

Section I

LABORATORY TUTORIAL AND REFERENCE GUIDE

Chapter 1: Laboratory tutorial on techniques and procedures

Introduction

Before reading the procedures in this book, you should take a quick lesson in laboratory techniques to better help you understand the procedures discussed in this book. Many of the procedures in this book include vacuum distillation, which will be discussed in detail later on in the chapter. Unlike the Preparatory Manual of Explosives, this book has much less extractions and recrystallization to deal with; nonetheless, these two subjects will be discussed in the same manner as in The Preparatory Manual of Explosives.

First of all, laboratory safety is crucial, especially when carrying out procedures on chemical warfare. It should be noted that the procedures discussed in this book are very dangerous, and should only be carried out by qualified and trained personnel only. The riot control, disabling, and irritating substance can be prepared without too much safety precaution, but in no way should the blood agents, blister agents, or nerve agents be prepared unless the proper safety precautions are used. Furthermore, let it be understood that the preparation of chemical warfare agents (other than the riot control, disabling, and irritant agents) is illegal, and possession by unlicensed persons is a federal offense; nerve agents are controlled substances.

This book is intended to educate people about the chemistry and laboratory techniques involved in the preparation of chemical warfare agents. It should be understood that this book represents over 100 years of chemical warfare development, and is intended for educational purposes only. Police officers, other civil authorities, and military personnel can use this book for training purposes. Inventors and researchers may find this book an invaluable tool for their own research and development programs. **Note: Do not attempt to prepare the compounds detailed in this book if you are not of the scientific community.**

Lab safety

Lab safety is the first step in proper laboratory techniques. For each chemical procedure, read directions carefully, and know precisely what you need to do, before you actually do it. After reading the procedure think about the procedure, and know the hazards associated with it. Know the chemicals used in the procedure and how to properly handle them. Do not attempt to alter the procedure or change chemicals. The best safety is to prevent accidents before they happen.

Carry out all procedures involving riot control agents, disabling agents, and irritant agents using proper ventilation. Fume hoods work in most cases, but not all. Even in well-ventilated fume hoods, irritant agents can expand outward contaminating the entire lab. Vapors can travel long distances and cover large areas despite well ventilation.

Under any circumstance, eye protection should be used at all times. Eye protection should include eye goggles that completely seal the eyes; glasses are not proper eye protection.

Blister agents should be prepared in properly ventilated areas, and the preparer should wear a certified full-face gas mask. Nitrile gloves, and proper chemical suit should also be worn. The fume hood used should have maximum flow ventilation, with suction venting coming from multiple areas of the hood. In any case, it is better to use clean boxes, containing a positive nitrogen atmosphere.

Blood agents and nerve agents should definitely be prepared in clean boxes under all circumstances. Nerve agents and blood agents can spread over wide areas, even under high ventilation conditions. Preparers should have full body chemical suits and full-face gas masks within reach. With any preparation, electronic monitoring systems should be used to immediately alert personnel to the slightest presence of blood agent or nerve agent.

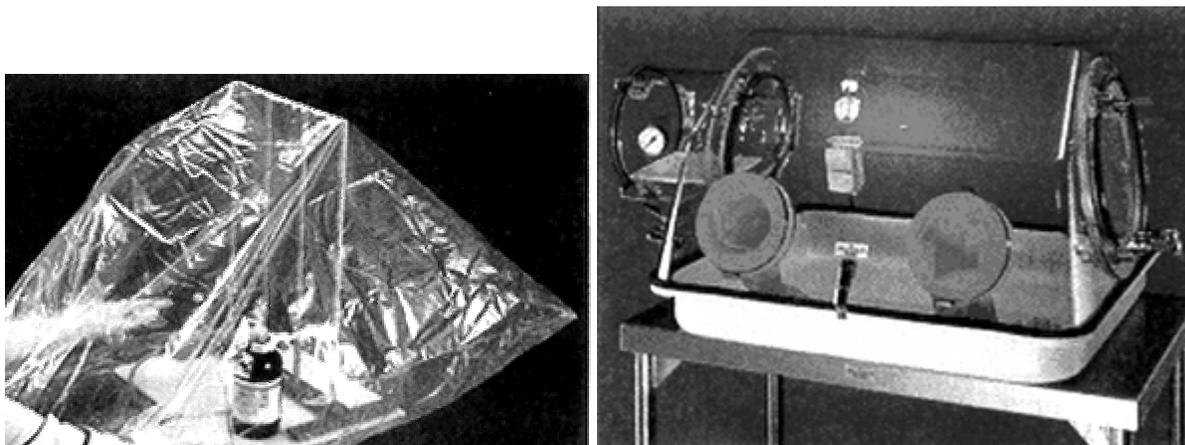


Figure 001. Left: A common laboratory “two-handed” atmosphere bag. Right: A classic laboratory clean box (other wise known as a “nitrogen dry box”).

