study of the theory of smoke was made. The concentration of the smoke was determined by precipitation in a modified Cottrell apparatus consisting of a central wire cathode surrounded by a cylindrical aluminum foil anode about 1/1000 inch in thickness. A 15000 volt rectified direct current was used and complete precipitation was obtained with fairly concentrated samples of smoke even when drawn through the apparatus of about five liters per minute. The aluminum foil and adorning smoke were then weighed. Microscopic examination showed whether the smoke particles were liquid or solid. The size of the particles in a smoke can be determined ultra-microscopically with fair accuracy by measuring the velocity of a charged particle in an electric field of measured intensity, photographing the path of the particle while the direction of the electric field is reversed regularly by a rotating commutator whose speed is known accurately. When the convection due to the source of light is perpendicular to this motion, a zigzag line is obtained. Since about one-third of the smoke particles are charged electrically, photographs of these oscillations show simultaneously the behavior of a large number of particles, thus simplifying the study of size distribution. For the more rapid study of smokes an instrument called the Tyndall meter was devised which measured the brightness of the Tyndall beam set up in the smoke to be examined. For low concentrations of smoke the brightness of the beam increases with the concentration and the degree of viscosity of the smoke material, so that if either factor remains practically constant the readings give a measure of the variation of the other.

Toxic smokes are prepared by condensation, disintegration, or some combination of the two. The condensation or thermal method consists essentially in the volatilization of a substance having a sufficiently low vapor pressure, followed by a recondensation of the vapor when the temperature has fallen
Ultramicrograph showing zig zag path of charged smoke particles subjected to a rapidly reversing electrical field.
TYNDALL METER AND AUXILIARY APPARATUS

PLATE V
Curve showing relation of Illuminometer readings to concentration of silica solutions.

reading with water 0.004
Tyndall Meter Reading
Foot Candles

Relation of the Dilute Concentrations of \( \text{NH}_4\text{Cl} \) Smoke to Tyndall Meter Reading.
Relation of Concentration of NaCl Smoke to Tyndall Meter Reading.
sufficiently. The smoke consists of relatively large particles if the rate of condensation is low as when vaporization takes place in an open container from which the condensing vapors pass slowly into the air without being diluted sufficiently. Each particle remains an appreciable time in contact with saturated vapor and consequently increases very rapidly in size. On the other hand relatively small particles will be formed if the vapor is swept away by a current of hot gases under pressure because it is then diluted before super saturation occurs. The possibility of the very small particles coalescing is also less at high dilutions.

In the disintegration or explosive method we have the partial mechanical shattering of the material owing to the shock of the explosion and a partial vaporization of the shattered material due to the heat of explosion. The full violence of the mechanical shock is not utilized since the smoke material does not possess sufficient inertia to receive the full force of the shock; and the effectiveness of the heat of the explosion is reduced greatly owing to the very brief contact of a particle in the heated zone. Consequently the explosive method does not give so many finely divided smoke particles as does the thermal method. Starting with a powder of about 0.1 mm in diameter, the explosion will shatter these more or less completely into particles about 0.001 mm. The heat produced will vaporize some of these particles which will then precipitate in a finer form; but the percentage having diameters 1 = 0.01 μ is very much less than when the thermal method is used. With the substances studied, very few particles are obtained having diameters less than 0.01 μ, apparently because the very small particles are too instable.
Some substances give good smokes by explosion while others do not. The important factors are the vapor pressure, the latent heat of vaporization, the melting-point, and the strength of the crystalline forces. The vapor pressure should have a medium value at ordinary temperatures; if it is too high, recondensation does not take place sufficiently rapidly and if it is too low, a very fine state of subdivision cannot be obtained on explosion and the temperature must be raised too high in the case of the thermal method. This objection is serious with combustible smokes and does not apply of course to phosphorus pentoxide for instance.

If the latent heat of vaporization is low, vaporization by the heat of explosion is facilitated and a cooling of the superheated vapor is obtained more easily. If the melting point is low, the substance will melt on slight heating; and the cohesive force of a liquid is generally less than that of the corresponding solid. With any given solid, disintegration will take place more readily the lower the cohesive force and the meltility. If the smoke material is a solid it should be very actively dry and should be ground very fine. As a rule liquids, semi-solids, and relatively low-melting solids give much better smokes than high-melting solids.

