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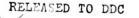
SAFETY PRINCIPLES FOR LABORATORY AND PILOT-PLANT OPERATIONS WITH EXPLOSIVES, PYROTECHNICS, AND PROPELLANTS

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PUBLISHED UNDER THE TECHNICAL DIRECTION OF THE NAVAL ORDNANCE LABORATORY, WHITE OAK

SAFETY PRINCIPLES FOR LABORATORY AND PILOT-PLANT OPERATIONS WITH EXPLOSIVES, PYROTECHNICS, AND PROPELLANTS



BY THE NIVAL ORDNANCE LABORATORY

Without restrictions

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SAFETY SUMMARY

LISTED BELOW IS EVERY "WARNING" CONTAINED IN THIS PUBLICA-TION AND THE PAGE ON WHICH THE "WARNING" IS LOCATED. ALL PERSONNEL INVOLVED IN EXPLOSIVE, PYROTECHNIC, AND PROPEL-LANT OPERATIONS MUST FULLY UNDERSTAND THE "WARNING" AND THE PROCEDURE(S) BY WHICH THE HAZARD IS TO BE REDUCED OR ELIMINATED.

WARNINGS

A serious explosion can result when halogen-containing solvents (carbon tetrachloride, chloroform, "Perclene", and "Triclene") are heated with strong reducing agents such as free metals or their hydrides.

Page 25

Perchloric acid and perchlorates are especially dangerous. Because of their sensitivity and tremendous potential destructive power, special permission from a supervisor must be secured before working with them.

Page 25

To avoid fires, explosions, and burns, handle volatile oxidizing agents with care. Avoid high temperatures and high concentrations. Clean up any spilled oxidizing agent promptly.

Page 26

The first evidence of the physiological action of the chlorohydrocarbons is a narcotic effect. Symptoms of excessive exposure are headache, undue fatigue, or nausea. Immediate access to fresh air rapidly eliminates the above symptoms from the body. However, repeated exposure may produce delayed or cumulative physiological effects. Liver damage from carbon tetrachloride has been reported.

Page 27

Pay attention to the low allowable concentrations for tetrachloroethane (5 ppm) and pentachloroethane (5 ppm). The maximum allowable concentrations for these compounds are lower than for hydrogen sulfide and hydrogen cyanide.

Page 27

The use of trichloroethylene - dry ice freezing mixture must be conducted from behind an efficient hood. Do not allow the mixture to stand in an open container between uses.

Page 27

SAFETY SUMMARY (Cont'd)

WARNINGS

Avoid skin contact with both liquid and vapor chlorohydrocarbons. Absorption through the pores can cause the same symptoms as inhalation. Removal of the skin's natural oils can cause inflammation. Avoid vapor contact with the eyes.

Page 28

Do not add sodium, caustic, or caustic solutions to 1,2-dichloroethylene or to trichloroethylene; these may form monochloroacetylene and dichloroacetylene, respectively. Also, the addition of sodium, caustic, or caustic solutions to tetrachloroethane may form either of the above chloroacetylenes. Avoid the contact of caustic with the above chlorohydrocarbons except under carefully controlled experimental conditions.

Page 28

Many dangerous reactions are described in public literature without a word of caution. Hence, literature precedent is a poor basis for disregarding any known indication of hazard. On the other hand, hazards of specific compounds can be found in library searches.

Page 33

Never ask the glass blower to repair contaminated equipment. Page 35

When procedures are released by a supervisor to his operators, there shall be no deviation from the procedures, except by written authorization of higher authority.

Page 54

Do not apply information valid for one explosive system to another without careful study and testing.

Page 56

Do NOT smoke while wearing contaminated protective clothing. Page 59

Do not use copper sulfate solution in the eyes.

Page 64

OP 3237

SAFETY SUMMARY (Cont'd)

WARNINGS

Experimental delay powders can be very hazardous depending on the metal used, particle size, and oxidizers used. Delay work is made more hazardous as many of the compositions require very sensitive oxidizers. Delays using fine zirconium or titanium are in the hazardous category. Those using manganese or zirconium-nickel alloys are much less sensitive and burn without flames but emit much heat.

Page 66

Isocyanates and acrylates are toxic compounds. Acrylate monomers can cause irritations to the mucous membranes, gastro-intestinal disorder, headaches, and kidney irritation. Concentrations of toluene di-isocyante as low as 2 ppm have been shown to cause bronchial pneumonia in animals.

Page 74

Any facilities at which mixing, blending, and loading operations of a high-energy propellant are in progress must be separated by double the intraline distances prescribed in OP 5, Volume 1, Table 7.11.

Page 80

Detonable propellants and motors with detonable propellants shall be machined only by remote control

Page 89

The quantity of waste propellant plus the propellant in processed and unprocessed motors shall not exceed the explosive limit of the room or cubicle.

Page 90

Only trained and competent personnel shall adjust, clean, and repair propellant operating equipment.

Page 94

Avoid ingesting toxic materials through the mouth. Skin afflictions account for the largest number of occupational disease cases. Differences in individual susceptibilities to this type exposure make it very difficult to set up standards for safe handling of any chemical.

Page 99

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SAFETY SUMMARY (Cont'd)

WARNINGS

Wash the hands and face thoroughly and frequently when exposed to chemical dusts or solutions.

Page 99

Avoid long contact with even those chemicals which are supposedly harmless, as many of these are readily absorbed through the skin. A change in solvent may nullify the protective action of skin oils and make even short exposure harmful. For example, a short exposure to an aqueous solution may be harmless, while the same exposure time to the same agent dissolved in methanol or trichloroethylene may produce a major lesion.

Page 100

Wear glasses, aprons, face shields, and protective creams to prevent contact with the body. Report any sign of skin irritation to the doctor. In working with chemicals of known hazardous type, consult another person who is familiar with the materials or classes of materials. The toxic limits in this publication are intended for general guidance. Obtain definite confirmation on the use and handling of certain chemicals.

Page 100

Assume that all chemicals are toxic.

Page 100

A mild anaesthesia or unsuspected intoxication can impair judgment and cause an accident. An unpleasant odor is not necessarily evidence of the presence of toxic materials. However, it may be sufficiently distracting to become a contributing factor in a seemingly unrelated accident.

Page 100

Do not use carbon dioxide extinguishers on sodium or magnesium fires. Use only dry chemical extinguishers approved for use with these materials.

Page 103

Chapter 1

INTRODUCTION

PURPOSE

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OP 3237 is directed at personnel associated with the research, development, and pilot-plant manufacture of explosive, pyrotechnic, and propellant materials. The objective of the information presented is to emphasize the importance of safety programs and the necessity that all employees adhere stringently to them. The publication will explain the nature of certain hazardous conditions and point out the proper steps which must be followed in order to eliminate a hazardous condition which could lead to a serious injury or even death.

SCOPE

The publication is concerned with safety measures necessary in handling explosives, pyrotechnics, and propellants; this information is directed at three facilities: the laboratory, the pilot-plant, and initial production.

Chapter 2 contains safety responsibilities and mandatory safety rules.

Chapter 3 describes personal protective safety equipment and the fixed safety equipment used in laboratories and pilot-plant facilities.

Chapter 4 sets forth the study regimen which shall be followed before proceeding to production of a larger quantity of explosive, pyrotechnic, or propellant.

Chapter 5 discusses design criteria and safety objectives for explosives in the laboratory.

Chapter 6 discusses special hazards of larger-scale operations with rules and practices which shall be promulgated by plant organization personnel.

Chapter 7 contains safety precautions for laboratory work with pyrotechnics, safe operating procedures, and uses of pyrotechnics.

1

Chapter 8 covers pilot-plant pyrotechnic operations.

Chapter 9 describes preliminary study methods and preparation and handling of solid composite propellants.

Chapter 10 sets forth design criteria for propellant operations buildings and the safety guide-lines which shall be observed in the several operations necessary to fabricate composite propellants, case-bonded propellants, and non case-bonded propellants.

Chapter 11 is a discussion of various types of hazards and preventive safety measures.

Chapter 12 pertains to fire prevention, fire symbols, and safety regulations.

Those persons responsible for the design of propellant manufacturing facilities are required to observe all the applicable safety precautions set forth in this publication. Many of the safety precautions may be applicable to full-scale manufacturing facilities and should be given due consideration in their design.

Appendix A is the bibliography to Reference Publication (1), "Dangerous Properties of Industrial Materials" by N. Irving Sax, which contains information about hazards of 9000 chemicals or compositions.

Appendix B is a sample check list. This form is to be used to record all information about previous work done in ALL locations which are pertinent to a given scale-up operation. The check list should also include ALL persons with knowledge of the original work, public abstract literature where applicable information may be found, and the DOD facilities from whom literature may be requested.

Appendix C is a sample APPROVAL FOR POTENTIALLY HAZARDOUS OPERATION. This form shall be completed and signed by the supervisor before a hazardous operation is assigned to an operator in a laboratory or pilot-plant.

An index is provided to give users of this publication easy reference to various subjects pertinent to their work.

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Chapter 2

SAFETY

GENERAL

The Navy has ascertained that the major causes of explosive accidents in research, development, and manufacture of explosive, pyrotechnic, and propellant materials are (1) improper or inadequate operating procedures, (2) failure to observe regulations, (3) failure to understand hazards, and (4) failure to take necessary precautions.

The use of the word "failure" in the above statement denotes human error. Further, human error denotes ignorance, specific carelessness, or poor judgment. The first experimenters with electricity were made safetyconscious with the WARNING "Keepone hand in your pocket."

In the case of explosives operations, ignorance, specific carelessness, or poor judgment on the part of any operator actually make that operator a hazard in himself. To eliminate the "self-hazard" condition, it is imperative that the operator "keep one hand in his pocket" by strict adherence to an established safety program. This adherence will eliminate the human errors to such a degree that explosives operations will be as safe as possible.

Accidents are usually caused by a chain of events, and the safety program must keep this chain of events broken. SAFETY IS EVERYONE'S RESPONSI-BILITY, and the Navy has found it necessary to develop safety-consciousness with new technical employees. Indifference or bravado expressed by employees in the statement that "accidents are bound to happen" can best be prevented by a well-planned safety program in which accidents cannot and will not happen. Good communications up and down the line organization are a MUST. The rules and regulations promulgated by the Commanding Officer, Safety Officer, and supervisors must be explicitly understood and carried out in accordance with the information presented in this publication.

SAFETY RESPONSIBILITIES

By their very nature, safety responsibilities cannot be delegated to any one person or to any one group of persons. Specific safety responsibilities of the individual employee and of supervisors are given in the remainder of this chapter.

INDIVIDUAL EMPLOYEE. The individual employee must realize and concern himself with the principle that ALL PERSONAL INJURIES ARE

AVOIDABLE. It is expected that all laboratory and pilot-plant employees think, act, and live according to the safety program in force in their work areas. They must realize that the program depends on their sincere safetymindedness and good judgment as an integral part of their daily activity. Only in this manner can injury-free performance become a reality.

The primary purpose of this publication is to warn each individual employee in a laboratory or pilot-plant that he is responsible for his safety and for the safety of his fellow employees. The individual employee shall realize that when he does not comply with safety regulations, he not only endangers his own life, he puts his fellow employee's life in jeopardy. Every employee shall be thoroughly familiar with the information in this publication.

<u>SUPERVISORS</u>. The Navy strives to provide its management and line supervision with safety devices. Safety responsibility follows the line organization. The commanding officer is responsible for the accident-free performance of his supervisors and their individual employees. A superior is not exonerated for an employee's poor judgment, carelessness, or failure to comply with SOP's.

The supervisor must acknowledge his duty to educate and supervise his men on all aspects of safety. He must also promote safety-mindedness by persuasion and by authoritative force.

MANDATORY SAFETY RULES

In compliance with the above paragraph, the supervisor is hereby directed to instruct those personnel in his charge with the following mandatory safety rules. These rules apply to all laboratory and pilot-plant areas. Every employee will comply with the rules in spirit as well as to the letter.

NO SMOKING. Obey the NO SMOKING signs and all other fire or explosion preventive rules in your area and at all other locations.

EQUIPMENT MAINTENANCE. Maintain all moving parts equipment in optimum operating condition.

EMERGENCY SITUATION. Keep access to exits clear at all times. Stow personal protective safety equipment and fire-fighting apparatus within easy reach.

SAFETY GLASSES AND GOGGLES. WEAR SAFETY GLASSES AND GOGGLES as minimal eye protection.

DISTRACTION. Do not startle or distract the attention of anyone involved in hazardous laboratory or pilot-plant procedure.

HORSEPLAY. Do not engage in horseplay or practical jokes.

FOOD. Do not bring food into the laboratory or pilot-plant. Do not eat or

drink any food in the laboratory or pilot-plant. Do not take any food into handling or storage facilities.

WORK REQUIREMENT. Prohibit hazardous laboratory and pilot-plant work unless a second person is within hearing distance. This rule shall be stringently enforced and adhered to during both regular and overtime working hours.

IMPORTANT GUIDING PRINCIPLES FOR SAFE WORK

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The following safety principles are intended to guide the employee in the successful completion of his task. They require a constant exercise of common sense and good judgment in their interpretation and application. Furthermore, the supervisor will construe an employee's obvious neglect of these principles or indifference to them as a major employee deficiency.

USE THIS MANUAL. Familiarize yourself thoroughly with this publication. Be particularly certain that you read and understand the section applying to any new work which you are planning to do.

EMERGENCY EQUIPMENT. Know the location and use of all emergency equipment in your area. Also know the types of personal protective devices which are to be used for work under especially hazardous conditions. Use the appropriate device in a situation which calls for its use. These devices are described in the third chapter of this publication. The supervisor must warn his employees about specific hazards (fire, explosion, and missile) and protect them from any hazard with adequate WARNING signs and placards.

WARNINGS. Observe and comply with all WARNING signs and placards in your area.

HOUSEKEEPING. Keep your work area in a neat and orderly condition at all times.

<u>REMEDIAL PROCEDURES.</u> Watch for unsafe conditions and especially for unsafe actions. Remedy these conditions and actions by reporting them at once to the individual and his supervisor.

WORK PLAN. Follow an obligatory work plan or standard operating procedure. Avoid emergencies by careful thought and planning. Try to foresee your own actions in an emergency condition.

EXPERIENCE. Call on all experience to guide you in anticipation of possible hazards.

FIRE OR INJURY. Keep away from fire unless you are assigned a firefighting duty. Keep away from injured personnel unless you can administer first aid.

SAFETY. Think SAFETY, act SAFETY, talk SAFETY, and live SAFETY until it becomes your most firmly established habit.

<u>RULES</u>. Know and abide by all applicable safety rules in the areas to which your work may take you. When entering a strange area, identify yourself to the supervisor, and inform him of your mission.

REPORTING. Promptly report all fires, injuries, or explosions in your area.

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Chapter 3

SAFETY EQUIPMENT

GENERAL

This chapter describes personal protective equipment which shall be procured and used by individual operators. ALL personnel who enter a laboratory or pilot-plant area where chemical operations are in process must make use of this protective equipment as required by the counsel area supervisor. The supervisor shall exercise his authority in evacuating the work area of any persons who are not wearing the proper safety equipment.

PERSONAL PROTECTIVE EQUIPMENT

The following safety equipments are designed for personal use and shall be worn as directed.

SAFETY GLASSES. Safety glasses are the minimum eye protection and must be available to all laboratory and pilot-plant operators. It is imperative that they be worn in all areas where "EYE HAZARDOUS" signs are posted. Eye protection is of paramount importance in the safety program because (1) the eye is irreplaceable, and (2) the eye, more than any other part of the body, is most susceptible to serious injury and damage by minuscule amounts of chemicals, flying particles, excessive heat, or radiation. Goggles shall be worn as indicated in the following paragraph.

GOGGLES. Goggles are necessary protection against splashes, fumes, dusts, flames, and flying particles. Supervisors are responsible in determining that their men are supplied with adequate protective equipment. Welder's goggles, glass blower's spectacles, flash goggles, and gas-tight goggles shall be readily available.

RESPIRATORY PROTECTIVE DEVICES

Respiratory protective devices are required and must be worn in areas of oxygen deficiency and when the atmosphere is contaminated with gas fumes or dust. It is imperative that each employee acquaint himself with the capabilities of these devices and that he use the device most applicable to his working environment. In general, these devices include respirators, gas masks, and canisters. The respirator must be properly fitted to the individual wearer and must not be worn by another person.

The following devices are to be used only where there is no oxygen deficiency.

<u>POCKET RESPIRATOR</u>. This respirator consists of a rubber mouthpiece, a nose clip, and a cartridge (filter) retainer mounted at the front of the mouthpiece. This respirator enables personnel to make a quick escape from concentrations of a variety of gases including acid gases, ammonia, organic vapors, and highly toxic aerosols.

GASFOE.* This respirator consists of a basic facepiece, a cartridge (filter) retainer, and four replaceable, interchangeable chemical cartridges (filters). The respirator provides protection against chlorine, organic vapors, acid gases, ammonia, and metallic mercury vapors. Used only where there is no oxygen deficiency, this device does not provide protection from HCN. Auxiliary screens can be procured for paint spraying operations.