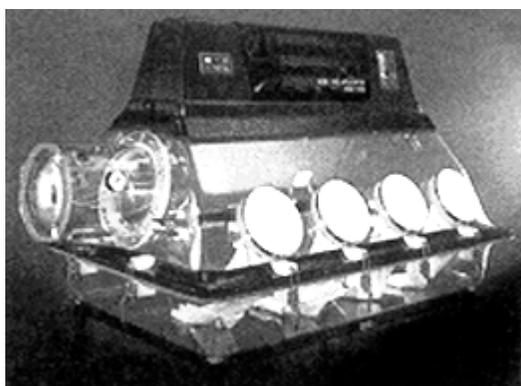


Figure 002. Another common clean box with electronic monitoring systems.

For general handling of chemicals (not involving any warfare agent) including common solvents, reagents, and intermediates, the following checkpoints should be observed:

- 1) Always remember to wear safety goggles at all times. Clothing and equipment can be replaced, but your eyes can't. Contact lenses or glasses are not a substitute for safety goggles. If you get chemicals in your eyes (liquid, gas, or vapor) immediately flush with large amounts of water.
- 2) Immediately wash off any chemical you happen to spill on yourself. Most chemicals are dangerous only if they linger, so take action at once. Concentrated sulfuric acid is not very harmful if washed off immediately, and most acids do little or no skin damage if they are immediately washed off with water.
- 3) In case of an accident such as a fire, save yourself first. Keep fire extinguishers in arms reach, and have an adequate water source within reach. For acid spills, simple baking soda can be used to neutralize it.
- 4) Avoid open flames in a laboratory setting, and do not smoke in the lab. In the event of a fire, calmly but quickly move away from the burning area. Fight the fire only if you are confident the fire can be extinguished.
- 5) Do not eat or drink food products while in the lab. Food and drink can become contaminated by accident, and never use laboratory glassware for eating or drinking.
- 6) Never taste chemicals, and never smell chemicals by sticking your nose right up to the container. Smell chemicals by wafting the vapors with your hand to your nose. Many accidents have occurred when fingers were contaminated in the laboratory and then later used to rub eyes or for eating snacks. Remember to wear gloves at all times. Latex gloves work for most cases, but in some cases nitrile gloves are recommended. Especially when handling strong acids, or chlorinated solvents. If bare handed, wash hands after touching chemicals and/or their storage bottles.
- 7) Breathing or handling small amounts of noxious substances does not pose immediate danger, but you should avoid contact with any potentially noxious chemical under all circumstances. Toxic chemicals should be handled with great care, and proper ventilation (fume hoods with maximum settings) should be used. If fume hoods are not available, the toxic chemicals should be handled in well-ventilated rooms with open windows to allow good airflow. Most organic solvents are very volatile and flammable, so proper ventilation should be exercised as well. Always remember, if you can smell a substance, you are breathing it into your lungs.

8) Wear inexpensive clothing when working in a lab. Since there is a possibility of clothing being destroyed in a laboratory accident, a lab coat or an apron should be worn at all times. Do not wear sandals or thong shoes when in the laboratory. Confine long hair and/or.....**Much More**

Section II

LACHRYMATOR, DISABLING, AND IRRITANT AGENTS

Chapter 3: Physical Nature of Lachrymator, disabling, and irritant substances

Introduction

Riot control, disabling, and irritant substances are quite common in the field of law enforcement and civil authorities. They are used by the military for covert operations where disabling, confusing, and harassing the enemy is more important than killing. Common riot control agents include CS, chloroacetophenone, and bromobenzylcyanide. Common disabling agents are biphenyl arsenicals, bromoacetone, and chloropicrin, and irritant substances in general include BBC, chloroacetone, and dichloroacetone.