Particles of a diameter 1 - 0.01 μ, such as are found in good, relatively permanent smokes, are small compared with the capillary diameter of the smoke filter. Consequently, the filter does not act primarily like a sieve. The actual mechanism of the filtering consists in the sticking of smoke particles to the wall of the capillary when they are entrapped in contact with it. Particles are brought in contact with the wall by Brownian movements, by direct collision when the current of air carrying the particles turns a sudden corner, and by gravity and settling.
With a given filter the percentage penetration of smoke decreases during a run partly because of clogging of the filter and partly because of coagulation of the smoke with time. For the same sized particles the percentage penetration is practically independent of the concentration. At very low rates of flow the penetration is practically zero. With increased rate of flow there is increasing penetration, beyond which the percentage penetration varies very little with the rate of flow. The shape and position of the penetration curve depends of course on the smoke and on the filter.

This study of the theory of smoke production made it possible to devise methods of using smoke which would undoubtedly have had great military value had it not been for the signing of the armistice. Large-scale production of the new devices was already under way.

A study was also made of the possibility of rendering the German masks valueless by coating the charcoal with a material which would prevent its absorbent action, or by clogging the smoke filter thus making breathing through it difficult. Since part of the improvement in the charcoal consists in removing hydrocarbons, experiments were made with kerosene, benzene, etc. testing the charcoal against ammonia. It was found that exceedingly large quantities of these materials were necessary in order to impair the absorbing power of the charcoal appreciably and consequently this method was discarded.

It would apparently be feasible to clog the German smoke filter with titanium chloride smoke, but smoke penetrates the German mask so readily that an attack with these seems more promising than one with titanium chloride.
TOXIC SMOKE CLOUD FROM 500 D.M. CANDLES (Second Test)

(Arrangement of Candles similar to that in First Test)

The cloud drifted over the garrison nearly three miles from the origin causing unprotected men severe burning in the throat and lungs, accompanied by coughing and in some cases vomiting. The smoke was detectable to an irritating degree in Lakehurst, N.J. which is about 4 1/2 miles from the origin of the cloud.

Test at Lakehurst Proving Grounds.
Dec. 12, 1918.
TOXIC SMOKE CLOUD FROM 500 D.M.I. CANDLES (First Test)

The candles were placed in 5 parallel rows which were 2 yards apart, each row containing 100 candles on a 100 yard front. The rows of candles were ignited in succession, the total time of active smoke emission being 23 minutes.

Practically all German and U.S. M. Canisters were penetrated unbearably to a distance of 1600 yards. Logan Feltz slightly penetrated up to 1000 yards.

Lakehurst, N.J. Proving Grounds
Dec. 12, 1918.
VIEW FROM BEHIND ORIGIN AFTER IGNITION OF
THIRD ROW OF CANDLES.
PENETRATION APPARATUS USED TO TEST
THE SMOKE FROM TOXIC CANDLES.

PLATE LXXXI
PENETRATION APPARATUS IN ACTION
all the filters devised for stopping smoke introduce a very high resistance to breathing. The Defence Chemical Research Section therefore tackled the problem of electrical precipitation. They succeeded in developing a portable electrical precipitation apparatus of the Cottrell type, weighing about four to five pounds which removed smokes completely and introduced only an immeasurable resistance. While the cost and complication of the special apparatus would undoubtedly prevent the general use of such an instrument on the battlefield, it might be of considerable value in special cases and might well be used for a variety of industrial purposes. It was found, for instance, that the electrical precipitator removes bacteria completely from the inhaled air.
MEMORANDUM THRU Director, Edgewood Chemical Biological Center (ECBC), (RDCB-D, Mr. Joseph L. Corriveau), 5183 Blackhawk Road, Aberdeen Proving Ground, MD 21010-5424

FOR Office of the Chief Counsel, US Army Research, Development and Engineering Command (RDECOM), (AMSRD-CCF/Ms. Kelly Knapp), 3071 Aberdeen Boulevard, Aberdeen Proving Ground, MD 21005-5424


1. The purpose of this memorandum is to recommend the release of information in regard to request to RDECOM FOIA Requests FA-14-0054.

2. ECBC received the request from Ms. Kelly Knapp, the RDECOM FOIA Officer. The request originated from gathering information on the Chemical Warfare Service.

3. The following documents were reviewed by Subject Matter Experts within ECBC:

   a. History of Research at Yale University, dated 20 Nov 1918, 11 pages.


   c. A Historical Sketch of Edgewood Arsenal, by Lt. William McPherson; AD 498494; date unknown, 20 pages.

   d. The Diary of Jet Parker; C390D1; dated Sep - Dec 1918, 26 pages.

RDCB-DPS-RS

4. ECBC has determined that all of the reviewed documents are suitable for release, however, all documents must have the classification/distribution changed through the Defense Technical Information Center prior to any release.

5. The point of contact is Mr. Ronald L. Stafford, ECBC Security Manager, (410) 436-1999 or ronald.l.stafford.civ@mail.mil.

[Signature]
Ronal L. STAFFORD
Security Manager