DUSTFOE.*. This respirator consists of the same components mentioned in the above paragraph, with the exception that the Dustfoe has only one type cartridge (filter). This is a general purpose respirator and provides protection against dusts which are designated nuisance, lung damaging, and toxic. The nuisance dusts include coal, ashes, cement, sawdust, charcoal, and carbon. Quartz, asbestos, and sand are lung damaging dusts. The major toxic dusts are arsenic, barium, beryllium, chromium, copper, lead, and manganese.

These masks are to be used where there is a maximum concentration of about two percent.

CANISTER-TYPE GAS MASKS. Industrial canister-type gas masks consist of the following integral parts: a single lens facepiece, a corrugated rubber hose, a canister harness, and a canister. The facepiece is made of plastic with plastic straps to adapt it to the wearer's head. At the base of the facepiece is an adapter which receives the upper end of the rubber hose. The lower end of the hose is attached to the canister. The harness fits around the wearer's neck and around his waist to secure the canister at waist level. A two-inch opening at the bottom of the canister permits easy air flow. The canister has a particulate filter; gas flow through the canister is controlled by internal baffles. Canisters are color-coded to designate the type of protection afforded. (See Table 1.)

These masks are for use only where there is no oxygen deficiency and where the maximum gas concentration encountered is about two percent. They should not be depended upon for exposures longer than five minutes, which would normally be time enough to escape from the contaminated area or to carry out a quick rescue operation. The canister should be replaced after each such use. When such a mask is used for occasional exposure to very low concentrations of gas, a regular replacement schedule should be set up based on the frequency and severity of exposure.

<u>ALL-SERVICE SEAMLESS CANISTER.</u> The All-Service mask is similar to the industrial type described above. Its designation is MSA*, and it is equipped with a red canister. The red canister affords protection from a combination of gases including two percent carbon monoxide by volume and three percent ammonia by volume.

* Mine Safety Applicance Collaria

* Mine Safety Appliance Co. designations.

cylinder which is strapped across the wearer's chest with a nylon harness. A pressure gauge on the demand regulator indicates pressure and available supply.

ACCESSIBILITY

The respiratory equipment described in the foregoing paragraphs must be made easily accessible to all employees at all times. The replaceable cartridges (filters) used with the pocket respirator, Gasfoe, and Dustfoe have the same color coding or letter designation as the canisters.

HAND, SKIN, AND BODY PROTECTION

The following equipment is designed to protect the wearer from injury and his clothing from damage. The most common incidents of this type are chemical splashes, excessive heat, and falling objects. Although the use of this equipment is dependent upon the existing hazards, the supervisor will determine the needed protection and inform his men.

The following items shall be used for hand, skin, and body protection:

SKIN CREAMS. Baby oil, mineral oil, lanolin-based emollients, or industrial skin creams shall be available. These may be liberally applied to the face and hands when necessary.

GLOVES. The types of gloves to be used by the employee in laboratory and semi-works areas are divided here into two categories: (1) chemical and caustic resistant, and (2) heat and cut resistant. Chemical and caustic resistant gloves include those made of neoprene, rubber, latex, molded rubber, and disposable plastic. The neoprene glove is used to handle organic solvents and chlorinated hydrocarbons. Heat and cut resistant gloves include those made of chrome leather, asbestos, cotton with leather palms, and moleskin mitts. Leather finger guards are also available, and these must be worn under a particular type glove when hazardous conditions exist.

APRONS. Several types of aprons are available to the personnel in a laboratory or pilot-plant. They are made of plastic, chrome leather, cotton materials, and synthetic rubber. They protect the wearer's clothing from acids, caustics, chemicals, oils, water, greases, and gasoline. Detailed regulations for their use shall be enforced.

FLAME-RESISTANT COATS AND COVERALLS. Their use is mandatory in any location where a fire hazard exists. Coveralls shall be worn in work areas to protect the wearer's personal clothing. Coats and coveralls shall be kept clean, and changed as often as necessary.

SAFETY SHOES. Safety shoes are termed "conductive." They are designed to dissipate static electricity accumulated on the body. The term "conductive" signifies that the shoe has a minimum resistance of 25,000 ohms. This protects the wearer against grounding in case of accidental contact with live conductors. They shall be worn in all areas where static The following devices are used with a fixed air line supply system:

CONSTANT FLOW AIR LINE. This mask consists of a single lens facepiece, a corrugated rubber hose, and a plastic valve. The facepiece is similar to the facepiece used with the canister-type gas masks. The corrugated rubber hose is longer to provide freedom of movement around the supply air line. The control valve permits selectivity of air flow by the wearer.

Table 1

MSA Designation*	Protects Against	Color
GMD	Three percent ammonia by volume	Green w/gray stripe around the top
GMK	Two percent hydrocyanic acid gas (HCN) (equipped with COMFO half-mask facepiece)	White w/green stripe around the bottom and gray stripe around the top
GMA	Two percent organic vapors by volume	Black w/gray stripe around the top
GMC	Two percent acid gases and organic vapors by volume	Yellow w/gray stripe around the top

MSA CANISTER-TYPE GAS MASK DESIGNATION

* Mine Safety Appliance Co. designation.

DEMAND FLOW AIR LINE. This mask consists of a single lens facepiece, a corrugated rubber hose, and a demand regulator fastened to the wearer's belt. Air flows to the facepiece only when the wearer inhales and stops when he exhales. The demand regulator affords one man up to eight hours of service from a standard 244-cubic foot cylinder.

The following devices have self-contained oxygen supplies.

CHEMOX* OXYGEN BREATHING APPARATUS. This mask is a selfcontained unit which generates its own oxygen and provides complete respiratory protection for periods up to one hour. The mask is used in any area of oxygen deficiency or concentration of toxic gases. A replaceable canister absorbs the carbon dioxide from the wearer's exhaled breath. An automatic bell rings at the end of a pre-set time to warn the wearer that he must return to fresh air. The mask does not afford protection for poisoning through the skin.

EIGHT-MINUTE OXYGEN DEMAND. This mask provides respiratory protection for up to eight minutes. For jobs of short duration or for emergency escape, the mask is equipped with an eleven cubic foot air or oxygen

* Mine Safety Appliance Co. designation.

electricity in the wearer's body might create a spark which could ignite sensitive explosives, gas-mixtures, pyrotechnics, incendiary mixtures, or flammable vapors. They must also be worn by personnel who walk on conductive floors where exposed explosives are present.

These shoes lose their efficiency if worn under all conditions. Therefore, they shall only be worn in specifically designated areas. Mud and dirt will clog the pores of the shoes and prevent static electricity from passing into the ground. Pads, inner soles, and arch supports shall not be worn; the user must wear cotton socks.

<u>Testing</u>. Test of semi-conductive shoes shall be made prior to use and whenever necessary to insure that the shoes' resistance is within the required limits. The tests shall be made while the shoes are being worn by the employee.

The test instrument used for testing consists of two conductive plates. The test voltage shall be approximately 90 volts, and the test current in normal operation shall not exceed one milliampere. No more than two milliamperes shall flow across the plates. Tests shall not be performed in rooms in which explosives are present. Positive safeguards must be incorporated into the design of the instrument to eliminate all possibilities of electric shock to the person undergoing the test. Dirt and oils on the soles increase resistance. If removal of accumulated dirt and oils does not reduce the resistance below one megohm, the shoes shall no longer be worn for the purpose intended.

A record of tests shall be maintained at the location of the testing equipment. The following information shall be recorded for each shoe test:

- 1. Name of employee to whom shoes are issued;
- 2. Shoe condition at time of test;
- 3. Resistance readings;
- 4. Date of test; and
- 5. Initials of person conducting test.

HARD HATS. Hard hats shall be worn by all employees in any area where a hazard from falling objects exists.

Additional information with regard to procurement of personal protective equipment may be found in NAVEXOS P-422, Safety Equipment Manual, Revised Edition, (1960).

MECHANICAL SAFEGUARDS

The following safety equipments are either built into operational areas, or they are placed in these areas for fire prevention purposes.

SHIELDS AND BARRICADES. Shields are fixed to work tables, and the tables are permanently fixed to semi-conductive floors. A typical shield consists of three pieces of Lucite shielding and associated aluminum reinforcement. The front shield is bent back about 12 inches from its base to afford

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operator accessibility to the work area. The Lucite shields at each side are attached to aluminum bases, and clamps at the bases hold the side shields in a vertical position. The side shields are movable to give a larger work area when needed; the Lucite is from one-half- to one-inch thick, depending on the explosive hazard of the items to be tested. An aluminum block in the center of the work area is grounded, and the aluminum bases supporting the sides are also grounded.

Regardless of their designs, shields shall be tested and stencilled with the following quantity-distance information:

- 1. Number of grains of TNT;
- 2. Distance from center in inches.

Table 2 is presented as a general guide in the design and procurement of safety shields.

Table 2

SAFETY SHIELD CHARACTERISTICS

Material	Thickness (inches)	Limit in TNT Explosive Equivalents (grams)
Laminated Safety Glass	1/4	7
Laminated bullet-resistant glass	1/2	25
Laminated bullet-resistant glass	1	55
Armored fume hood*		60

* In some cases, the fume hood is equipped with a hinged blow-out wall in the rear and is always equipped with an exhaust fan to vent gases to the exterior of the operating facility.

SHIELD TESTING. When new explosives or new shield designs are contemplated, the shield and the manner in which it is secured to the bench shall be tested to two times the maximum explosive weight to be used at the bench.

SPRINKLER SYSTEMS. The deluge sprinkler system is most applicable to the laboratory or pilot-plant facility in that large amounts of water may have to be applied over large areas as soon as possible. In most cases, the system is activated by heat-responsive devices. The sprinklers have a temperature rating determined by the maximum temperature to which the ceiling areas are subject during normal (no fire) conditions. Table 3 shows the ratings and colors of sprinkler heads.

Table 3

Rating	Operating	Color	Maximum Ceiling
	Temperature (F)	Coding	Temperature (F)
Ordinary Intermediate High Extra High Very Extra High	135 - 150 - 160 - 165 $175 - 212$ $250 - 280 - 286$ $325 - 340 - 360$ $400 - 415$ 450 500	Uncolored * White * Blue Red Green Orange Orange	100 150 225 300 375 425 475

STANDARD TEMPERATURE RATINGS FOR AUTOMATIC SPRINKLERS¹

* The 135°F sprinklers of some manufacturers are half-black and half-uncolored; the 175°F sprinklers are yellow.

¹ Accident Prevention Manual, National Safety Council, 4th Edition, 1959

ALARM SYSTEMS. Alarm systems shall be used in conjunction with sprinkler systems to warn operators of impending danger. The alarm system shall be integrated with the sprinkler system to allow operators ample time to evacuate the danger area.

<u>CONDUCTIVE FLOORING.</u> Conductive flooring is necessary in all areas where there is a danger of static electricity. Static electricity must be dissipated to ground, and to this end an operating area may have a concrete floor imbedded with metal particles. In this case, the static electricity is dissipated into the floor and carried to ground through the steel members of the building's framework to a ground common to the entire building. Conductive rubber flooring can be used in either block or sheet form, and some blocks and sheets actually contain a ground wire running through them. The wires are connected to one another as the floor is put down and connected to the common ground. Resistance of conductive floors shall be tested every six months. (Refer to OP 5, Volume 1, Chapter 42.)

SHOWERS. Safety showers shall be installed in appropriate locations, and conspicuously placarded SAFETY SHOWER FOR EMERGENCY USE ONLY. With regard to non-emergency showers, it is advantageous to set up a doublelocker arrangement shaped like the letter H, with the non-emergency shower located between two locker rooms. In this type facility, the operator leaves his work clothing in a locker; then he must go through a shower room and take a shower before he can reach the second locker, where he changes into street clothes.

EXPLOSION-PROOF ELECTRICAL EQUIPMENT. The National Electrical Code prescribes the minimum requirements applicable to the design, installation, and use of electric motors, equipment and wiring. The NEC regulations are given in Article 4026, Volume 1, of OP 5. Explosion-proof lighting and motors are mandatory, and motors must be grounded with lowest feasible resistance. Article 4026 also specifies the following additional requirements:

1. Electric motors, controls, and wiring used in operating buildings, including equipment in separate motor rooms, shall be suitable for the particular hazardous locations involved.

2. Electric motors of any type shall not be installed in rooms where exposed explosives are handled unless the Bureau of Naval Weapons grants specific authorization in each case.

SAND BUCKETS AND FIRE EXTINGUISHERS. Sand buckets and fire extinguishers shall be placed at appropriate locations throughout the laboratory and pilot-plant facility. The supervisor shall instruct his men in firefighting procedures. Each man shall be assigned a separate and distinct duty during fire drills. If a small fire should occur, and can be confined to a small area, only the men necessary to carry out the fire-fighting operation shall remain within the area. In most areas, fire-fighting, especially by non-professional fighters, would be forbidden.

NON-FERROUS HAND TOOLS. Non-ferrous hand tools shall be used in all areas where a spark from a steel tool might become a fire-initiator. This rule applies especially to all handling and storage rooms where exposed explosives or explosive dusts or vapors are present. The tools are made of wood or non-sparking materials and will not produce sparks under normal conditions. The materials include bronze, lead, beryllium alloys, and monel metal.

EXTRA-HAZARDOUS OPERATIONS. Where extra-hazardous operations are contemplated, operations shall be remotely controlled from a protected personnel room.

<u>CLOSED-CIRCUIT TELEVISION</u>. Where applicable and feasible, closedcircuit television is an excellent investment in laboratory and pilot-plant areas. Closed-circuit television should be applied to those areas where the risk and hazard element is high, especially in the development of new propellants, pyrotechnics, or explosives.

AUDIO HOOK-UP. This publication further suggests an audio hook-up. The change in sound level during a process development is often a clue as to whether the process is proceeding normally.

Chapter 4

PRELIMINARY STUDIES

GENERAL

This chapter deals with the basic importance of the acquisition, compilation, and analysis of all previously recorded information about the hazards to be expected during the synthesis of high energy compounds. High energy compounds or compositions are understood by users of this publication to be those which will release more than about 800 calories per gram by burning or explosion. While both industry and the U. S. Government have been the source of many publications on explosive safety, none of these have been uniquely devoted to the sequence of operations required to develop new compounds and new compositions from the initial research worker at the laboratory through manufacture and production.

The research worker who accomplishes the initial synthesis of a high energy compound will frequently have no specific experience to guide him in the safety of these experiments. However, he may, by reducing the operation to very small scale, receive perfect protection by remote control or shielding. The initial experiments will then be repeated at the same small scale until a sufficient amount of material has been obtained and used for preliminary testing and evaluation. The information gained by these experiments may justify recommendation of a larger scale evaluation. The experience gained in these initial experiments must be used to insure the safety of those undertaking more advanced work.

The first act of those who undertake more advanced work, whether it be to increase the scale or not, must be to learn everything known about the equipment used, the chemicals, their interactions, and all experience of prior workers. The previously obtained experience and knowledge may not be available at the installation where additional work is to be done. It will be necessary to obtain all the experience and reports from all installations. This should be done by study of reports first, and then almost always it will be necessary to confer at least once with the previous workers. Such conferences may elicit news about incidents that were harmless but which under other circumstances may not be harmless and may cause personal or property damage. Such installations should be thoroughly investigated and all details reported fully.

It is strongly recommended that pilot plant employees seek information and counsel from the laboratory or person, irrespective of employment

location, having knowledge of the previous preparation of the high energy material in question. It is recommended that production plant personnel seek information and counsel from the pilot plant or personnel, irrespective of employment or location, having detailed knowledge of the manufacture of the high energy material in question. It is also strongly recommended that the user of high energy magerials seek information from all those having detailed knowledge of the properties of this material.

The characteristics of intermediate compounds in any synthesis must be known and understood since intermediates may be more likely to be dangerous than the final product. Furthermore, conditions during any synthesis are more favorable to violent reaction or even explosion than the conditions after the synthesized compound has been isolated as a pure explosive.

Arrangements for demonstration of the synthesis shall be set up to instruct new operators. Dummy run-throughs with inert materials shall be performed by the new operators so that they may become thoroughly acquainted with the process.

More detailed statements of safety principles, personnel responsible for safe operation, and explanation of origins of hazards are given in the following text. These nearly all involve chemicals or chemistry because the hazards of work with pyrotechnics, propellants, and explosives are caused by chemicals and their chemistry. Section 10 of reference publication (2) is highly recommended as a source of information about hazards of 9000 chemicals or compositions. A bibliography of Section 10 is included in this publication as Appendix A for the user's convenience. Also, chemical manufacturers supply free detailed information about their specific products.

CHECK LIST

To insure that all sources of information have been reviewed, the operator shall prepare a check list. A sample check list is included in this publication, Appendix B. Sources of the data required are available in reference publications 1 through 13, page 2. It is important to note that the literature on high energy compounds does not furnish all pertinent information about them, nor does it necessarily indicate where all pertinent information can be found. It must be noted that warnings may not appear in these publications. All workers with high energy compounds are emphatically warned against complacency due to absence of warnings in the research literature. For this purpose, the check list requires recording experience of men who have worked with the materials.

A suggested form of approval for potentially hazardous operation is included as Appendix C.