Most riot control agents are non-toxic, and their persistence is very low. The duration of action for most riot control agents is only 5 to 15 minutes on average, and their effects on the body are generally mild. Disabling agents such as adamsite are quite common vomiting agents, which are used by security personnel, bank vaults, and security sectors to disable intruders. Vomiting agents are virtually non-toxic, but the action upon the body can be very severe, producing nausea, vomiting, and general incapacitating results. Effects of vomiting agents upon the body can last hours, but produce no temporary illness. Irritant substances include a family of compounds called lachrymators. Lachrymators irritate the eyes, nose, throat, and skin resulting in itching, redness, rash, and general discomfort and pain. As with riot control agents, and disabling agents, lachrymators are considered non-toxic, but their effects upon the body can last for days.

Disabling and irritant substances are hardly ever used in riot control operations because of their general toxicity and severity of symptoms; although, some irritant substances are used as riot control agents, and may be classified as riot control agents rather than general irritants.

Physical properties of riot control, disabling, and irritant agents

Riot control agents are white to light colored solids, which have moderate to high melting points. Despite their high melting points, riot control agents are very volatile, and therefore easily disseminated through pyrotechnic munitions or other means. Most riot control agents are insoluble in water, but readily soluble in organic solvents such as alcohol, methylene chloride, ether, and benzene. Riot control agents like CS can be dissolved into water with the addition of propylene glycol for making irritant spray solutions.

Disabling agents are usually solids, with low to moderate melting points, but they may include colorless liquids, which may be volatile or non-volatile. The solids are not very soluble in water, but soluble in the usual organic solvents, but the liquids may be partially soluble in water, or only slightly soluble therein. Chloropicrin and bromoacetone are both very soluble in the usual organic solvents. Adamsite is a colorless to light colored solid, with a moderate melting point. It is not considered volatile under normal conditions, but can be generated into a smoke rather easily. Chloropicrin and bromopicrin are powerful disabling agents, which are colorless liquids. They are considered volatile under normal conditions, and can be disseminated using various techniques.

Irritating agents are colorless to light colored solids with low melting points, or colorless volatile liquids with high boiling points. Many irritating substances are either readily soluble in water, or partially soluble, and all are soluble in the usual organic solvents. Most irritating agents, which are solids, are very volatile and rapidly volatilize on standing. Liquid irritating agents tend to be quite volatile under normal conditions, and evaporate on standing under moderate rates.

Protection

Ordinary clothing gives adequate protection against riot control agents and disabling agents; although, some irritant agents can penetrate ordinary clothing leading to skin irritation. Some riot control agents like CS can stick to clothing, rendering a delayed irritant action. In most cases, ordinary respirators or gas masks protect against riot control, disabling, and irritant agents.

Decontamination

Decontamination is not needed for riot control agents, disabling agents, or irritants under field conditions. Within enclosed environments, general decontamination of disabling or irritants may be needed, including aeration, bleach treatment, or caustic soda. Under field conditions, riot control, disabling, and irritants are readily swept away through the slightest breeze. Decontamination of exposed personnel is not needed when riot control agents are used, and exposed personnel should be taken to fresh air. For disabling agents, there's little that can be done to decontaminate exposed personnel, and they should be taken to fresh air, and allowed to rest. Skin and eye exposure to irritating agents should be flushed with large amounts of warm water, with intermittent treatments with warm soapy water. In essence, there is no real method of decontaminating irritating agents under most conditions.

Mechanism of body action

Riot control and some irritating agents act upon the nerve endings, the cornea, and mucous membranes causing excessive fluid discharge, irritations, and general discomfort. The body detoxifies riot control and irritant agents quite readily, and the body recovers from any symptoms within minutes. Disabling agents act upon thio containing enzymes inhibiting them, and disrupting the pyruvate dehydrogenase system. The thio containing enzymes play a part in the energy production within the cells. Inhibition of these enzymes interferes with the respiration of the cell system, resulting in destruction of the cell structure. The result is a multitude of complex reactions that result in nausea, vomiting, muscle spasms, irritation, and a general sense of incapacitation.

Signs and symptoms

Exposure to riot control agents and irritant agents produces a multitude of symptoms including severe eye irritation, irritation to the nose and throat, skin irritation, skin redness, itching, rash, runny nose, profuse nasal discharge, soar throat, coughing, and severe bodily fluid discharge. Most symptoms are experienced within seconds of exposure, but only last for short periods of time. Ingestion, although far less common, leads to nausea and vomiting, with mild gastrointestinal disorders. Some irritants produce pronounced eye irritation followed by numbing or stinging pain, effects which can last hours or days, and some can even produce mild vesication. Inhalation of disabling agents produces severe burning and pain to the nose and throat, with excessive fluid buildup. Severe discomfort including nausea, vomiting, muscle tremors, and confusion usually follows within minutes of exposure. In some cases, symptoms may be delayed for up to 5 minutes, whereupon sneezing begins, and ends with headache and vomiting. Eye exposure to most disabling agents simply produces general irritation followed by itching, redness, and mild pain. In general, the eyes are relatively unaffected by the action of arsenical disabling agents, but bromoacetone and chloropicrin can produce severe eye irritation even under very low concentrations. Under high concentrations, arsenicals, and bromoacetone are capable of producing mild vesication, resulting in skin blisters; although, on the battlefield, high concentrations of these disabling agents is unlikely to be encountered.