FATIGUE

Work with high energy compounds or compositions must not be undertaken by anyone who is mentally fatigued. It is highly advisable that physical fatigue be considered as forbidding such work also, since mental fatigue usually accompanies physical fatigue. Those who are mentally fatigued are less able to prevent occurrence of dangerous situations or to manage them safely if they do occur. It is well to arrange work with high energy materials so that it will be completed during the first six hours of the working day. The worker with high energy materials is strongly advised to disqualify himself during periods of mental or physical fatigue. This also applies to periods of mental stress or distraction.

TRAINING BY SUPER VISOR

The supervisor must advise and train his men to become innately aware of the hazardous nature of their work. To this end, he shall arrange fire drills and safety education on a periodic basis. He shall observe and review his employees with regard to their sharp-mindedness and emotional stability, and immediately remove those employees not fitted to their work environment. He shall constantly remind his men that each individual is responsible to himself and to his fellow worker.

Chapter 5 EXPLOSIVES IN THE LABORATORY

GENERAL

The information and directives in this chapter are for the training and guidance of those individuals who are concerned with explosives in the laboratory. Because of the use of chemicals to produce explosives, and because of the general use of glass in the laboratory, this chapter includes safety precautions pertaining to chemicals and glass.

DESIGN CRITERIA FOR EXPERIMENTAL ASSEMBLIES

It is recommended that the following factors be considered and used in the planning stages of the laboratory and its assemblies when performing research work with explosives.

OBJECTIVE AND SCOPE. Apply a great deal of thought to the problem assignment as to its objective, scope, and limitations.

FREE AND OPEN DISCUSSION. Discuss the problem assignment with other persons familiar with the operation and the hazards involved.

SIZE OF OPERATION. Hold the size of the operation to a minimum.

LOCATION FACTORS. Consider the following factors before deciding where to set up the operation.

Toxicity. Be sure that there is adequate protective equipment in the area to protect against a toxic reaction.

Explosive Properties. Be sure that the work area is large enough to contain the explosive force, or that effective venting in a safe direction is provided, should an explosion occur.

Flammability. Avoid the use of flammable materials in the area.

Pressure. Be sure that piping can withstand the estimated pressures.

Special Utilities. Use special utilities as required.

Other Work. Give special consideration to other work performed in an area under consideration.

Egress. Locate the equipment so that it does not obstruct escape from the area.

DESIGN SIMPLICITY. Design the equipment as simply as possible to perform the objective.

SAFETY OBJECTIVES

To establish and maintain a safe working area, the following considerations shall be taken into account during design.

HAZARDS. Consider the hazards involved in the release of toxic, flammable, or corrosive materials. Make provision for the most extreme manifestation of any and all hazardous events. Provide for positive termination of reactions by dumping of chemicals and mixtures.

PROTECTIVE EQUIPMENT. Exercise good judgment in the selection of pressure releases, guards, screens, splash pans, fire-fighting equipment, and signs to protect the operators, men, and equipment in the area.

ACCESSIBILITY OF CONTROLS. Be sure that access to controls is not obstructed by guards, screens, hot pipes, or apparatus.

MATERIALS. Use adequate materials in construction.

SUPPORTS. Use adequate supports to avoid vibration and strains on glassware.

LUBRICANTS. Use compatible packing, gaskets, and lubricants that cannot start hot spots or contaminate the product during operation.

APPARATUS ASSEMBLY. Observe the prescribed precautions in handling glassware. Provide supports and ladders as required. Provide protective devices for other workers and apparatus in the area.

INSTALLATION REVIEW. Review the completed installation with the supervisor in charge. Consider the advisability of an inspection. If the set-up is unusual or complicated, or if the set-up introduces new principles which might cause unknown or unforeseen hazards, make tests and/or dummy runs.

OPERATOR GUIDANCE. Show the set-up to the operators, and explain the reasons for the necessary safety precautions.

CHANGE IN SCALE. Review the installation if changes in scale or method of operation are contemplated to preserve the essentials of safety and simplicity.

DISM ANTLING PROCEDURE. Thoroughly analyze the dismantling procedure. Consider hazardous chemical disposition, pressure release, and vacuum relief. (Sudden air intake into the system can be hazardous.)

GENERAL SAFETY PRECAUTIONS FOR CHEMICAL MATERIALS

The following paragraphs deal with the safety precautions which must be observed and the hazardous elements involved in the handling and storage of peroxidizable solvents, oxidizing agents, chlorohydrocarbons, and other common chemicals.

PEROXIDIZABLE SOLVENTS. Peroxidizable solvents should not be used in work with explosives. However, if their use cannot be avoided, peroxide concentration must be prevented, because it can initiate detonation. Peroxides, forming in common laboratory solvents, have caused frequent laboratory explosions. These formations have occurred during distillation and during equipment dismantling after distillation. The rate of formation of peroxides is very rapid and is increased by exposure to light, heat, and air. A detectable amount of peroxide has occurred in cyclohexene within one hour after distillation from sodium. It was stored in a glass-stoppered bottle.

Table 4 contains examples of ethers and hydrocarbons that peroxidize easily.

Table 4

ETHERS AND HYDROCARBONS

Ethers	Hydrocarbons
Diethylene glycol dimethyl ether Diethyl ether Diisopropyl ether Dioxane Ethylene glycol dimethyl ether Tetrahydrofuran	Alkyl-substituted cycloaliphatics Branched-chain saturated hydrocarbons Cyclohexene Decahydronaphthalene (Decalin) Tetrahydronaphthalene (Tetralin)

PRECAUTIONS. Observe the following precautions in handling and storage of peroxidizable solvents. The three protective measures which must be taken are: (1) operators shall remain behind adequate shielding, (2) they shall perform the operation by remote control, and (3) the reaction scale shall be limited to the smallest practicable quantity.

Protection and Blanketing. Protect peroxidizable solvents from light, heat, and air during storage. Use well-closed containers and blanket with nitrogen.

Container Marking. Store these materials in containers with adherent tape. "WARNING - May Contain Explosive Peroxides" must be imprinted on the tape.

Container Dates. The containers shall be dated: (1) upon receipt from vendor, (2) when first opened, and (3) after removal of peroxides and storage under nitrogen.

Test Procedure. Test the solvent for peroxide content as described in DETECTION OF PEROXIDES, as follows:

Maximum Storage Time. Do not keep peroxidizable solvents that have been exposed to air for longer than six months. At the end of this period, they should be discarded or treated to remove peroxides. See <u>REMOVAL OF</u> PEROXIDES, this page.

Distillation. Do not allow air to come into contact with the hot solvent.

DETECTION OF PEROXIDES. The following two methods are presented as methods to detect peroxides. Proceed in the following manner:

1. Dissolve a sample glacial acetic acid.

2. Add a few drops of a ten- to fifteen-percent solution of sodium iodide in acetone.

3. Look for color development.

4. A yellow color indicates a low concentration; a brown color indicates a high concentration in the sample.

Or proceed in the following manner:

1. Add some of the sample to an acidified five- to ten-percent aqueous solution of sodium or potassium iodide.

2. Agitate vigorously if the material does not dissolve.

3. Add a starch solution to the aqueous phase.

4. If a blue color develops, peroxides are present. (If the peroxide concentration is high, a yellow color may develop before the starch is added.)

<u>REMOVAL OF PEROXIDES</u>. There are several established laboratory procedures for removing peroxides from solvents. Whichever is used, the treated solvent should always be tested to check the effectiveness of the treatment. One of the following four methods may be used.

1. Peroxides can be removed conveniently by passing the solvent through a short column of ordinary activated alumina. This method is effective for both water-insoluble solvents and water-soluble solvents (except for lower alcohols). It has the advantage that no water is introduced into the solvent, and small amounts of water that may be present are removed. When not even traces of peroxide can be tolerated, it is convenient to have the solvent flow directly into the reaction vessel from the alumina column.

Use care in disposing of the activated alumina after this operation. Experiments with peroxidized cyclohexene and dioxane indicate that at least these peroxides are decomposed on the alumina rather than simply absorbed. It is recommended that alumina, used for peroxide removal, be flushed with or dumped into a dilute acid solution of potassium iodide or ferrous sulfate. 2. Peroxide impurities in water-insoluble solvents (ether and hydrocarbons) are easily removed by shaking with a concentrated solution of a ferrous salt. A frequently used ferrous salt solution is prepared either from 60 g of ferrous sulfate, six ml. of concentrated sulfuric acid, and 110 ml. of water or from 100 g of ferrous sulfate, 42 ml. of concentrated hydrochloric acid, and 85 ml. of water. With some ethers, traces of aldehydes are produced by this treatment. If ether of a high degree of purity is required, it should be further shaken with 0.5 percent potassium permanganate solution, then with five percent sodium hydroxide solution, and finally with water.

3. One of the oldest methods of peroxide removal is refluxing the peroxidecontaining solvent over metallic sodium, followed by direct distillation. Recently, sodium hydride and lithium aluminum hydride have become popular for this use. Peroxide impurities are safely reduced to alchohols which remain behind as insoluble alcoholates during distillation. This procedure can be used generally for hydrocarbons and ethers.

WARNING

A serious explosion can result when halogen-containing solvents (carbon tetrachloride, chloroform, "Perclene," and "Triclene") are heated with strong reducing agents such as free metals or their hydrides.

Organic reagents such as alcohols, phenols, and acids, which contain acidic hydrogen atoms that react with sodium, cannot be purified by this method.

4. Where practicable, an inhibiting agent should be used to prevent the formation of peroxides. Example: five percent alcohol in ether.

OXIDIZING AGENTS. By their very nature, oxidizing agents are hazardous compounds with respect to explosions, fires, and burns. Some are poisons and allergens. The following list is not complete, but it does classify the oxidizing agents according to their primary hazards.

WARNING

Perchloric acid and perchlorates are especially dangerous. Because of their sensitivity and tremendous potential destructive power, special permission from a supervisor must be secured before working with them.

CHEMICALS WHICH CAUSE FIRES, EXPLOSIONS, AND BURNS. Handle nitrates, chlorates, and any other compounds of oxidizing acids with care when mixing with reducing agents and combustible material. Systems which contain an oxidizing acid or salt with active metals or organic matter constitute a potential explosive. Mixtures with salts of oxidizing acids are particularly dangerous in the anhydrous state and if subjected to heat or strong acids. Common laboratory cleaning solutions and nitrating mixtures are good examples. These solutions and mixtures are presented in Table 5.

WARNING

To avoid fires, explosions, and burns, handle volatile oxidizing agents with care. Avoid high temperatures and high concentrations. Clean up any spilled oxidizing agent promptly.

Table 5

CHEMICALS WHICH CAUSE FIRES, EXPLOSIONS, AND BURNS

Oxidizing Acids and Their Salts	Volatile Chemicals	Solid Chemicals
Iodates Periodates Bromates Hyprochlorites Chlorites Chlorates Perchlorates Nitrites Nitrates Permanganates Chromates Salts w/reducing or Catalytic Ions	Ozone Oxygen Halogens Interhalogen compounds Cyanohalogen Compounds w/nitrogen-halogen bonds Compounds w/oxygen-halogen bonds	Phosphorous Higher-valence Compounds of: Tellurium Mercury Thallium Lead Arsenic Vanadium Selenium

CHLOROHYDROCARBONS. The chlorohydrocarbons have low flammability, but their toxicity is their principal hazard.

Flammability. Chlorohydrocarbons in wide commercial use vary in flammability as follows:

Methyl chloride is moderately flammable with a moderate explosion hazard. Methylene chloride is practically non-flammable at ordinary temperatures. Trichloroethylene is non-flammable at ordinary temperatures. Carbon tetrachloride and perchlorethylene are non-flammable.

Chloroform, methylene chloride, and trichloroethylene form weakly combustible mixtures at high temperatures with high concentrations of solvent vapor in air.

<u>Toxicity</u>. The principal hazard in the use of chlorohydrocarbons is their toxicity. Generally, humans should not be exposed to an atmosphere in which chlorohydrocarbon odor is detectable. However, the chlorohydrocarbons do vary considerably in toxicity. A table of threshold limit values is defined as the maximum concentration in air to which humans may be subjected over a long period of time without undue discomfort. Table 6 cites some of these values in comparison with other solvents in common laboratory use.

Table 6

Solvents	Parts per Million (by volume)
Acetone	1000
Benzene	25
Carbon tetrachloride	25
Chloroform	50
Diozane	100
Ethylene dichloride	100
Ethyl acrylate	25
Ethyl alcohol	1000
n-Hexane	500
Methyl alcohol	200
Methyl chloride	100
Methyl chloroform	500
Methylene chloride	500
Pentachloroethane	5
Perchloroethylene	200
Propylene dichloride	75
iso-propyl alcohol	-100
Tetrachloroethane	5
Toluene	200
Trichloroethylene	200

THRESHOLD LIMIT VALUES (APRIL 1960)¹

Maximum concentration values have also been established by State health authorities.

The following WARNINGS are presented to guard the employee against inhalation of chlorohydrocarbons.

WARNING

The first evidence of the physiological action of the chlorohydrocarbons is a narcotic effect. Symptoms of excessive exposure are headache, undue fatigue, or nausea. Immediate access to fresh air rapidly eliminates the above symptoms from the body. However, repeated exposure may produce delayed or cumulative physiological effects. Liver damage from carbon tetrachloride has been reported.

WARNING

Pay attention to the low allowable concentrations for tetrachloroethane (5 ppm) and pentachloroethane (5 ppm). The maximum allowable concentrations for these compounds are lower than hydrogen sulfide and hydrogen cyanide.

WARNING

The use of trichloroethylene-dry ice freezing mixture shall be conducted from behind an efficient hood. Do not allow the mixture to stand in an open container between uses.

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The following WARNING pertains to the absorption of the chlorohydrocarbons through the skin.

WARNING

Avoid skin contact with both liquid and vapor chlorohydrocarbons. Absorption through the pores can produce the same symptoms as inhalation. Removal of the skin's natural oils can cause inflammation. Avoid vapor contact with the eyes.

<u>Stability</u>. There are no hazards in the handling of commercial chlorohydrocarbons under ordinary use conditions. However, on exposure to open flames or open electric heaters, the chlorohydrocarbons can decompose to form toxic and corrosive products.

Reactivity. The chloroacetylenes are highly explosive.

WARNING

Do not add sodium, caustic, or caustic solutions to 1, 2-dichloroethylene or to trichloroethylene; these may form monochloroacetylene and dichloroacetylene, respectively. Also, the addition of sodium, caustic, or caustic solutions to tetrachloroethane may form either of the above chloroacetylenes. Avoid the contact of caustic with the above chlorohydrocarbons except under carefully controlled experimental conditions.

OTHER COMMON CHEMICALS. The handling hazards of some of the more dangerous chemicals have been described in the foregoing paragraphs. But various types of hazards arise when other chemicals are not handled correctly. Although it is beyond the scope of this manual to include all of these chemicals, the following paragraphs set forth the general safety precautions which must be observed with acids, alkalies, mercury, dry ice, and ethyl ether.

<u>Acids.</u> Do not handle mineral acids carelessly. They are very corrosive and capable of causing serious injury or death. Follow these precautionary measures in handling these materials. -

1. Wear goggles when pouring acids.

2. Avoid acid contact with skin. If spilled on skin, wash immediately with copious amounts of water. Report any evident burn to the doctor immediately.

3. Clean up any spillage on floors or benches immediately by flushing with water, dilute sodium carbonate, or dilute bicarbonate. Spread generous quantities of ground limestone on spilled acid. 4. Do not mop up acid spills with cloths, sawdust, or any other organic materials.

5. Pour sulfuric acid and other concentrates into water. Never pour water into the concentrate.

6. Avoid inhaling acid fumes.

Alkalies. Follow these precautionary measures when handling alkalies in solid or solution:

1. Wear goggles when handling alkalies.

2. Avoid alkali contact with skin or eyes. Should the skin or eyes become affected with alkalies, wash the area immediately with generous amounts of water. Report to the doctor immediately.

3. Use cold water and wear the necessary protective equipment. Avoid boiling and spattering by pouring flake or granular alkalies slowly in small portions.

4. Do not leave any spillage unattended; flush it away quickly. Use a shovel to remove any solid spillage. Use dilute acetic acid to neutralize any remaining traces of the material.

5. Avoid inhalation of ammonia vapor. If it is necessary to work in an area with a high concentration of ammonia, wear a Chemox breathing apparatus or an eight-minute oxygen demand mask that covers the eyes.

Mercury. Observe the following precautions for storage and handling of mercury and its compounds:

1. Store mercury and its compounds in a cool, well-ventilated location at a safe distance from any acute fire hazard area. Keep all containers closed and plainly labeled.

2. Do not heat mercury or its compounds in an open area. Perform this operation only under a fume hood.

3. Clean up any mercury spills immediately. If this cannot be done, wet the area with water or sprinkle calcium sulfide on the area until the spillage can be cleaned up.

4. Pick up droplets of mercury with a small steel pipe or glass pipette. The pipe or pipette can be connected to a suction flask by small-bore rubber tubing, or devices for picking up mercury are available commercially.

Dry Ice. Dry ice, or solid carbon dioxide, will turn into gaseous carbon dioxide at ordinary temperatures. High concentrations of gaseous carbon

dioxide can paralyze the respiratory center and cause asphyxiation. Observe the following precautions for handling and storage of dry ice.