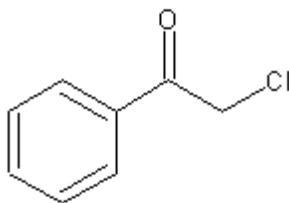
Treatment

Treatment for riot control agents is limited to exposure. In most cases, exposure simply dissipates after several minutes. In most cases, exposed personnel should be taken to fresh air, and allowed to rest until the symptoms disappear. Warm water can be used to treat soar eyes, by simply flushing. The eyes should not be rubbed by fingers or coarse materials as infection may set in. For most cases, riot control agents are detoxified in the body within 30 minutes, so no real treatment is necessary.

Treatment of irritant agents is similar, but some may require general care. For exposed eyes, the eyes can be flushed with a dilute baking soda solution, followed by large amounts of warm water. The eyes should not be rubbed, so as to avoid infections. Irritated or itching skin can be treated by the effected area with warm soapy water, followed by applying a calamine lotion, phenol ointments, or aloe vera gel.

Disabling agents need not be treated under any severe medical conditions, and exposed personnel should be dealt with as severity of exposure dictates. Personnel exposed to low or mild concentrations should continue work and duties as active bodies detoxify the agents faster. Personnel exposed to high concentrations should be taken to fresh air, and allowed to rest.

01-010. CN. Chloroacetophenone. MACE. 2-Chloro-1-phenylethanone; 2-Chloroacetophenone



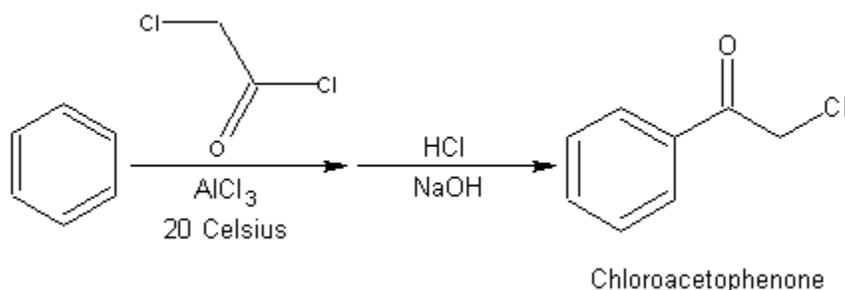
CN

CN forms colorless to white crystals with a melting point of 58 Celsius, and a boiling point 244 Celsius. The crystals have a slight apple odor, or odor of apple blossoms. The vapor has a characteristic pungent and irritating odor. CN is insoluble in water, but readily soluble in alcohol, chloroform, benzene, and hexanes. CN is a commonly used riot control agent used by police and civil authorities. CN is sometimes used by the military for riot control, especially when dissolved in chloroform, or benzene, but its overall use by the military is limited. CN causes general irritation to the nose, throat, and eyes. Eye exposure to the vapor causes moderate irritation with tearing and fluid drainage. Inhalation produces immediate irritation to the nose and throat leading to coughing, and congestion. Skin contact to CN can lead to mild irritation, and a sense of mild stinging under higher concentrations. The persistence of CN in the environment is very low; ranges from 30 minutes to several hours under normal conditions. CN can persist within enclosed areas for up to 2 days. CN is most commonly disseminated from smoke generating munitions, but numerous reports have noted that rioters and/or protestors can simply pick up the devices, and throw them back at the police. As a result, CN should be disseminated from aerosols for effective dissemination. CN can be decontaminated by washing with large amounts of hot soapy water. **CN is a fast acting riot control agent capable of dispersing personnel within minutes of dissemination. Eye exposure to 25 to 50 milligrams can produce irritation, and tearing. Inhalation of 50 to 80 milligrams can produce sneezing, coughing, irritation to nose and throat, and congestion. CN is regarded as non-toxic, but reports have shown chronic exposure may lead to slow systematic poisoning.**

OVERALL RATING (scale from 1 to 10)	
<i>Effectiveness (as riot control agent): 6</i>	<i>Field Stability: 9</i>
<i>Persistence (open area): 4</i>	<i>Storage stability: 9</i>
<i>Persistence (enclosed area): 5</i>	<i>Toxicity (as riot control agent): 7</i>
TOTAL EFFECTIVENESS (as riot control agent): 6.6	
OVERALL TOXICITY (as warfare agent): 1	

Procedure 1-010A: Preparation of CN

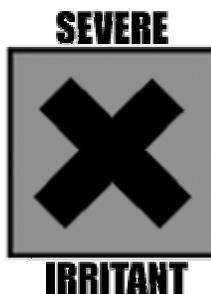
Summary: CN is readily prepared by reacting benzene with 2-chloroacetyl chloride in the presence of anhydrous aluminum chloride. The reaction is quite smooth, without much heat buildup. After the reaction, the reaction mixture is washed with acidic ice water, then with a sodium hydroxide solution, and then the resulting reaction mixture is treated with methylene chloride. The reaction mixture is then separated into two individual layers by the use of a separatory funnel. The removed methylene chloride layer is then evaporated to remove excess benzene, and to remove the methylene chloride solvent. The dry solid product is then recrystallized from hexanes. The collected crystals can be then dried via vacuum or air-dried.



Reaction Equation (by products omitted)

Materials:	1. 100 grams of dry benzene	5. 35.7 grams of sodium hydroxide
	2. 3 grams of anhydrous aluminum chloride	6. 100 milliliters of methylene chloride
	3. 43 grams of 2-chloroacetyl chloride	7. 10 grams of anhydrous calcium chloride
	4. 7.5 milliliters of 35 to 38% hydrochloric acid	8. 300 milliliters of dry hexanes

Hazards:



Do not attempt in anyway to prepare CN using the following procedure unless proper safety precautions are taken. 1) Perform all operations with good ventilation, maintain proper eye protection using eye wear that completely covers the eyes and forms a good seal on the face, and wear nitrile gloves. 2) After each procedure, all glassware and non-electric equipment should be soaked in hot soapy water before removing from the clean box (do not use bleach), and/or before rinsing and storing. Any electrical equipment that may be contaminated (even if suspected), such as hot plates and stirring equipment should be carefully wiped down with a rag soaked in a hot soapy solution (do not use bleach) followed by wiping down with a rag soaked in hot water. 3) The desired CN product should be stored in amber bottles, preferably non-breakable containers, and stored in a cool dry place away from sunlight. The bottles should also be placed inside an airtight sealed plastic bag, such as a 'ziplock' bag. 4) Storage of any and all riot control agents should be in cabinets, drawers, or the like, and said storage spaces should be equipped with proper ventilation.

Use caution when handling anhydrous aluminum chloride, which reacts with water producing toxic and corrosive fumes. Concentrated hydrochloric acid, and sodium hydroxide are corrosive to tissue, handle with care. Benzene is a known carcinogen, avoid inhalation of the vapors, and extinguish all flames before using; benzene is highly flammable. Use care when handling 2-chloroacetyl chloride, which is corrosive.

Procedure: Into a suitable flask, add 100 grams of dry benzene, and then add and dissolve 3 grams of anhydrous aluminum chloride. Thereafter, place this mixture into a cold-water bath, and chill to about 20 Celsius. Then slowly add drop wise, 43 grams of 2-chloroacetyl chloride over a period sufficient to keep the benzene mixture below 30 Celsius at all times. During the addition, rapidly stir the benzene mixture. After the addition of the 2-chloroacetyl chloride, continue to stir the reaction mixture for 1 hour at a temperature below 30 Celsius. Then add a solution prepared by adding 7.5 milliliters of 35 to 38% hydrochloric acid to 150 milliliters of ice water, and then add this acidic ice water solution to the reaction mixture at all once. Then stir the whole reaction mixture for about 20 minutes. Thereafter, carefully add a sodium hydroxide solution prepared by adding and dissolving 35.7 grams of sodium hydroxide into 564 milliliters of water, and then add this sodium hydroxide solution to the reaction mixture all at once. Note: Sodium hydroxide develops much heat when dissolved in water. Allow the sodium hydroxide solution to cool to room temperature, and then chill this sodium hydroxide solution to 5 Celsius by placing it into a freezer before adding it to the reaction mixture. After the addition of the sodium hydroxide solution, rapidly stir the whole reaction mixture for about 10 minutes. Thereafter, add 100 milliliters of methylene chloride to the reaction mixture, and then stir the reaction mixture for 30 minutes. Then place the reaction mixture into a separatory funnel, and drain-off the lower organic layer. Thereafter, quickly dry this lower organic layer by adding 10 grams of anhydrous calcium chloride, and then stir the mixture for 10 minutes; then filter-off the calcium chloride. Then place this filtered lower organic layer into a rotary evaporator, or vacuum distillation apparatus, and remove the methylene chloride, and excess benzene under vacuum until dry solid remains. When both solvents have been removed, and dry solid remains, remove this dry remaining solid, and then dissolve it into 300 milliliters of dry hexanes. Note: A small amount of the dry solid may not dissolve, if this is the case, filter-off these insoluble impurities. Thereafter, recrystallize the desired product from the hexanes, and afterwards, vacuum dry or air-dry the collected crystals. The result will be about 60+ grams of dry CN crystals. Note: The dry CN crystals should be stored in amber glass bottles in a cool dry place until use.