1. Wear dry cotton gloves when handling dry ice. Do not allow dry ice to contact any part of the body, because of danger of frostbite.

2. Wear goggles and be extremely careful when breaking large pieces of dry ice. Do not allow flying pieces to endanger others in the area.

3. Never store dry ice in gas-tight containers.

4. To prepare mixtures of dry ice and trichloroethylene, wear goggles and add the dry ice slowly to control the rate of gas evolution and to avoid spattering.

Ethyl Ether. The greatest hazards in the handling of ethyl ether are fire and explosion. It is a highly flammable liquid with a flash point of -45 degrees C. Its explosive range is 1.85 percent to 36.5 percent by volume in air. Ether, although not very poisonous, can produce anaesthesia, narcosis, and intoxication. Ether may become explosive if a distillation of ether containing ethyl peroxide concentrates the peroxide. Observe the following precautions in heating or distilling ether.

1. Test ether for peroxides before distillation. See foregoing paragraphs DETECTION OF PEROXIDES and REMOVAL OF PEROXIDES.

2. Heat or distill preparations containing ether only over a steam or hotwater bath with an unexposed heating element. Perform this operation under a fume hood.

3. Do not place materials containing ether in electrically heated ovens.

4. Use a non-flammable solvent such as methylene chloride for extraction procedures.

SAFE HANDLING OF FLAMMABLE LIQUIDS. By the open cup test, all combustible liquids with flash points of 80 degrees F., or lower, are flammable by I.C.C. definition. It can be safely assumed that any combustible compound which boils at 150 degrees C. (302 degrees F.) or higher will have a flash point considerably above 80 degrees F. In order to reduce the flammable liquids' fire and explosion hazards, the following precautions have been established. They must be observed when storing and handling flammable liquids in the laboratory.

1. The maximum volume of flammable liquid shall be six quarts per laboratory chemist, not including waste solvents.

2. The maximum volume of flammable waste solvent shall be one gallon per laboratory chemist. Flammable waste solvent shall be stored in a safety can. 3. The maximum capacity storage bottle for flammable liquids shall be one quart.

4. The metal safety can or polyethylene bottle for storage of flammable liquids shall have a capacity of one gallon. These are the only types of greater-than-one-quart containers to be used in storing flammable liquids in the laboratory.

5. The research supervisor must approve exceptions to the above limits. In approving any exceptions, the research supervisor must invoke special precautionary measures. These measures include prohibition of smoking at all areas in the laboratory, the addition of extra fire extinguishers, and the use of metal containers under or around large glass containers.

Flammable liquids not in use and exceeding the above quantity limits shall be stored in the areas especially equipped for this purpose.

To avoid ignition in case of spillage, use steam or hot water to heat flammable liquids, wherever possible. When heating flammables in glass containers over flame or electric element, place a metal catch pan large enough to hold the container's contents underneath.

Waste flammables in liquid form are to be collected in a properly labeled can provided for this specific use. Do not pour flammable liquids immiscible with water into sinks or sewers. Dispose of miscible flammable liquids by first mixing them with enough water to make them non-flammable. Use at least ten parts of water to one part flammable liquid; then flush with water.

STORAGE OF CHEMICALS. Containers of stored chemicals must be clearly labeled to show their identity. The following rules shall be observed in the storage of chemicals.

1. Keep the quantities of chemicals to a minimum consistent with work needs.

2. Make certain that excess quantities of chemicals are stored in an isolated location.

3. At the termination of a job, place all chemicals used in excess-chemical storage. Retain any reference samples.

4. Check retained samples at regular intervals.

5. Discard samples when no longer needed.

6. Discard samples if they show evidence of decomposition.

7. Consider the chemical's properties (stability, toxicity, and flammability), its frequency of use, and the quantity involved to determine location and method of storage.

<u>REFRIGERATED STORAGE</u>. Each chemist shall determine whether a chemical can be stored at room temperature or must be refrigerated.

EXPLOSIVE OR VIOLENT REACTIONS

The following paragraphs describe the general nature of an explosive or violent reaction. As a class, high-energy substances are capable of exothermic reaction. But potential violent reaction or explosion in the laboratory can be forecast. Experimenting can be safe, even with an explosion. Any personal injury from explosive or violent reactions must be attributed to lack of foresight and forethought.

FORECASTING VIOLENT REACTION OR EXPLOSION DANGER. Exothermic reactions can self-accelerate; reaction rate goes up with temperature, and temperature increases as reaction proceeds. Also, a reaction which makes gaseous products can become violent unless adequate facilities are present to disengage and vent the gaseous products. Explosive reactions are exceptionally violent reactions with almost instantaneous release of heat, gas, or both. This occurs throughout a quantity of material.

If a compound or mixture can decompose to form gaseous products, or if this decomposition is quite exothermic, the material is a potential explosive. Only to a very limited extent can the laboratory rely on supposed low sensitivity. The following paragraphs contain means by which possible explosive nature may be determined.

Heats of Reaction. Calculation of heats of reaction is helpful in forecasting danger and explosive hazards. Consider all possible reactions, not just the desired reaction. This is not difficult, in that the products are usually simple (water, carbon dioxide, carbon monoxide, nitrogen, carbon, and copper). This is not true in all cases, notably some of the organic peroxides and polymerizable organic compounds.

Heats of Combustion. When heats of reaction are not available, they can be obtained by measuring heats of combustion, or they can be approximated from bond energies.

Chemical Structure. Knowledge of chemical structure is helpful in the recognition of potentially explosive organic compounds. The following groupings tend to confer explosive characteristics:

 $-ONO_2$, $-NO_2$, $-NO_2$, -N = N-, -O-O-, $-N_3$, $-C \equiv C-$.

Two or more of these groups in a molecule usually make the compound potentially much more dangerous. Compounds with enough oxygen to oxidize most of the oxidizable elements are likely to be very dangerous.

Combine your experience, other persons' experience, and analogous reasoning to help you in recognizing dangerous materials. Memorize lists of hazardous compounds, especially those items which you have not found

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to be dangerous by your own experience and your studies. Even when rereagents and main products are safe, do not overlook the possibilities of explosive intermediates, by-products, or residues. For example, peroxide formation can occur in diethyl ether and tetrahydrofuran.

WARNING

Many dangerous reactions are described in public literature without a word of caution. Hence, literature precedent is a poor basis for disregarding any known indication of hazard. On the other hand, hazards of specific compounds can be found in library searches.

RECOMMENDED PROCEDURES FOR POTENTIALLY VIOLENT REACTIONS

Many reactions with large heat or gas release are potentially violent (but not explosive) because the materials react very rapidly during mixing, and no dangerous quantities of un-reacted mixture are built up. The following procedures are to be applied to prevent a violent reaction.

1. Protect yourself and others in the experiment area with shielding and adequate personal protective equipment (gloves, goggles, and aprons).

2. Take the necessary fire precautions should the reaction get out of control.

3. Keep the first run on a very small scale, unless the specific reaction is well known.

4. Do not consider a larger-scale reaction until the small-scale reaction is found to proceed promptly and smoothly.

5. Heed any induction period, or delayed reaction, as a danger signal which can classify the reaction as one of an explosive nature.

6. Keep reaction control measures simple. These control measures shall include gradual addition of reactants, effective stirring, proper heat dissipation, gas venting, and temperature indication.

7. Provide means for quenching the reaction.

RECOMMENDED PROCEDURES FOR POTENTIALLY EXPLOSIVE REACTIONS

Review all plans with your supervisor before starting any reaction that may be explosive, may yield explosive products, or may yield explosive by-products. Before starting a reaction, distillation, or any other operation, think through the whole procedure and make adequate preparation for safe handling of potentially explosive products or residues. The following procedures are to be applied in preventing an explosive reaction.

1. Limit the scale of the reaction to the smallest practicable quantity. If you are assigned the first experiment of its kind, the quantity of the potentially

explosive material shall be limited to one-hundredth mole. In no case shall the quantity be more than one-tenth-mole. Micro-analytical techniques can help in drawing valid conclusions from this small-scale work.

2. Shield all workers in the work area from direct-line missile hazards. For the above scale, use one-quarter inch standard safety glass (not tempered glass), one-quarter inch lucite, or one-sixteenth inch mild steel.

3. Maintain at least a ten-inch distance between the shield and the reactant to prevent high-velocity fragmentation of the shield.

4. If feasible, conduct all operations by remote control. Do NOT reach around the shield.

5. Keep adequate supplies of a diluting solvent close at hand to render the material non-explosive. However, bear in mind the possibility of dangerous concentration of the material by fractionization or separation of liquid phases.

6. Use dry ice or liquid nitrogen to cool unstable explosive materials. However, bear in mind that these coolants may not prevent explosion when used with some compounds.

7. Regard gas mixtures in the non-flammable range as explosive materials. They can become explosive by incorrect mixing.

8. Do not assume any absence of hazard when a small-scale experiment is successfully completed.

USE OF GLASS IN THE LABORATORY

The misuse of glassware in the laboratory can result in painful and severe injury. These injuries can be averted by proper choice, inspection, and handling.

CHOICE OF GLASS. Hard glass (Pyrex, Vycor, or Kinnex) is usually best suited for general laboratory use.

<u>INSPECTION OF GLASSWARE BEFORE USE</u>. The strength of glass is directly related to its surface condition. The following inspection procedures are recommended.

1. Do not use glass which is cracked or severely scratched.

2. Inspect glassware for pin holes, sharp edges, paper-thin areas, badly fitting joints or plugs, and any dangerous contamination.

3. Discard and replace damaged or defective glassware immediately.

4. Follow the advice of a glass blower.

WARNING

Never ask the glass blower to repair contaminated equipment.

HANDLING LABORATORY GLASSWARE. The following procedures are recommended in handling laboratory glass tubing and rods.

1. Wear leather gloves.

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2. Fire polish or grind the sharp edges of all glassware.

3. Where available, use a strong, heavy-wall, hard-glass tubing in 15 mm or lower sizes.

4. When breaking glass tubes or rods up to 20 mm O.D., use a flat mill file to apply a single transverse scratch at the desired breaking point.

5. Grasp the tubing or rod firmly so that the scratch is between the hands and opposite the thumbs.

6. Bend the tubing or rod at the scratch to create tension on the scratched side and, at the same time, pull the hands apart.

7. If the rod or tubing is too short to permit a firm grasp, use hard plastic tubing or cork borers to extend the length of the segment.

If the glass tubing or rod is more than 20 mm in diameter, or if it is connected to a fixed object, proceed as follows:

1. Scratch the tubing or rod with a flat mill file as above.

2. Touch with a white-hot piece of glass at the center of the scratch; then wet immediately with water.

3. Consult a glass blower before cutting larger-gauge tubing and rods with a hot-wire technique.

RUBBER-TO-PLASTIC CONNECTIONS. When glass tubing, rods, or thermometers are inserted into holes in stoppers, rubber tubing, or plastic tubing, the glass may break leaving sharp, jagged edges to inflict severe cuts. Proceed as follows:

1. Do not try to FORCE any glass tube, rod, or thermometer through an opening.

2. Insert types of glass equipment in cork and rubber stoppers by holding the stopper firmly with thumb and forefinger. Keep the palm of the hand away from the opposite face of the stopper.

3. Grasp the glass firmly as close as possible to the stopper, and insert with a series of twists, pressing gently with each twist.

4. Use a lubricant. Water, glycerol, silicone stopcock grease, or powdered graphite is recommended.

5. If equipment salvage is necessary, and the equipment is frozen, cut the rubber or plastic material away from the glass.

GROUND-GLASS JOINTS, STOPCOCKS, AND GLASS STOPPERS. These ground glass connections must be clean and lubricated to make them liquidand vapor-tight and to prevent "freezing." The following procedures are recommended for these items.

<u>Ground-Glass Joints.</u> Observe the following procedures for ground-glass joints:

1. Use spring devices to secure ground-glass joints.

2. Free frozen ground-glass connections as follows.

a. Wear leather gloves.

b. Apply hot water or hot air to the outside surface and free by gently twisting.

3. Insert a piece of paper between the surfaces of the joint when not in use.

4. Keep all ground-glass and/or threaded joints void of sensitive materials.

Stopcocks. Observe the following procedures for stopcocks:

1. Secure stopcocks with standard retainers.

2. Use a mechanical opener to free frozen stopcocks.

3. Consult the glass blower in extreme cases.

Glass Stoppers. Observe the following procedures for glass stoppers:

1. Keep glass stoppers well-lubricated.

2. Insert a piece of paper between the stopper and neck of the container when not in use.

TEST TUBES, FLASKS, AND BEAKERS. Observe the following precautions.

1. Bear in mind the strength of glass when inserting stoppers in test tube and flask mouths.

2. Bear in mind the strength of glass when lifting or supporting heavy flasks and beakers.

3. If beaker or flask size is one liter or more, use bath or electrical heating instead of direct flame.

4. Flasks and tubes are often used under high-vacuum conditions. The operator shall use a hood, wire screen, or other properly designed piece of equipment. The flat surfaces shall be of minimum area, and the equipment shall be of sufficient thickness to withstand one atmosphere of pressure.

GENERAL SAFETY REGULATIONS FOR DEVELOPMENTAL LOADING

The following general safety regulations apply to developmental loadings of high explosives. The safety regulations in this chapter are not meant to be all-encompassing. Constant change in technology and its application to new materials and equipment necessitate that all proposed operations be examined very carefully for potential hazards. Only after careful hazard examination can a complete safety instruction be prepared. Mixing and handling hazards are usually easily defined and controlled. The hazards attributable to the chemical and physical nature of the explosive are more difficult to analyze. Impact and friction sensitivity data, vacuum stability information, and other test data must be obtained prior to beginning any work. This information must be well known and understood by supervisors before beginning work. Never depend on laboratory-scale data, until this data has been substantiated by tests of plant-produced material.

UNDERSTAND THE HAZARDS. The supervisor and his operators shall:

1. Analyze the possible hazards of every operation;

2. Know the nature of all work being done;

3. Know the exact properties of all materials handled; and

4. Know how to avoid expected hazards.

KEEP HAZARDS TO A MINIMUM. The supervisor and his operators shall keep hazards to a minimum by:

1. Keeping volume of explosives handled, the number of persons and equipment exposed, and exposure time to a minimum.

2. Observing all posted limits of persons and equipment in a given area.

3. Observing personal cleanliness at all times.

4. Wearing gloves and protective clothing as operations require.

5. Washing hands and face before all intermissions, smoking, and meals.

6. taking showers at the end of the working day if toxic explosives or materials are used in operations.

CONSTANT INDIVIDUAL CARE. The operator shall care for himself constantly in the following manner:

1. Form correct work habits.

2. Be on the alert for hazardous conditions.

3. Never assume that the proper safety precautions have been taken; make sure they have been exercised.

HOUSEKEEPING. Supervisors and operators shall maintain good housekeeping standards in the following manner:

1. Conduct loading operations in a neat and orderly manner.

2. Conduct clean-up operations at the end of each day and a complete cleaning operation each weekend to remove explosive dust from floors, walls, ceilings, ledges, and tables.

3. Maintain effective inspections in the loading areas.

4. Perform complete washdowns of plant and equipment at 90-day intervals.

5. Perform periodic clean-ups of duct and ventilator interiors, overhead pipe, auxiliary equipment, and bearings.

6. Be particularly attentive to areas where explosives are exposed to heat and friction between moving surfaces.

STORAGE RECEPTACLES. Operators shall maintain storage receptacles in the following manner:

1. Keep explosives and explosive parts in approved, covered receptacles with covers intact.

2. Maintain adequate grounding for receptacles by connections to wellgrounded wires or by placing the receptacles on well-grounded metal plates.

3. Avoid unnecessary transportation and handling of explosives.

PROTECTION FROM ELECTROSTATIC CHARGES. The operators shall protect themselves from electro-static charges in the following manner:

1. Ground all machinery, equipment, and fixtures.

2. Use grounded conductive coverings for floors, work benches, and tables.

3. Wear semi-conductive safety shoes and cotton clothing.

4. Make periodic resistance measurements to insure that wires, lightning rods, and conductive floors are within prescribed limits.

PROTECTION OF EXPLOSIVE FROM PHYSICAL SHOCK. The operator must protect the explosive from physical shock in the following manner:

1. Avoid dropping explosives.

2. Conform to specified unit and weight limits when handling explosives with cranes, dollies, or other handling gear.

SAFETY EQUIPMENT. The supervisor shall enforce the use of conductive shoes, goggles, gloves, respirators, hard hats, and impregnated work clothes as necessary to protect employees from burns, poisoning, and associated industrial-type hazards.

REMOVAL, HANDLING, AND STORAGE OF EXPLOSIVE SCRAP.

Removal. Proceed in the following manner:

1. Flush explosive scrap to the drainage system with water.

2. Empty the catch basin at regular intervals to prevent accumulation of explosive scrap. Use extreme care in this operation.

3. Do not allow the accumulation to dry.

4. Destroy the accumulation immediately.

5. If the accumulation cannot be immediately disposed of, measure the temperature to determine any heat evolution or decomposition. This measurement should be done remotely.

6. Be extremely careful of reactivity between components in the catch basin.

7. Be on the lookout for lack of oxygen and toxic gases in pits.

8. Use non-spark generating shovels when frictical sensitivity permits; otherwise, use wood or plastic tools.

Handling. If handling of explosive scrap is necessary, observe the following basic rules:

1. Do not allow a scrap explosive to be combined with another unless they have been proven to be compatible.

2. Keep the amounts of scrap from sensitive explosives or unknown sensitivity scrap to a minimum and dispose of them frequently.

3. The amount of explosive scrap must be included in the allowable amount of explosives in the work area. Remove the amount of scrap frequently so that it does not restrict operations. This will keep the amount of explosives present in the work area under the allowable limit.

4. Do not allow explosive scrap from another facility within the plant (for instance, a chemical laboratory) to enter the drain system for scrap from the loading operation.

5. Consider the possibility of rendering explosive scrap insensitive or inert by removal of water-soluble components.

6. In conjunction with the above rule, do NOT allow evaporative accumulation during disposal of the solution.

Storage. There are no simple rules for storage of many explosives. The user of this publication is instructed to observe storage instructions in OP 5; if a material is not included in OP 5, the storage of the material shall be in accordance with the following.

1. Avoid storage of potentially unstable material or material of questionable stability in working areas. Keep the material separate from explosive scrap.

2. Regard all new compositions with any potential instability as questionable, and maintain a direct temperature record of the material and the magazine until the material's stability has been established by experience.

3. Keep in mind that a slight change in components or a change in an ingredient's purity can cause the explosive system to become unstable.

4. Be extremely suspicious of systems which do not have the vacuum stability characteristics of Composition B and HBX.

5. Never store high-energy materials of unknown stability with other explosives.

6. Do not store powdered metals with explosives or oxidizers.

7. Do not store oxidizers (ammonium nitrate, ammonium perchlorate) with explosives.

8. Keep explosives and high-energy materials of low or unknown stability in isolation magazines under close surveillance. Provide means to measure their temperatures remotely.

9. Be alert during all operations for any unusual gas, fumes, or odors. The brown fumes of the nitric oxides, the odor of formaldehyde, or chlorine gas indicate a reaction which can go out of control. 1

10. Failure of a cooling system to keep the temperature below maximum permissible is a critical warning that the situation is getting out of control. Do not ignore this warning. If no action has been planned in advance, vacate the premises without delay.

11. When using solvents, keep in mind that the toxic, flammable, or explosive characteristic of the solvents may present a greater hazard than the explosive itself.

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Chapter 6

EXPLOSIVES IN THE PILOT PLANT

GENERAL

This chapter pertains to pilot plant explosives operations. Every operator who engages in pilot plant work with explosives shall be guided by Chapter 4 of this publication. Studies, check lists, and careful planning are MUSTS before pilot plants can proceed with larger-scale operations.

The original operators at the laboratory-scale level should be invited to the pilot-plant to review their work and point out the safety precautions which they consider applicable to the scaled-up operation to be performed by the pilot-plant.

SPECIAL HAZARDS OF LARGER SCALE OPERATIONS

The hazards involved in larger-scale operations are prevalent in the pilotplant or semi-works. The term semi-works includes facilities which range in size between a large laboratory and a commercial-scale facility. The semiworks facility performs single-process steps (or unit operations studies), or it may engage in a completely integrated pilot-plant operation. The pilotplant, its operations, and its size may be used to demonstrate an over-all process. The data gathered in the pilot-plant operation can be applied to the design of a commercial plant.

Semi-works research carries over most of the hazards inherent in a chemical process from the laboratory. In some cases, the hazards are magnified. For example, no apparent control problem may be necessary when a rapid exothermic reaction is performed on laboratory scale. When the process is assigned a semi-works facility, and the reaction is to be scaled up 100 or more times (two increases in scale by a factor of ten), the venting of a large volume of heat presents a serious problem to the safe and effective operation of the process. This is true because the increase in quantity of reactants and heat generators is greater than the increase in radiation area or the surface from which heat can be removed. Before any new chemical processing operation can be performed on a semi-works scale, the important physical and chemical properties of the material must be known. The knowledge of these properties will aid in the anticipation of potential operating hazards and in the selection of proper equipment designed to perform the operation safely.

The same engineering standards applied to plant-scale equipment shall be used in semi-works areas. Metal has a greater resistance to physical shock and greater heat conductivity. For this reason, metal equipment is used in the semi-works facility. No glass equipment may be used unless it has been designed for a specific operation. The physical state of the material in process must be monitored at all times. The operator must have advance warning of hazardous conditions. To this end, sight glasses, measuring instruments, control apparatus, relief devices, and other "tell-tale" systems must be provided with metal equipment. Avoid reliance on makeshift and untried measuring and control devices.

RULES AND PRACTICES

The following paragraphs set forth typical practices which must be carried out by the various members of a plant organization when a pilot-plant or semiworks operation is contemplated. Directives to personnel may be outlined by a committee selected from the representative groups within the plant organization. This committee should include a member of the safety office, the project manager, the supervising chemist, the plant engineer, and members of the construction and engineering groups as deemed necessary. Tasks assigned to the above personnel in the paragraphs below are recommendatory, and will vary with the particular facility.

SUPERVISING CHEMIST. It is recommended that the supervising chemist perform the following tasks.

- 1. Prepare a comprehensive flow sheet and indicate the following:
 - a. Locations of Equipment;
 - b. Piping and Valve Arrangements;
 - c. Pressure and Temperature Controls;
- d. Direction of Material Flow.
- 2. Submit the flow sheet to the project manager for his approval.

PLANT ENGINEER. It is recommended that the plant engineer perform the following tasks.

1. Review the flow sheet.

2. Advise if any other plant facilities may be endangered.

3. Consult with the supervising chemist.

4. Point out any hazardous condition arising from interruption of a vital plant facility.

<u>CONSTRUCTION GROUP.</u> It is recommended that the construction group perform the following tasks.

1. Inspect and test all newly purchased or fabricated units before installation.

2. Inspect and test all newly acquired safety devices.

3. Inspect, repair, and test any used equipment at periodic intervals to insure safe operation.

PROJECT MANAGER. It is recommended that the project manager perform the following tasks.

1. Prepare detailed operating and safety instructions with the flow sheet.

2. Include a simple description of the process.

3. Prepare emergency shut-down procedures.

4. List all hazards.

5. List precautions which must be observed.

6. Prepare for emergency by scheduling practice fire drills at regular intervals.

7. Use all safety equipment during practice drills.

COMMITTEE. It is recommended that the committee proceed as follows:

1. Organize an inspection team to check safety and engineering standards.

2. Review the flow sheet and operating instructions.

3. Request representative groups to advise any changes which would add to the safety factor of the over-all, larger-scale operation.

4. Make the necessary changes.

SUPERVISING CHEMIST. It is recommended that the supervising chemist perform the following tasks.

1. Use phantom or dummy runs to acquaint his operators with all hazardous conditions.

2. Train his operators to check the system's over-all performance and safety.

3. Set up a periodic schedule for the performance of steps 1 and 2 above.

4. Inspect and maintain all moving-parts equipment to prevent accidents from mechanical failure.

5. Start the pilot-plant reaction with great care.

6. Do not allow reactants to accumulate and accelerate out of control.

7. Have adequate controls to minimize explosive hazard.

8. Have suitable protection for his operators.

PILOT-PLANT OR SEMI-WORKS OPERATOR. The operator in the pilotplant shall proceed as follows.

1. Wear the proper personal protective safety equipment as prescribed by the supervising chemist or safety office.

2. Operate the equipment by remote control from behind a barricade if possible.

3. Stay behind the barricade until positive that the operation is proceeding as planned.

4. Monitor all valves, temperature indicators, and other control instrumentation at regular intervals to ascertain any deviation from expected process behavior.

5. At the slightest deviation in pressure or temperature, call the supervising chemist and alert other operators in your vicinity.

6. Follow your supervisor's instructions.

7. Man alarm systems, sprinkler systems, and sand buckets as directed by your supervisor.

8. Check to see that escape routes are clear.

9. Vacate the premises on order of your supervisor.

CONSTRUCTION GROUP. At the cessation of the pilot-plant operation, it is recommended that the construction group proceed as follows.

1. Clean and check the equipment thoroughly.

2. Dismantle the equipment.

3. Scrap or repair any damaged equipment.

4. Keep the equipment in reserve so that it might be used in another pilot-plant operation.

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SAFETY PRECAUTIONS FOR UNIT OPERATIONS AND PROCESSES

The following precautions must be observed in the preparation of samples for analysis.

SIZE REDUCTION. Observe the following precautions in obtaining small amounts of explosive compounds.

1. Use a mortar and pestle made of lignum vitae.

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2. Protect operators with a sufficient barricade when processing oxidizer materials.

MILLING. Observe the following precautions in milling:

1. Be certain that the material is free of contaminants and foreign materials.

2. Inspect equipment to determine that no lubricants can leak into the milling chamber.

3. Provide positive means to keep explosive materials out of contact with bearings.

4. Determine that grinding surfaces will not initiate fire or explosion. Use wooden, plastic, or rubber balls in ball mills where practicable.

5. Pulverize inert ingredients separately.

6. Do not attempt size reduction with a sensitive material containing perchlorate, explosive compositions containing nitroglycerine, or explosives containing RDX, HMX, or PETN.

7. Perform size reduction by recrystallization prior to formulation.

8. Perform milling on insensitive ingredients prior to formulation.

9. When advisable, use water or other inert disperant to decrease sensitivity during milling.

10. Prevent dispersion of explosive dust by use of dust collectors.

11. Provide the necessary equipment to prevent a fire hazard.

FILTRATION. Perform this operation in a building with automatic sprinkler fire protection. Observe the following precautions in filtration:

1. Provide operators with the required protective clothing and respirators to prevent injuries from burns, toxic gases, and flammable solvents.

2. If flammable solvents must be used with explosives, limit the operator's quantities of both.

3. Use acetone, alcohol, or other water-soluble solvents.

4. Use gravity flow when filtering explosives.

5. Do not use gear or piston pumps with metal working parts.

6. If pumping is necessary, use a diaphragm or similar pump with rubber or plastic parts.

7. Consider the development of a static charge caused by solvents and plastic parts.

8. Determine the compatibility of explosives with solvents and parts of equipment.

SIZE SEPARATION. Observe the following precautions in size separation;

1. When size separation is necessary, use a wet screening method, or other safe process.

2. Impose rigorous safety measures if an explosive in the sensitivity range of RDX or PETN must be dry screened. Use safe, remote, and barricaded facilities.

3. As a safety precaution when dry screening is necessary, coat the particles of explosive with graphite to minimize generation of static electricity.

CRYSTALLIZATION. Observe the following precautions in crystallization:

1. Use water-soluble solvents when possible.

2. Provide automatic sprinkler system protection with the use of flammable solvents.

3. Control toxic solvent concentration by selection of proper solvent and proper control of solvent vapors, splashes, and spills.

DRYING. Observe the following precautions in drying:

1. Determine whether a remote control operation is necessary.

2. Determine the effect of the drying operation on material sensitivity.

3. Determine that drying will not create a reactive material with possible decomposition.

4. Eliminate solvent vapors from the area.

5. Do not pulverize caked or agglomerated material except by a provensafe procedure.

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GENERAL SUMMATION. The following precautions are also presented to aid the pilot plant in unit operations and processes:

1. Remember that liquids, water included, will not always desensitise the solid explosives with which they are mixed. Never take for granted that a wet solid explosive is less sensitive than a dry explosive.

2. Avoid handling of dry, sensitive explosives and explosive formulations.

3. Establish an effective preinspection before beginning mixing or grinding operations.

4. Be sure that equipment installation complies with the National Electrical Code.

5. Be sure explosives are compatible with solvents.

6. Use toxic or flammable solvents only when necessary.

7. Use non-conductive containers for flammable solvents and explosives sensitive to static charges.

8. Keep oxidizers from organic materials, except when they are required in process.

9. Add the oxidizer to the formulation as a last step.

10. If maximum sensitivity is developed by the addition of the explosive, add it last.

SAFETY PRECAUTIONS FOR MIXING AND BLENDING

It should be pointed out here that these operations do not include any intentional particle size reduction other than a reduction caused by concomitant abrasion of the particles. For size-reduction information, see SAFETY PRECAUTIONS FOR UNIT OPERATIONS AND PROCESSES.

PROPER SELECTION OF MIXING EQUIPMENT. The sensitivities of explosives to impact, rubbing, friction, and other energy-imparting actions have a tremendous range during mixing and blending operations. Silver amide is useless during this operation. Because of its extreme sensitivity, silver amide may explode on the slightest disturbance.

Silver azide, mercuric fulminate, PETN, RDX, TNT, picric acid, and ammonium picrate having decreasing sensitivity ranges, in that order. Ammonium picrate can withstand "setback" in shell.

The mixing and blending equipment used on a given composition must minimize any mechanical action which can ignite or initiate explosion. Three types of mixing equipment are described in the following paragraphs.

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<u>"Jelly Bag" Mixer.</u> The "jelly bag" mixer consists of a bag made of soft dust-impervious material suspended by a rigid ring at its mouth. A rope is attached through the mouth of the bag to the bottom. The materials to be mixed are placed in the bag. The rope passes over suitable pulleys to an operator protected by a barricade. The operator can tumble the materials in the bag by alternately pulling and releasing the rope. When mixing is completed, the operator empties the bag by pulling the rope until the bag is inverted and turned inside out. The mixed material falls in a container placed under the bag. This equipment is suitable for mixing primary explosives.

"Twin Shell" Blenders. "Twin Shell" blenders are suitable for mixing explosives less sensitive than primary explosives.

Cylindrical Tumblers. Cylindrical tumblers are also suitable for mixing explosives. The tumblers rotate about axes placed at various angles to the axis of the cylinder.

Mixing and blending is a unit process in chemical engineering, and the discussion of equipment design and operation is beyond the scope of this publication.

Those persons who have a problem of designing and installing equipment for a specific application to explosives, pyrotechnics, or propellants should consult publications of manufacturers of mixing and blending machinery.

SAFETY PRECAUTIONS. Mixing and blending operations are categorized into two types. Safety precautions are listed here for those which may be safely performed without a barricade; then more stringent precautions are listed for mixing operations which require remote control.

No Barricade Protection Needed. Barricade protection is not required during the following operations. Before starting any mixing operation, observe the following precautions:

1. Insure that equipment is clean and in good working condition.

2. Be sure that agitator clearance is correct at maximum operating temperature.

3. Be sure that temperature recording and sensing instruments are operating correctly.

4. Use a suitable automatic control device to keep the heating fluid's temperature level below a level which would cause a thermal decomposition of the systems.

Charging Ingredients. Observe the following precautions before charging ingredients:

1. Preheat unit.

NOTE: Where possible, swirl breakers should be heated with steam or hot water.

2. Always add the materials which provide the liquid phase first.

3. Avoid chance of producing large lumps of explosive by controlling rate of heat and addition of ingredients. Wet solids with non-solvent, if one is to be used, before adding solvent.

4. Use extreme caution in addition of metals or any other material which could cause the batch to solidify due to the metals' high heat capacity or rapid heat conduction.

5. Avoid creation of dust.

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6. Handle RDX and explosives of similar sensitivity water-wet when possible. The water can be driven off later by extended heating, vacuum drying, or other suitable method.

Vacuum Treatment. Observe the following precautions during vacuum treatment operations:

1. Insure that drop valves prevent leakage of explosive onto working parts.

2. Keep vacuum lines clean. Inspect as necessary and install traps.

3. Be sure there is an effective filter between kettle and pump. Clean the filter as often as necessary.

4. Set up routine oil changes for pump and adhere to this schedule.

5. Make a daily visual inspection to insure that filter is operating.

NOTE: A liquids trap is not needed for Composition B, HBX explosives, TNT, tritonal, picratol, cyclotols, octols, and other conventional explosive systems.

6. Ascertain the need for a liquids trap in systems other than those listed above. Install the trap where vapor pressure, temperature, and reaction products show a need for it.

<u>REMOTE CONTROL MIXING OPERATIONS</u>. The following precautions must be observed to determine if a mixture or compound will require remote control operations:

1. Examine sensitivity data of the mixture and individual explosive ingredients. Examine accident data on similar systems, type of mixing operation; and possible reactivity. 2. Mix these systems by remote control until sensitivity, fire, and detonation tests show the process to be safe.

3. Use remote control with any formulation containing appreciable nitrocellulose, nitroglycerine, or explosive of similar sensitivity.

4. Use remote control to mix any mixture of an oxidizer (ammonium perchlorate, ammonium nitrate) and an organic explosive with or without a metal.

5. Mix any mixture with a sensitivity in the range of RDX by remote control.

EQUIPMENT. Operate the following mixing equipment by remote control:

1. Pan mixers.

2. Mixing rolls.

3. Kneaders.

4. Sigma blade mixers (vertical or horizontal type).

5. Make sure that mixer rotor blades have sufficient clearance, that close-fitting or rubbing parts are eliminated, and that bearings on glands are always outboard of the mixer.

6. Be sure clearances are sufficient to allow for dimensional changes brought about by load weight, temperature changes, or pressure changes.