Section IV

BLISTER AGENTS (POTENT VESICANTS: TISSUE DAMAGING AGENTS)

Chapter 7: Physical Nature of Blister agents

Introduction

The blister agents, or vesicants are a class of chemical compounds that react with tissue producing series damaging effects. Blister agents are capable of producing burn like damage to the skin, eyes, respiratory tract, and gastrointestinal tract. Many of these burns resemble fire burns. Exposure to blister agents leads to horrific blisters, soars, welts, and burns within 24 hours of exposure and these effects can be very painful in nature. The blisters themselves usually contain secondary toxic compounds as the fluid, which can cause the blisters, soars, or burns to spread over wider areas of the body.

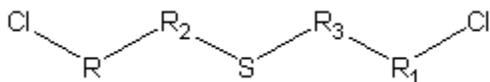
There are four major classes of blister agents: 1) Sulfur mustards, 2) Nitrogen mustards, 3) Arsenicals, and 4) Halogenated oximes. Sulfur mustards are the most well known, and were first prepared in the early 1800's by German and French chemists. Their damaging nature upon the skin was not realized until the late 1800's when they were then mass produced by the Germans for use in warfare; large amounts of mustard gas were used by the Germans during World war I, resulting in the deaths of over 120,000 French, and British troops. Note: American casualties from mustard gas and other chemical attacks by the Germans were virtually non-existent due to the fact the America did not get involved in the war until it was practically over with.

Mustard gas was the first chemical warfare agent used on a massive scale in war, and it paved the way for the future of chemical warfare. Nitrogen mustards were developed some decades later, and are more potent then the sulfur mustards. Arsenicals were adopted around the time period, and they have similar effects. Arsenicals can double not only as blister agents, but blood agents as well.

Blister agents are designed to contaminate areas to halt enemy troop movements, divert movements, force enemy troops to remain in full protective gear for long periods of time thereby degrading motivation and moral, and to harass, and confuse enemy personnel. Blister agents are used to produce injuries rather than to kill, although exposure to them can be fatal under appropriate conditions. Blister agents are very persistent in the environment, especially during dry and cold periods. They are capable of penetrating rubber, wood, clothing, vegetation, and some plastics. Areas contaminated with blister agents can give rise to casualties' weeks after dissemination. Simply sitting down on a patch of grass contaminated can lead to poisoning. Example, during World War I, a British soldier sat down on a patch of grass, which was unknowingly contaminated with mustard gas (in the form of small droplets). Several hours after the exposure, he developed severe blisters and burns on his butt and backside. This contaminated area was thought to have been cleared after its contamination three weeks earlier.

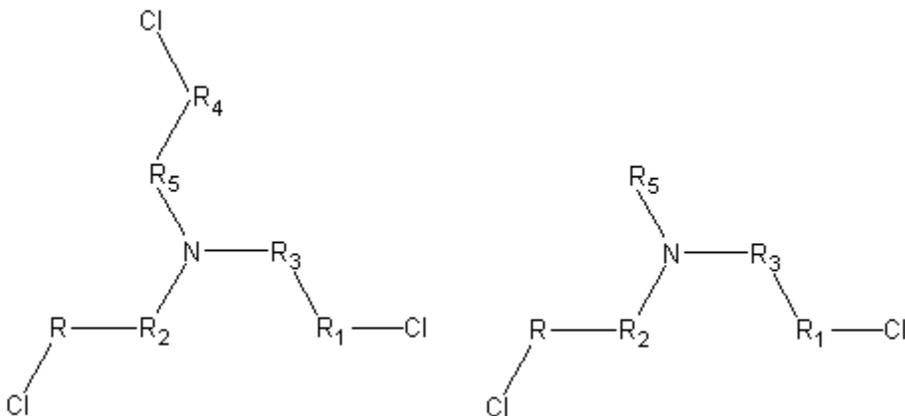
I. Mustard agents

Mustard agents are derived into two groups: 1) Sulfur mustards and 2) Nitrogen mustards. The sulfur mustards are classified by the following group.