7. Use well designed and well maintained mixers to avoid breakdown.

8. Install and use a shadow board to prevent misuse and misplacement of tools and fittings used in mixing, charging, or materials transfer operations.

9. Use a pilot-plant mix, not a laboratory mix, of all new explosives or explosive compounds under remote control conditions before determining that they are safe for processing without remote control.

SAFETY PRECAUTIONS FOR INSPECTION AND WEIGHING

All explosive materials and ingredients included in explosive compositions must be inspected for contaminating substances. If an effective visual inspection cannot be performed, the material must be screened.

INSPECTION. Observe the following safety precautions in screening:

1. Use a screen of a mesh just slightly larger than the size of the material to be inspected.

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2. Pass all materials over permanent magnets strong enough to remove any contamination of a ferrous metallic nature.

3. Use conductive containers fitted with hinged lids made of soft aluminum, strong fiber, or plastic compatible with explosives when storing a material sensitive to static discharge.

4. Avoid the creation of dust sensitive to static discharge.

WEIGHING. Observe the following safety precautions in weighing:

1. Use explosive in the sensitivity range of RDX water-wet.

2. Store and handle water-wet RDX, HMX, PETN, and similar hazardous explosives in seamless, conductive, containers made of plastic or rubber.

3. Set up the operating line so that the material, once prepared, is subject to a minimum of transfers.

4. If extended storage is necessary, restow under water as instructed in OP 5.

5. Take the necessary steps to insure that containers of the above materials are not dropped.

SAFETY PRECAUTIONS FOR PILOT-PLANT EXPLOSIVES LOADING OPERATIONS

In compliance with the <u>RULES AND PRACTICES</u> paragraphs in this chapter, the supervising chemist or the project manager shall include the following information with his flow sheets.

PREPARATION OF STANDARD OPERATING PROCEDURES AND INSTRUCTIONS. Standard operating procedures for explosives loading shall be written to include all available information about the chemical and engineering characteristics of the materials to be used.

Those operations which require remote control shall be explicitly pointed out to operating personnel with WARNINGS. It is recommended that standard operating procedures be prepared by the supervising chemist or project manager. The following provisions shall apply.

<u>Complete Unit Coverage</u>. Operating procedures will be prepared for each unit of process equipment.

Task and Operations Coverage. Operating procedures will be prepared for each task. Where several operations are involved and consist of many unrelated steps, the procedures will relate to the individual steps rather than the several operations. Whenever possible, a comprehensive instruction sheet applicable to several individual steps shall also be provided. However, every step assigned must have an operating procedure, and the operator may refer to the comprehensive instruction sheet. Safety Procedures. Safety procedures must be included for each step and operation.

WARNING

When procedures are released by a supervisor to his operators, there shall be no deviation from the procedures, except by written authorization of higher authority.

SAFETY PRECAUTIONS IN PREPARATION FOR LOADING

Explosive charge container assemblies must be designed to provide safety under expected loading, handling, and storage conditions.

MOLDS AND PRESSING DIES. Observe the following safety precautions.

1. Examine all molds and dies before use. Be sure the mold or die will not impart a frictional or pinching action against the explosive.

2. Select materials of construction design and surface finish which permit easy disassembly and easy remote control of the charge from the mold.

3. Be sure that materials of construction do not react chemically with explosives, form sensitive substances, nor form a soft or liquid phase at the container surface.

4. Be sure that molds and pressing dies are strong enough to insure no distortion under applicable loads.

5. If possible, use molds with venting holes and means to minimize fragmentation should an explosion occur in pressing operations.

SAFETY PRECAUTIONS FOR LOADING

The following safety precautions shall be observed during a loading operation.

ENVIRONMENT. The supervisor and his operators shall observe the following;

1. Maintain high housekeeping standards throughout all operations.

2. Clean all equipment thoroughly before each operation.

3. Use flexible conductive rubber buckets when they are necessary to transfer explosives.

4. Avoid spillage. Clean up spilled material immediately.

5. If the reactivity of the material permits, use wood or soft aluminum rods.

6. Provide all employees with personal protective gear as required, with mandatory instructions to wear the protective gear as required.

7. Keep number of employees, amount of explosive, and exposure time to a minimum.

8. Clean all equipment thoroughly after each operation.

VACUUM LOADING. See Vacuum Treatment under the preceding SAFETY PRECAUTIONS.

PRESSING. All pressing operations should be done by remote control with adequate shields and barricades. Proceed as follows:

1. Use epsom salts with two percent graphite to test the die above normal working pressure.

2. Perform ejection procedures by remote control with adequate shields and barricades provided. Wait at least one minute behind the barricade, because explosions have occurred during this operation.

3. Wear safety glasses, ear plugs, and hard hats to insure against injury.

4. Conduct periodic inspection of press parts to insure that they are within specified dimensional tolerances.

SAFETY PRECAUTIONS FOR OPERATIONS AFTER LOADING Observe the following precautions after loading operations:

1. Be sure that explosives do not contaminate fasteners before disassembly of molds.

2. Perform all machining, sawing, and shaping by remote control until experience, problem evaluation, and sensitivity data show that the operation need not be remotely controlled.

3. Perform any disassembly of explosive-loaded ordnance by remote control if the operation is hazardous.

4. Be extremely careful, and be certain that no explosive is crimped between riser and riser frames when they are removed.

SAFETY PRECAUTIONS FOR ASSEMBLY

The following safety precautions must be observed during assembly:

1. Perform all crimping operations by remote control.

2. Supervisors shall aid their operators to control contamination during assembly of cover plates and fuze wells. This contamination can be prevalent in all threaded fittings and openings subjected to extreme forces and friction.

3. Use a compatible grease to avoid contamination.

GENERAL SAFETY PRECAUTIONS FOR MATERIALS

1. When powdered metals are used as an explosive component, surface treatment of the metal may be necessary to reduce reactivity. For instance, the dichromating process will form a non-reactive coating on atomized aluminum.

2. Materials which are normally desensitizers of explosives can become sensitizing agents when used in other explosive systems. For instance, wax used as a desensitizer for HBX explosives will function as a sensitizer or explosive component when used with ammonium perchlorate.

WARNING

Do not apply information valid for one explosive system to another without careful study and testing.

3. As part of explosive assembly operations, the latest adhesives, cements, filters, epoxies and polyesters are being used. These materials can be very toxic, dangerously reactive, and are fire hazards.

Chapter 7 PYROTECHNICS IN THE LABORATORY

GENERAL

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Pyrotechnic compositions are physical mixtures of finely powdered chemical compounds and elements. When ignited, they can undergo combustion (chemical reaction) with the evolution of a considerable amount of heat and light in a relatively short period of time. Various organic binders and color intensifiers may be included in the mixtures. The reaction may be accompanied by smoke and/or sound.

The heat of reaction of pyrotechnic compositions may range from 200 to 2500 calories per gram. This amount of energy, while considerable and often extremely dangerous, is not generally released in as destructive a manner as the energy of explosives except in the cases of photoflash combinations, loose illuminant, and igniter compositions. The burning rates usually required of pressed pyrotechnic compositions are relatively low, and only a relatively small proportion of the reaction products are gaseous. This causes the released energy to be dissipated as heat and not converted to mechanical energy.

However, in the research and development of pyrotechnic compositions, it must be remembered that loose compositions, as photoflash mixtures, igniter mixtures, and fast-burning illuminant compositions, usually react with destructive violence.

This reaction is essentially due to three factors (1) the amount of air contained in the sample as well as the gaseous products of the reaction, (2) the great speed at which the energy may be released, and (3) the high heat of reaction.

All these factors favor shock wave formation and, thus, destructive effect. The temperature reached during the reactions which take place in pyrotechnic compositions ranges from 1000 degrees C to over 3500 degrees C.

This chapter deals with safety precautions which must be observed when working with up to five pounds of pyrotechnics in the laboratory.

Before beginning any work with pyrotechnics, the supervisor and his operators shall refer to Chapter 4. All safety information which they gather from studies, check lists, and former operator experiences must be applied to the processing of pyrotechnic materials. The design criteria and safety objectives discussed in Chapter 5 must also be applied to laboratory work with pyrotechnics.

SAFETY PRECAUTIONS FOR WORK WITH PYROTECHNICS

UNDERSTAND THE HAZARDS. Before beginning a project, the supervisor and his operators must thoroughly analyze and understand all details. The supervising chemist shall write a plan of operations with particular attention to those safety precautions which will avoid hazards. He must thoroughly discuss the plan of operations with his operators to be sure that they understand any hazards which they may encounter.

The equipment must be inspected, tested and deemed safe for the planned operation. If equipment is not safe, defects in facilities and equipment must be corrected before the planned operation can proceed.

SAFETY EQUIPMENT. Safety equipment shall be provided in the pyrotechnics laboratory as follows:

1. Tables, scales, ovens, presses and all other processing equipment shall be made static-proof by grounding. The floors shall be covered with a conductive, non-sparking material.

2. All electrical apparatus and wiring shall be installed in accordance with the National Electrical Code (see Article 4026 of OP 5, Volume 1).

3. Non-spark generating tools shall be used in the laboratory.

4. Ovens shall be equipped with covered heating coils and explosionproof latches. They shall be equipped with double thermostatic controls to prevent overheating. Relays actuated by the thermostats shall be set for "normally open" operation with respect to the heating power supply.

5. Laboratory hoods shall be equipped with one-half inch laminated safety glass, strongly supported to resist blast pressures.

TEST FACILITIES. Pyrotechnics test facilities shall include the following structural items as minimal equipment to maintain safety standards:

- 1. Tunnels to measure smoke and light outputs
- 2. Firing Bays
- 3. Instrument Rooms
- 4. Isolated Test Facilities
- 5. Adequate Storage Magazines

Additional features to be considered are as follows:

- 1. Blow-out walls
- 2. Roof construction
- 3. Locations of barricade doors.

OPERATORS. The operators shall wear the following personal protective safety equipment at all times:

1. Fire-resistant coats and other apparel

2. Conductive safety shoes or grounded wrist bracelets

3. Safety glasses, goggles, and shields to protect the neck area.

Flash or Explosion. When the work involves experimental compositions which may flash or explode, the operator shall proceed as follows:

1. Wear fire-resistant gloves and goggles.

2. Stay behind an approved, strongly-supported safety shield.

3. Wear a full-face shield which covers the neck area.

FIRE-FIGHTING EQUIPMENT. All laboratories shall be equipped with approved, strategically located fire extinguishers, fire blankets, and safety showers.

OPERATOR TRAINING. Operators shall be trained to use fire-fighting equipment as follows:

1. Fire drills shall be used to train the operators; each man shall be assigned a station.

2. The operator shall not attempt to extinguish a fire that can cause a dangerous explosion.

3. All operators shall evacuate the laboratory in accordance with an established plan proven by practice fire drills.

WARNING

Do NOT smoke while wearing contaminated protective clothing.

SAFE OPERATING PROCEDURES IN THE PYROTECHNICS LABORATORY The following procedures must be observed in the pyrotechnics laboratory:

1. Do not perform any experimental or hazardous work in the laboratory unless two persons are present. At least one of these persons must be experienced in work with pyrotechnics. Separate work areas must be assigned to each person to avoid the danger of both persons being injured by the same accident.

2. Conduct initial experiments with the smallest possible amount of material.

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3. Determine the following properties of new formulations:

a. Ingredients compatability

b. Electrostatic sensitivity

c. Impact sensitivity

d. Friction sensitivity

e. Ignition temperature

f. Vacuum stability at a suitable temperature

g. Toxicity of ingredients and combustion products.

4. Record results of the above sensitivity tests with any other information pertinent to the safe handling of the compositions.

5. Make this information readily available to persons engaged in pilotplant operations.

6. Establish quantity limits for areas and do not exceed them. Keep on hand those quantities necessary only for the assigned task.

7. Do not leave any experiment unattended. Use a placard or other means to warn other operators of hazardous conditions.

8. Do not grind or crush any pyrotechnic composition by any method which may cause impact or friction.

9. Use wet methods of mixing when material is very sensitive.

10. Mix small quantities by remote control.

11. When feasible, use closed-circuit television to observe mixing operations.

12. When non-flammable solvents can be proven compatible, they must be used.

13. Never charge ovens with incompatible materials. Do not leave sensitive materials in laboratory ovens overnight or over weekends.

14. Dry extremely sensitive materials in a high vacuum at a temperature not to exceed 60 degrees C.

HOUSEKEEPING. Observe the following housekeeping rules at all times:

1. Clean up spilled materials immediately.

2. Clean individual working area at the close of every working day.

3. Do not throw residual chemicals in waste baskets, general purpose waste disposal cans, or down any drains.

4. Keep waste materials segregated, and itemize them when sending them to disposal areas.

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<u>HAZARD PROTECTION.</u> The worker shall be protected against hazards at all times. This publication urges the user also to refer to OP 2793, TOXIC HAZARDS ASSOCIATED WITH PYROTECHNIC ITEMS.

Observe the following rules:

1. Avoid physiological hazards. Do not inhale pyrotechnic components as a dust, ingest them by way of mouth, nor allow them to come in contact with the skin.

2. Wear approved respirators in dusty operations.

3. Do not eat in the laboratory.

4. Wear approved protective clothing to protect the skin.

5. Where possible, perform dusty operations under a fume hood equipped with a good exhaust system.

6. Wash thoroughly before lunch, and shower thoroughly at the end of the working day.

USES OF PYROTECHNICS

Pyrotechnic compositions are used for illuminants, smokes, flares, delays, igniters, infrared flares, incendiaries, tracers, heat powders, photoflash, and simulated ammunition compositions. The compositions include military firecrackers, simulated land mines, and other noisemakers.

<u>ILLUMINANTS.</u> These compositions usually consist of a finely divided metal, an oxidizer, and a binder. These compositions are sensitive to impact, friction, electricity, open flames, and sparks. The usual metals employed are magnesium and aluminum, or any alloy of the two. Sodium nitrate is commonly used as an oxidizer. Paraffin used as a binder in older formulations has been largely supplanted by synthetic resins; some of these resins have toxic properties. The standard illuminating compositions can be very hazardous. Under the right circumstances, their potential energies can be released with destructive violence.

Experimental compositions can be even more hazardous. Smallest possible amounts must be used in initial experiments until sensitivity tests are made to determine the extent of the potential hazard.

Operations involving these compositions must be conducted behind strongly supported, laminated safety glass at least one-half inch thick.

Ball Milling or Grinding. Before attempting any ball-milling or grinding operation, the pyrotechnics operator shall wear the following personal protective safety devices:

1. Safety glasses

2. Coveralls

3. Conductive shoes or wrist bracelets

4. Head shield which covers the face and neck area

5. Protective gloves.

Mixing. The chemist shall observe the following precautions when mixing illuminants:

1. Do not mix compositions by mortar and pestle or by any other means which would subject the material to friction or impact.

2. Use well designed and well maintained mixers to prevent breakdown.

3. Be sure all mixing apparatus is well grounded.

4. Use non-spark generating tools.

5. Do not mix oxidant and metal dry unless a non-friction type mixer can be used.

6. Coat the metal with a binder prior to adding the oxidizer.

The newer binders used in illuminants usually consist of a synthetic resin, a polymerization agent, and a catalyst. The chemist shall be thoroughly familiar with the chemical, toxicological, and explosive hazards involved in work with these newer binders. He shall determine the proper time and order for addition of accelerators.

7. Do not add flammable solvents to illuminants to improve mixing procedures.

8. Establish the compatibility of all solvents.

Pressing. Observe the following precautions when pressing illuminants:

1. Be sure that all molds and tools are made of spark-resistant materials.

2. Inspect all molds and rams before use to insure that they do not contain any foreign material.

3. Inspect all molds and rams to insure that they are free from burrs which might cause friction.

4. Place the mold in the holding device and bring the ram down to check for proper alignment. Perform ram removal by remote control.

5. Perform pressing behind a one-half inch thick barricade and by remote control when possible.

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6. If the body to be pressed contains more than one composition, keep the compositions separated by baffles.

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7. Perform weighing operations on the composition at a safe distance from the pressing operation.

8. Do not allow pressed bodies to accumulate. Remove them from the pressing area as they are completed.

9. Sweep the base of the working area at frequent intervals with staticproof brushes to remove any flow-by or spilled compositions.

10. Test all dies and molds frequently to insure that they are within dimensional tolerances.

11. Install and use a shadow-board to prevent misuse and misplacement of tools used in mixing, charging, or transfer of materials.

SMOKE-PRODUCING COMPOSITIONS. These pyrotechnic compositions are designed to produce as much smoke as possible. Burning usually takes place less energetically than with other types of pyrotechnics, because at lower temperatures less of the solid smoke particles are consumed. Nevertheless, the grinding, mixing, and pressing precautions listed above under ILLUMINANTS shall be applied to the processing of smoke compositions.

<u>RED PHOSPHORUS.</u> Some of the smoke compositions may contain red phosphorus, and the user must observe the following precautions stringently when working with a phosphorus-base composition.

1. Determine impact, friction, and electrostatic sensitivities before beginning any production.

2. Guard against igniting a smoke composition by spark or friction.

3. Guard against spontaneous combustion in air.

4. Wear goggles to protect the eyes.

5. Wear protective equipment to prevent respiratory or percutaneous ingestion.

6. Keep adequate supplies of sand and water on hand to extinguish fires.

7. Use non-spark generating tools to open red phosphorus containers to avoid hot sparks.

Fire Prevention. Observe the following fire prevention measures:

1. Do not contaminate other buildings by entering a building not designated for red phosphorus. Contaminated shoes and clothing can cause a serious fire.