R, R1, R2, and R3 can be methyl, ethyl, or isopropyl groups, but are usually methyl groups. The chlorines on the R and R1 groups can be replaced by bromine, but this is not usual. The chlorine atoms are usually on the beta position, meaning off the R and R1 groups. If the chlorines were off the R2, or R3 groups, vesicant properties would still exist, but would be less severe. All sulfur mustards are classified by their central sulfur atom, which is clearly visible at this point.

Nitrogen mustards are similar to sulfur mustards, but the central sulfur atom is replaced by nitrogen. The chlorines of nitrogen mustards are in the usual places.

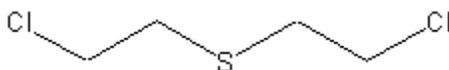


In the above figures, all the R groups can be methyl, ethyl, propyl, isopropyl, or tert-butyl. The chlorines can be replaced with bromine, but this is not usual.

Physical and Chemical properties.....Much more

Chapter 8: Preparation of Blister Agents

03-001. Sulfur Mustard. Mustard gas. Kampfstoff. Yperite; 2,2'-Dichlorodiethyl sulfide; bis(beta-chloroethyl)sulfide



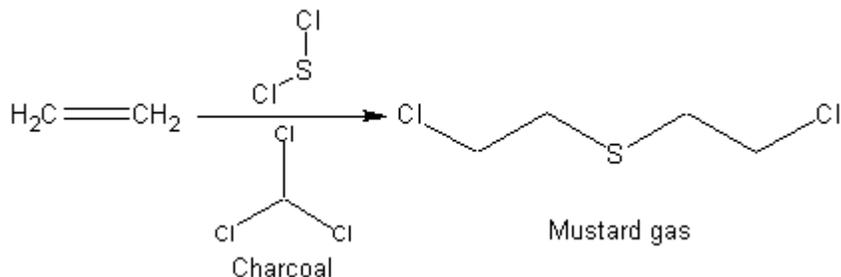
Mustard Gas

Mustard gas forms a colorless to yellowish to brown, oily liquid, which produces a colorless vapor. The vapor has a tendency to linger in low lining areas such as ditches, holes, trenches, and the like. The impure liquid has an odor of freshly cut mustards, hence the name. Pure mustard gas has a relatively mild sweet, agreeable odor, or no odor at all. Dangerous levels of mustard gas cannot be detected by the average soldier, even when the mustard gas is impure. It has a melting point of 14 Celsius, and a boiling point of 217 Celsius (begins to decompose from 150 to 170 Celsius); it can be distilled at 98 Celsius under a vacuum of 10 millimeters of mercury. Mustard gas is insoluble in water, but soluble in most organic solvents, and in lipids. It is volatile with steam, and can be steam distilled under normal conditions. Mustard gas is a suspected carcinogen, but this is of no importance due to its vesicant properties. Mustard gas is easily decomposed by alkalis, or by bleaching powder, and bleaching powder is the most effective material for its decomposition; ordinary bleach can be used as well. Because mustard gas has a high melting point, its use in cold climates has its disadvantages. Although, in dry cool climates, mustard gas can remain somewhat “dormant”, until the temperature rises, whereupon the crystals of mustard gas change to liquid. This effect can be used to contaminate areas with mustard gas for up to 3 months under affluent conditions. During wet and warm conditions, mustard gas has very poor persistence, and may only last for up to 7 days under normal conditions; although, reports have indicated that dangerous levels of mustard gas can remain in said environments for up to 30 days. Mustard gas can be mixed with HN1, HN2, or HN3 for use in military operations. It may also be thickened by admixture with oils. Mustard gas is capable of producing violent burns and blisters to any exposed skin upon contact. The actual onset of burns and blisters can be delayed by up to 36 hours. Exposed personnel will be unaware of skin contact with said agent until it's too late. Blisters should be broken, and scrubbed immediately with bleaching powder, or bleach. All wounds should be treated immediately to prevent potential bacterial infections. Eye exposure to agent can lead to delayed illness including blindness. Mustard gas is less toxic, and less efficient than HN1, HN2, or HN3, and is no longer used by most modern militaries. Mustard gas can be disseminated using aerosols, explosives munitions, atomizers or humidifiers, or foggers. **Mustard gas is a moderate delayed action casualty producing agent capable of causing casualties within 4 to 24 hours after exposure. The lethal dose for 50% of population in rats through inhalation is 1500 to 3000 milligrams per kilogram. The lethal dose through inhalation by the average man may be as little as 1200 milligrams, but may be as high as 2 grams due to slow onset of symptoms. Mustard gas may cause severe illness if inhaled or ingested including violent coughing, choking, stomach disorders, digestive disorders, bleeding, and severe discomfort. Mustard gas on the skin can go unnoticed until symptoms are observed (4 to 24 hours). Skin exposure to as little as 100 milligrams can produce blisters within 32 hours exposure. Eye exposure to as little 100 to 200 milligrams may cause severe eye damage resulting in potential blindness.**