2. Clean red phosphorus equipment and work areas as often as necessary to prevent a fire.

3. Do not use brushes with metal ferrules around scales or balances when weighing red phosphorus.

Fire Fighting Procedures. In the event of a red phosphorus fire, proceed as follows:

1. Use generous amounts of water to extinguish the fire.

2. If burning phosphorus has come into contact with clothing, remove the clothing or keep it wet.

Personal Protection. The operator should protect himself as follows:

1. Keep any body area wet if it has come in contact with red phosphorus.

2. Keep small bandages impregnated with copper sulfate in the immediate area.

3. Wash the burned area in a five percent solution of copper sulfate, and see a doctor immediately.

4. If phosphorus enters the eye, use a bubble-type fountain or inverted faucet to cleanse the eye. Use large quantities of water.

5. See a doctor immediately.

6. Irrigate the eye with castor oil.

WARNING

Do not use copper sulfate solution in the eyes.

Decontamination. To decontaminate an area after a red phosphorus fire, proceed as follows:

1. Move the bulk of the partially burned material to a safe burning location. Keep the material wet in transit.

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2. Wash down the contaminated area with large quantities of water; then wipe down walls and floors with a vegetable oil. Wash the area again.

3. Disassemble all equipment and clean all parts with trichloroethylene in a properly ventilated work area.

4. Allow equipment to dry for 16 hours.

5. Inspect the building at night for phosphorescent residues.

USE OF CHLORATES. The chlorates used in smoke-producing compositions can present hazardous conditions. The chlorates increase a composition's sensitivity to impact and explosion. To avert any hazardous conditions, smokes must be mixed in two stages.

Proceed as follows:

1. Make a premix of retardant and oxidizers.

2. To this premix add fuel, dyes, and volatile solvent.

3. Prior to granulation, dry the composition to evaporate the volatile solvents.

4. Use a forced-draft oven heated by steam coils or a vacuum oven at temperatures below 60 degrees C.

<u>Dyes.</u> The dyes used in colored smokes can inflict physiological hazards. Find out specific information about toxic properties of dyes from their manufacturers.

FLARE COMPOSITIONS. Flare compositions are designed to produce white and colored light in various ordnance devices. A typical flare formula is that used to produce the red flare. It consists of potassium perchlorate, stronium nitrate, magnesium coated with paraffin, hexachlorobenzene, and gilsonite.

All the precautions listed under <u>ILLUMINANTS</u> (Mixing and Pressing) and Use of Chlorates, must be strictly observed.

Fuels and binders are premixed, oxidizers and other components are premixed; then the two premixes are combined to form the flare composition.

Infrared Flares. Infrared flares are compositions consisting of finely divided metal and oxidizers selected to emit energy in the infrared region. Observe the following safety precautions.

1. All safety precautions under ILLUMINANTS.

2. Avoid inhaling fumes from burning flares, as they are very toxic.

<u>Tracer Compositions.</u> Tracer compositions consist of an oxidant, a fuel, and a binder, except in a starter mixture where the binder is usually omitted. A typical composition consists of a finely powdered metallic fuel, stronium nitrate oxidant, polyvinyl chloride binder, and color intensifier. Observe all safety precautions listed under ILLUMINANTS in this chapter.

DELAY COMPOSITIONS. Commonly used delay compositions are the so-called gasless type. They consist of metal as the fuel and chromates as the oxidizer. The sensitivities of some are similar to those of illuminants.

WARNING

Experimental delay powders can be very hazar dous depending on the metal used, particle size, and oxidizers used. Delay work is made more hazardous as many of the compositions require very sensitive oxidizers. Delays using fine zirconium or titanium are in the hazardous category. Those using manganese or zirconium-nickel alloys are much less sensitive and burn without flash but emit much heat.

Observe the following safety precautions:

1. Use remotely controlled, non-friction-type mixers.

2. Be sure mixers are installed behind adequate shielding.

3. Wear the proper clothing including fire-proof gloves, safety glasses, face shields, conductive safety shoes, and/or grounded wrist bracelets.

4. Do not hand-mix an experimental formulation exceeding five grams without adequate sensitivity tests to determine the hazards involved.

5. Hand-mix experimental formulations in a hood behind laminated safety glass with the exhaust system ON.

6. Do not perform any mixing operation by a grinding or crushing operation.

7. During weighing, keep the component containers sufficiently separated by baffles to avoid a spark jumping from one container to another.

8. Test and use all grounding devices.

9. Use non-spark generating spatulas.

10. Limit components to 25 grams.

<u>Pressing</u>. Observe the safety precautions listed under <u>ILLUMINANTS</u>. Use static-proof brushes to remove any spilled composition from weighing and pressing areas. Remove the pressed delays from the work area. Operate the press from behind one-half inch, laminated safety glass or other suitable safety shield.

IGNITERS. Observe the safety precautions listed under ILLUMINANTS. Also observe the following safety precautions:

1. Mix titanium, zirconium or other very sensitive fuels under a nonflammable solvent.

2. Load the ignition devices wet.

3. Dry the units in a vacuum oven under argon.

4. Guard against the possibility of explosion of titanium, zirconium, lead dioxide, boron, and potassium nitrate compositions by storing them in static-proof containers.

PHOTOFLASH COMPOSITIONS. Photoflash compositions are composed of finely divided metals, an oxidant, and a depressant. A typical composition consists of powdered aluminum, barium nitrate, and calcium stearate. These mixtures are very sensitive and can explode with great violence. All applicable explosive and pyrotechnic safety precautions in this publication must be observed in photoflash operations.

HEAT POWDERS. Heat powders consist of high-energy metallic fuels and usually chromate oxidizers. All precautions must be observed, with particular attention to protection against sparks or static electricity.

INCENDIARIES. Incendiaries may involve pyrophoric fuels, phosphorus compositions, thermite mixtures, gelled gasoline, or other organic fuels.

Fire hazards are of the highest order. Explosive and physiological hazards are also prevalent.

All applicable explosive and pyrotechnic safety regulations must be followed stringently.

Chapter 8

PYROTECHNICS IN THE PILOT PLANT

GENERAL

This chapter deals with the safety precautions which must be observed by a pilot plant producing up to and more than 100 pounds of pyrotechnics.

The information presented in Chapter 7 is also applicable to pilot-plant pyrotechnic operations, and this information shall be reviewed thoroughly by the supervisor and his operators before proceeding on any pyrotechnic processing.

STATIC ELECTRICITY

Possibly the greatest danger to a pilot plant pyrotechnic operation is static electricity. To prevent the generation of sparks and the possibilities of sympathetic ignition, observe the following safety precautions.

1. Wear conductive safety shoes and/or wrist bracelets at all times.

2. All operations performed by remote control shall be listed in Standard Operating Procedures with adequate WARNINGS and/or explicit instructions from supervisory personnel.

3. As an example of the above precaution, crimping by remote control is not necessary when joining cartridge cases to projectiles. However, a pyrotechnics with likely pinching or rubbing shall be performed by remote control.

4. Maintain adequate space and erect barricades between each operator.

5. Practice good housekeeping rules at all times.

Chapter 9

PROPELLANTS IN THE LABORATORY

GENERAL

This chapter is a discussion of the processing of propellants in the laboratory. Supervisors and operators are again advised to return to their library facilities to gather all previous information reported on a particular propellant. They are again enjoined to set up and make use of exhaustive check lists. Likewise, they should seek the personal advice of any and all persons experienced in earlier synthesis of the propellant. Follow the recommendations in Chapter 4. Laboratory propellant facilities are similar in design and construction to chemical laboratories. This presents an inherent inability to obtain explosion-proof laboratory equipment.

With respect to facility design, the user of this publication shall throughly acquaint himself with the information in Chapter 5.

PRELIMINARY STUDIES

When specific knowledge concerning a new propellant's characteristics is lacking, it is imperative that laboratory personnel assume the most disasterous hazards and prepare for them. Processing and handling hazards will be minimized only if the laboratory personnel regard a new operation as extremely dangerous. As laboratory personnel become expert in processing and handling, specific methods and safety practices will lessen the chances of an accident. Again, the three factors which must be held to a minimum are: amount of propellant, number of operators, and exposure time.

The information in Chapter 4 shall be diligently applied to propellant processing because, in many cases, the finished product is more hazardous than its components.

PREPARATION AND HANDLING OF SOLID COMPOSITE PROPELLANTS

Solid propellants and their components are hazardous materials and must be treated accordingly. Toxicity of propellant monomers, shock sensitivity of oxidizer, and flammability of oxidizer in contact with a combustible material are factors which necessitate every precaution in working with these materials. In combination with fuels and additives, and in other combinations, the intermediate processes or products can be more hazardous.

Assume that a propellant can become a HE. Calculate the heat of explosion and treat accordingly. With composite propellants, this chance is slight. For double-base propellants, the assumption is real, and for composite modified double-base types, the chance is excellent. All propellants in which the binder is also rich in oxidizing or reducing groups will behave as CMDB type.

This publication highly recommends that the user consult OP 3199, HANDLING AND STORAGE OF LIQUID PROPELLANTS. The materials cited in OP 3199 are used extensively in the synthesis of solid propellants.

The recommendations listed below are intended to serve as guides in anticipating the hazards associated with laboratory-scale preparation of solid propellants. They are not self-sufficient or all-inclusive. The supervisor or supervising chemist is responsible for the proper interpretation of these recommendations. In case of doubt concerning the safety of any operation, the supervisor or supervising chemist shall consult competent, experienced authorities before proceeding.

The following paragraphs contain general precautions, special precautions for established formulations, and special safety precautions for new propellants.

GENERAL PRECAUTIONS. The general precautions listed below pertain to individual ingredients, mixing procedures, casting, curing, aging, storage, and disposal.

Individual Ingredients. The following precautions must be observed:

1. Be sure toxicity, shock sensitivity, flammability, friction sensitivity, and general chemical reactivity of all ingredients are well understood by the supervisor and his operators.

2. Minimize the effect of a possible violent reaction by preparing one or more new ingredients in a five-gram batch.

NOTE: New ingredients are any materials not previously used in combination with other ingredients.

3. Perform auto-ignition and stability tests on the propellant resulting from the five-gram batch before preparing any larger batches.

Mixing Procedures. All propellant mixing operations must be carried out under supervision of a qualified chemist. Proceed as follows:

1. Exercise every precaution in mixing a fresh batch.

2. Perform mixing operations in an area containing explosion-proof lights and connections.

3. Wear safety glasses and safety shoes at all times.

4. Use face shields as necessary.

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5. Prepare batches over two pounds by remote control and utilize personnel shielding for a batch under two pounds. The supervising chemist shall determine when, because of unusual sensitivity, the batch shall be under two pounds.

6. Add ballistic additives thoroughly wet with fuel before oxidizer addition to prevent sensitization of the oxidizer.

7. Thoroughly vacuum all mixes during the mixing process.

8. Establish fuel polymerization exotherms and propellant cure exotherms by small-scale preparations. If large exotherms are anticipated, take the necessary precautions to maintain the propellant temperature within safe limits.

Casting. Casting operations shall be performed as follows:

1. Perform all casting and vibrating operations with an explosion-proof vibrator from behind a safety shield.

2. Wear plastic gloves when casting propellant from a glass mixing tube.

3. Perform the above operations in a hood to minimize the concentration of toxic monomers in the atmosphere.

Curing. Observe the following precautions in propellant curing:

1. Establish proper curing schedules by determining auto-ignition temperature, maximum temperature of adiabatic cure, and possibly of exothermal heat release after casting.

2. Do not, under any circumstances, place a propellant in a curing oven until the exothermal heat has been thoroughly dissipated at ambient temperatures.

3. Do not use an electrically heated oven for any propellant curing operation Use a steam-heated oven of suitable size with proper recording devices and circulating fans.

4. Do not cure experimental propellants in the same oven with production propellants, and do not cure different experimental propellants in the same oven.

Aging. Propellant aging operations shall be performed as follows:

1. Establish aging characteristics for experimental batches at subnormal, ambient, and high temperatures in areas provided for this purpose.

2. Conduct initial aging tests of new formulations in complete isolation from all other propellant aging tests.

3. Be sure ovens are equipped with safety thermo-switches to prevent overheating.

Storage. Observe the following precautions in storage of propellants:

1. Do not store any propellant in any laboratory area where a hazard could exist to personnel assigned to that working area.

2. Use explosive magazines for propellant storage.

3. Strictly observe the load limits of any magazine.

Disposal. Observe the following rules in the disposal of propellant ingredients and propellants:

1. Use specified waste drums for disposal of propellants.

2. Do not throw waste paper, glass, or oxidizers into a waste propellant drum.

3. Establish that there is no residual polymerization exotherm which could cause heating of the propellant after disposal and a possible fire in the disposal drum.

ESTABLISHED FORMULATIONS. The term "established formulation" pertains to one which has been in use for a sufficient amount of time for complete hazard evaluation. Those preparations which involve only changes in the proportions of established ingredients are also considered "established formulations."

Fuel Monomers. The following precautions shall be applied to fuel monomers:

1. Use fuel monomers in a well-ventilated area with adequate skin protection.

2. Wash any spillage on the skin immediately with water.

WARNING

Isocyanates and acrylates are toxic compounds. Acrylate monomers can cause irritation to the mucous membrances, gastro-intestinal disorder, headaches, and kidney irritation. Concentrations of toluene di-isocyanate as low as 2 ppm have been shown to cause bronchial pneumonia in animals.

Oxidizers. Nitrates and perchlorates are extremely hazardous compounds when contaminated with combustible materials. Observe the following precautions:

1. Store nitrates and perchlorates in separate buildings, away from organic chemicals.

2. Prevent dust formation.

3. Wash skin immediately on contact with any oxidizer.

4. Do not, under any circumstances, allow ballistic additives to be admixed with dry oxidizers. Most of these materials are known to sensitize nitrates and perchlorates.

NEW PROPELLANTS. Preparations involving untested combinations of ingredients (new fuels, oxidizers, plasticizers and/or additives) shall be considered "new formulations." The supervising chemist shall acquaint himself with the potential hazards of any new material and personally supervise **any** initial preparations using new materials. Observe the following precautions with new propellants.

1. Confine preparation of initial batches to five-gram samples until safe processing techniques have been developed.

2. Determine auto-ignition temperature, toxicity, impact stability; and chemical reactivity of the ingredients singly and in combination.

3. Observe the exothermal heat release of new fuels.

4. When using additives, watch for any increase in impact sensitivity or decomposition of monomers.

MECHANICAL PROPERTIES TESTING. All handling of solid propellants must be performed only when wearing safety glasses, conductive safety shoes, and a laboratory coat.

Temperature Conditioning. Proceed as follows:

1. Condition any new propellant for approximately 24 ± 4 hours at the test temperature. The test temperature must not exceed 60 percent of the auto-ignition temperature.

2. Perform all propellant conditioning in explosion-proof ovens.

3. Do not use electrically heated ovens.

Specimen Preparation. It is frequently necessary to repair the surface of test specimens by sanding. Since the propellant dust is hazardous as a high explosive, proceed in the following manner:

1. Wear rubber gloves or cover hands with protective grease.

2. Wear a face shield.

3. Sweep the sanding dust into a bucket of water after sanding four fivegram specimens. <u>Testing</u>. Mechanical property testing specimens must not be sensitive to detonation and should not evince low sensitivity to friction. Proceed as follows:

1. Test impact sensitivities down to 10 cm/2 Kg weight in low rate testing operations.

2. For high rate testing operations, establish a minimum of 25 cm/2 Kg weight until adequate safety requirements can be determined.

Chapter 10

PROPELLANTS IN THE PILOT PLANT

GENERAL

Safety requirements intended to reduce property damage and bodily injury during the manufacture of castable composite propellants are described in this chapter. The chapter also discusses safety requirements which must be observed in loading castable composite propellants into rocket motors.

Those responsible for the design and operation of propellant plants are required to observe all applicable safety precautions presented in any part of this publication.

To be effective, a propellant must release substantial quantities of heat energy and large volumes of gases. Though this energy is released at a slower rate than explosives energy, the rate of release from propellants is of such a speed that a propellants accident can create destruction equal to that of an explosives accident.

The hazards of propellant processing are usually greater than the hazards associated with the finished product. New hazards may be encountered in a particular processing operation. Therefore, essential safety information must be gathered by working on a small scale. Exhaustive tests must be made before larger-scale work can be started. The detonability or nondetonability of a new propellant formulation must be established early in its development.

Tests should also be run to determine the relative sensitivity of the propellant composition to various types of energy inputs which might be encountered during the preparation and handling of the composition.

The protections required when working with detonable propellants is substantially greater than those required in working with a non-detonable propellant. The increasing demands for more energetic propellants require that more energetic ingredients be used.

The use of more energetic ingredients, combined with the missile program's time table, means that propellant formulations cannot be thoroughly investigated. Therefore, the processing of a new propellant has inherent dangers which must be accepted as part of first production.