OVERALL RATING (scale from 1 to 10)	
<i>Effectiveness (as blister agent):</i> 8	<i>Field Stability:</i> 8
<i>Persistence (open area):</i> 8	<i>Storage stability:</i> 9
<i>Persistence (enclosed area):</i> 9	<i>Toxicity (as blister agent):</i> 7
TOTAL EFFECTIVENESS (as blister agent): 8.1	
OVERALL TOXICITY (as warfare agent): 3½	

Procedure 3-001A: Preparation of Mustard gas (levinstein process)

Summary: Mustard gas is easily obtained by heating ethylene gas and sulfur dichloride under pressure. A small amount of activated charcoal is added to act as an inert carrier. After the reaction, the mixture is simply filtered, evaporated to remove the solvent, and then fractionally distilled to obtain a refined mustard gas product of purity averaging 90%.



Reaction Equation (by products omitted)

Materials:	1. 50 grams of dry ethylene gas	3. 200 milliliters of methylene chloride
	2. 72 grams of sulfur dichloride	4. 5 grams of activated charcoal

Hazards:



Do not attempt in anyway to prepare mustard gas using the following procedure unless proper safety precautions are taken. 1) Perform all operations in a clean box, in which is completely sealed from the air. Note: A clean box in this case is not needed. Mustard gas can be safely prepared as long as the maker wears proper gas mask, and nitrile gloves. 2) After each procedure, all glassware and non-electric equipment should be soaked in a bleach solution before removing from the clean box, and/or before rinsing and storing. Any electrical equipment that may be contaminated (even if suspected), such as hot plates and stirring equipment should be carefully wiped down with a rag soaked in bleach, followed by wiping down with hot water. 3) The desired mustard gas product should be stored in amber bottles, preferably non-breakable containers, and stored in a cool dry place away from sunlight. The bottles should also be placed inside an airtight sealed plastic bag, such as a 'ziplock' bag. 4) Storage of any blister agent should be in airtight cabinets, drawers, or the like, and said storage spaces should be equipped with chemical agent detection monitors to alert of any potential leakage.

Use caution when handling sulfur dichloride, which reacts with water yielding toxic and corrosive vapors. Extinguish all flames before using ethylene gas, which is highly flammable.

Procedure: Assemble the apparatus illustrated in 038, and then fill the reaction flask with 200 milliliters of methylene chloride, 5 grams of activated charcoal, followed by 72 grams of sulfur dichloride. Then begin the nitrogen purge, and thereafter, bring the mixture to a reflux at about 60 Celsius, and when the temperature reaches 60 Celsius, begin rapidly bubbling a total of 50 grams of ethylene gas into the reaction mixture while stirring the reaction mixture, and maintaining its temperature at 60 Celsius. The ethylene gas addition should take no more then 3 hours. After the addition, continue to reflux the reaction mixture at 60 Celsius while vigorously stirring for an additional 30 minutes. After 30 minutes, remove the heat source and allow the reaction mixture to cool to room temperature. Thereafter, filter-the reaction mixture to remove any insoluble materials, and then place the filtered reaction

mixture into a rotary evaporator or vacuum distillation apparatus, and remove the methylene chloride under vacuum. When all the methylene chloride has been removed, remove the remaining residue, and then place it into a clean vacuum distillation apparatus, and fractionally distil the mustard gas at 98 Celsius under a vacuum of 10 millimeters of mercury to obtain a refined mustard gas product of purity from 85% to 98%. Further purification is technically not needed for use in military operations, but can be achieved through a second vacuum distillation.

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