To alleviate these dangers, a propellant plant must necessarily "spread out," reconsider their design criteria, and even double the intraline distances called out in OP 5 to assure that any property damage is minimal and that employee injuries are reduced.

For the purpose of this publication, composite propellants are those which consist of mixtures of fuels, binders, and oxidizers with or without other ingredients. The publication is not applicable to operations with only colloid propellants. However, it is applicable to colloid propellants (cast double - base) that are used as ingredients in composite propellant compositions.

BUILDING CONSTRUCTION

This publication recommends the use of expendable frame structures. The structure shall consist of four blow-out walls surrounded by earth barricades. It is also recommended that remote-control facilities be located outside the structure. The yield of the blow-out walls shall be 0.1 psi/square inch.

It is impossible to design a massive concrete structure that will adequately protect nearby personnel from an explosion of 50 pounds or more.

FIRE-RETARDANT MATERIALS. Maximum use of fire-retardant materials shall be obligatory. The missile hazard shall be minimized by proper construction and materials.

ROOFS AND CEILINGS. Roofs shall be of the venting type and shall be of material which will readily break up into small fragments. However, ceilings shall not be provided in explosives operating areas, because they add to the weight of vent-hatch design.

MULTIBAYS. In present multibay construction, when two separate operations must be performed at the same time, reserve an empty bay between the two operations to provide optimum protection.

Interior Surfaces. Exposed interior surfaces of propellant operating buildings shall be smooth, fire-retardant, and free from cracks and crevices. Joints shall be taped or sealed.

Paint. When walls and ceilings are to be painted, hard-gloss paint shall be used to facilitate cleaning and minimize impregnation of furnished wall and ceiling materials.

Molding. A flush, curved molding shall be provided where walls and floors meet.

Doors and Windows. Flush-type construction shall be used for door and window openings.

DRAINS AND SUMPS.

<u>Drains</u>. All drain lines handling propellant wastes shall be provided with sumps or basins of adequate capacity for the removal of explosives by settling. The drains shall be free of pockets and shall have sufficient slope (at least one-quarter inch per foot) to prevent settling of explosives in the drain line before they reach the sump or settling basin where wastes are to be collected.

<u>Sumps.</u> Sumps must be designed so that suspended solid explosive materials cannot be carried beyond the sumps by wash waters. Their design shall allow sufficient settling time based on the settling rate of the material and the normal rate of flow. The sump shall be constructed so that the overflow will not disturb any floating solids. The sump design shall permit easy removal of collected explosives, and retention of floating explosives until they can be skimmed off. Bolted sump tanks or other types of construction which allow the propellant to settle in obscure or hidden spaces are prohibited.

Care must be exercised to avoid the possibility of propellant Deposition from sump effluent due to drying, temperature changes, or interaction with other industrial contaminations. When explosives which are appreciably soluble in water are handled, sweeping and other dry collection measures shall be used to keep them out of the drainage system.

Some water-soluble waste liquids should not cause any trouble if they are flushed into the waste water system with an adequate quantity of water.

It may be more dangerous to try to collect the water-soluble propellants in a dry state than to wash them down into a proper drainage system. A proper drainage system in this case is one in which waste water is run into evaporation pools and chemicals added to destroy the hazardous watersoluble materials.

In all new construction subsequent to the date of this publication, drains between the source of propellant operations and the sump shall be troughs with rounded bottoms and removable ventilated covers to facilitate inspection for accumulation of propellants. Drains and sewers containing waste materials must not be connected in a manner which might empty these wastes into the normal sewage systems.

FLOORING. Flooring shall consist of non-sparking, conductive materials as described in paragraph 4207 of OP 5, Volume 1.

Operational Shields. The following specifications are to be met in operational shielding.

1. The protective wall between operators shall be a reinforced concrete wall not less than 12 inches thick.

2. Concrete shall have a design compressible strength of 2500 psi.

<u>REINFORCING.</u> The following specifications shall be met in reinforcing operational shielding:

1. Both faces shall be reinforced with rods at least one-half inch in diameter.

2. Rods shall be spaced not more than 12 inches apart at vertical and horizontal centers.

3. Rods shall be interlocked and secured with the footing reinforcement to prevent overturning.

4. Rods shall be approximately two inches from each face, and rods on one face shall be staggered with respect to the rods on the other face.

The reinforcing specified above constitutes adequate protection for operations with less than 15 pounds of detonable material when the nearest part of the material is three feet f rom the wall and two feet from the floor. A reinforced concrete wall 30 inches thick is satisfactory protection against the effects of up to 50 pounds of a massed quantity of explosive or detonable propellant. Thirty-six inches of reinforced concrete wall is satisfactory protection against the effects of 70 pounds.

The adequacy of these operational shields, including thickness, size, fastening, and location, must be proved by actual test with a 25 percent minimum safety factor before they are permitted in regular operations.

CONTROL BUILDINGS. Control buildings must be located to protect the occupants. Concrete and reinforcing steel shall be used in accordance with drawings approved by the Bureau of Naval Weapons. Earth cover shall be used as necessary.

When the amount of explosive or detonable propellant exceeds 70 pounds, earth-covered control buildings must be provided. Protection from missile hazard should be an adequately barricaded control shelter situated at a safe distance from the operations building. The mass of explosive or detonable propellant must be placed so that its long dimension is perpendicular to the shielding wall, at least three feet from the wall and two feet from the floor. When working with individual explosive or propellant components, operations and control buildings must be separated by the distances prescribed by Table 7.1 (Safety Distances - Intraline Separations) in OP 5, Volume 1.

WARNING

Any facilities at which mixing, blending, and loading operations of a high-energy propellant are in progress must be separated by DOUBLE the intraline distances prescribed in OP, 5, Volume 1, Table 7.11. ELECTRICAL EQUIPMENT. The following precautions shall apply with regard to the use of electrical equipment:

1. All types and installations of electrical equipment and wiring shall comply with the provisions of the National Electrical Code as modified herein.

2. Electric motors, wiring, lighting fixtures, and any other electrical devices shall not be located in rooms or buildings where propellant dusts, flammable vapors, and combustible dusts can form explosive or hazardous mixtures with air.

3. If the above installations are necessary for practical purposes, the electrical equipment or devices must be approved for use in Classes I or II hazardous locations. They shall be rated Class I or Class II if both hazard classes are present. If rated equipment is not available, use the equipment approved for the more hazardous condition.

4. For Class I and for II hazardous locations, install lighting fixtures which will not raise exposed surfaces' temperatures above 228 degrees F.

5. These provisions do not preclude installation and use of grounding circuits and static grounds in propellants operating buildings.

VENTILATION. The following precautions must be observed in the ventilation of propellant plants:

1. Adequate ventilation shall be provided for operations with vapors and dusts which are toxic or explosive in nature.

2. Use a system with intakes near the sources of vapors or dusts. Intakes shall be designed for type materials used -- heavier-than-air vapors or lighter-than-air vapors.

3. When possible, use operating buildings with no ceilings to keep any contaminants low at a normal working level.

4. Air-conditioning and circulating equipment shall be designed to prevent the re-circulation of combustible dusts through the equipment.

5. All duct openings at hazardous locations shall be designed to prevent contamination during any period when fans or blowers are not operating. Use filters as applicable.

6. As dust conditions vary with concentration, install dust collectors and gas scrubbers.

AUTOMATIC DELUGE AND SPRINKLER SYSTEMS. Automatic sprinkler systems shall be installed in accordance with temperature limits in Table 3. COMFORT-TYPE HEATING UNITS. The two types of heating units described below are applicable to propellant operations buildings.

Forced Hot-Air. This system is similar to the forced hot-air heating systems installed in homes; the hot air, however, is not re-circulated but vented to outside the operations building.

<u>Steam-Heated Coil</u>. In this system, a steam-heated coil is located OUT-SIDE the operations building, and fans are employed to force the heated air through ducts into the operations building.

These installations must be approved for Class I and Class II hazardous locations and the fansare made of non-sparking material.

FLOORING. This publication recommends that an initial concrete slab be poured; next, another concrete slab with imbedded lead particles shall be poured and connected to the building's secondary grounding circuit. Conductive safety shoes shall be worn.

GROUNDING. Manufacturing equipment and conductive flooring shall be grounded in accordance with Chapter 42 of OP 5, Volume 1.

LIGHTNING PROTECTION. Lightning protection shall be provided in accordance with Chapter 41 of OP 5, Volume 1.

PROTECTIVE SHIELDS. Protective shields, as discussed under CONTROL BUILDINGS, shall be utilized for the protection of employees, materials, and equipment. Shield design shall be based on potential hazard, and shields are mandatory in the use of detonable propellants. Provision shall be made for adequate venting, based on the rate of hot gas evolution. The specifications are intended as guides in designing operational shields which provide protection from other types of hazard (fire, pressure-vessel rupture, and highvelocity, high-temperature gas streams).

Shield Design. Shield design shall be controlled by propellant quantity, confinement, and potential initiation hazard. Processing hazards include the following:

1. Propellant detonation;

2. Unconfined propellant fires;

3. Case or vessel pressure failure with fragmentation (heavy-wall, mildsteel cases operated at high pressure can constitute a major hazard by producing fragments of high energy):

4. Vessel pressure failure without fragmentation (aluminum or highstressed steel cases);

5. High-temperature, high-velocity gas streams caused by ignition of restrained motors filled with composite propellant;

6. Motors, with or without nozzles, not adequately tied down, may take flight upon ignition.

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ESTABLISHMENT LAYOUT. The facilities of the individual operating line shall meet the following requirements:

Location. Composite propellant manufacturing and motor loading operations shall be performed in a special area that is separated from all other areas; boundaries shall be established by Tables 7.9 and 7.10 of OP 5, Volume 1.

Intraline Distances. Consistent with safety and efficiency, hazardous operations shall be separated from each other and from all non-hazardous operations. Where feasible, DOUBLE the intraline distances prescribed in Table 7.11 of OP 5, Volume 1. When more than one hazardous operation is performed in a single building, maximum use shall be made of substantial dividing walls and protective shields. An analytical study of blast effects in a cubicle may be found in Industrial Engineering Study to Establish Safety Design Criteria for Use in Engineering of Explosive Facilities and Operations -Wall Response, a report compiled by Ammann & Whitney in April 1963 for Process Engineering Branch, A. P. M. E. D., Picatinny Arsenal, Dover, N. J.

INDIVIDUAL EXPLOSIVE BUILDINGS. Individual explosive buildings within a composite propellant operating line shall be separated from each other and from buildings not containing explosives (within the same operating line) by appropriate intraline distances. Except for service magazines and curing facilities, adjacent buildings housing explosives of varying hazardous degree shall be separated by that distance protecting against the more severe hazard.

Service Magazines and Curing Facilities. Service magazines and curing facilities shall be separated from their operating buildings by intraline distances. These distances shall be based on the quantities of explosive in the service magazines and curing facilities.

Batch Mixing. Batch mixing operations shall be performed in buildings designed specifically for this purpose. Small mixers may be located in other operations buildings, but the mixers must each be enclosed in a separate bay with shields protecting all other operations.

Use shields to protect other equipment in the bay from the accidental explosion in a small mixer. This provides for the installation of a small mixer in a bay normally used for other operations without having to remove the other equipment.

Non-Explosives Facilities. Non-explosives buildings (paint and solvent storage) serving a single operating building may be located at less than intraline distance, but not less than 100 feet.

Class 2 Materials. In locating new facilities intended for Class 2 materials, consideration must be given the possibility that Class 9 explosives may be

processed in future operations, and new facilities should, therefore, be laid out on the basis of Class 9 hazards.

EXPLOSIVES HAZARD CLASSIFICATION

The hazardous chemical and physical properties of individual raw materials and all compositions shall be determined prior to their use in a propellant manufacturing process. Procedures for the determination and classification of a cured or uncured propellant hazard are included in BUWEPSINST 8020.3, EXPLOSIVE HAZARD CLASSIFICATION PROCEDURE. Spark sensitivity and friction sensitivity procedures are outlined in Department of the Army TM 9-1910, MILITARY EXPLOSIVES.

Recent studies at the Bureau of Mines have indicated that low-order detonations, which are generally disregarded in detonation tests, can cause almost as much damage as high-order detonations. Therefore, positive low-order detonations should be considered.

When warheads with high explosives are assembled to motors containing propellant, tests must be conducted to determine a possible contribution of the propellant to an explosion involving the warheads. The findings of these tests must be expressed in terms of explosive required to produce the same effects. Quantity-distances are based on the quantity of explosive in the warhead plus the explosive equivalent of the motor. Full-scale tests are required, and these tests are also outlined in BUWEPS INST 8020.3.

When high-explosive warheads are assembled to motors containing Class 9 propellant, quantity-distances shall be based on the combined weights of explosives in warheads and propellant weight in the motors.

OXIDIZER PROCESSING

The following safety precautions apply to oxidizer processing:

1. The safety regulations for the safe handling of oxidizing materials given in Chapter 33 of OP 5, Volume 1 shall apply.

2. Avoid contamination.

3. Determine the use of metals or chemicals that can sensitize the oxidizer and permissible location by use of exposure time, temperature, and moisture data.

4. Stay behind a twelve-inch, reinforced concrete wall during screening, blending, grinding, and mechanized drying operations.

5. Eliminate dust and contamination from outside sources with a closed ventilation system.

6. Use flexible connections (socks) which are fire-retardant and chemically compatible with the oxidizer in pipes or duct systems.

7. Pipes or duct systems shall be connected to secondary grounding systems.

8. Use flanged pipe connections in lieu of threaded joints wherever possible.

DRYING OF OXIDATING AGENTS. Observe the following precautions in all drying roomst

1. Maintain and do NOT exceed the maximum safe drying temperature for each material.

2. Use a dual thermostatic control to maintain a safe temperature level if the dryer is capable of attaining more than the maximum safe drying temperature.

3. Record temperatures at periodic intervals.

4. Eliminate possibilities of dust formation and accumulation.

SCREENING. When screening is performed for process purposes, observe the following precautions;

1. Use equipment to protect the oxidizer material from pinching, friction, or impact as a result of metal-to-metal contact.

2. Thoroughly clean screening rooms daily, or more often as necessary, to prevent hazardous accumulation of dust.

OXIDIZER BLENDING. If analyses indicate that gases are released during blending of oxidizer batches, a suitable pressure-relief shall be designed into the blender.

OXIDIZER GRINDING. The following precautions shall be observed in grinding of oxidizers:

1. Maintain sufficient clearance between stationary and moving parts of impact-type mills to prevent metal-to-metal contact. Check these clearances as often as necessary; at least once daily.

2. Pass oxidizer material through a screen and magnetic separator for removal of extraneous materials. The screen and separator should be located in the intake of the grinder.

3. Use the smallest screen openings that will permit oxidizer flow.

PREPARATION OF FUEL COMPOSITIONS

Equipment design and handling methods shall minimize formation and accumulation of dust. If a toxic or flammable material is being processed, use adequate exhaust ventilation and use the prescribed personal protective equipment. Keep the entire transfer system conductive with the use of conductive plastic material.

Establish compatibility of materials. Use controls to maintain proper mixing of materials and to prevent formation of a sensitive composition or a hazardous condition prior to processing.

FUEL COMPOSITIONS AND OXIDIZERS MIXING (BATCH METHOD) The following precautions shall be observed:

1. Mix fuel compositions and oxidizers by remote control.

2. Provide operator protection from the total quantity of materials being mixed.

3. Do not locate more than one production mixer in one building, unless protective construction assures that an explosion in one mixer will not damage other mixers or cause sympathetic detonation.

4. Add oxidizer to the mixer in a manner that minimizes formation of dust and static electricity. For any reasonable large-scale mixes, provisions shall be made for remote addition of oxidizer and all other ingredients after the mix is considered to be in a hazardous condition (for example, after addition of oxidizer to the fuel binder).

5. Provide effective means to prevent extraneous materials from entering mixers.

6. If a vertical-type mixer is to be used, make certain that its glands do not come in contact with propellant.

7. When, by reason of size, shape, and process requirements, it is impossible to screen dry solids before they are charged into a mixer, and charging in slurry form is impossible, develop an inspection technique to prevent extraneous materials from entering the mixer by using magnetic separators, fluoroscopes, and X-ray.

8. Inspect blades and other mixer moving parts through use of "Magnaflux" or X-ray for cracks, crevices and other imperfections before and during operation.

9. Maintain a record of mixer blade adjustment, inspections, and evidences of damage to blades and bowls.

10. If a propellant is NOT to be mixed under vacuum, place a noncombustible cover over the mixer bowl after charging to prevent the accidental entry of extraneous materials. The cover may be light-weight metal or screen with a mesh no greater than minimum clearances of the mixer blades.

11. Secure the cover to the mixer housing so that it provides for escape of gases and vapors evolved during the operation, and can not confine exhaust in the event of fire.

12. Do not spill or splash propellant during discharge of mixers.

13. Use only non-sparking devices to scrape down sides and blades of mixers. Use positive means (shadow board) to keep tools from being accidentally introduced into the mixer.

14. Provide adequate means for effective removal of toxic and flammable vapors, gases, dusts, and fumes from the mixing room.

15. Discharge operations operators shall station them selves so that exit routes are clear in case of an emergency.

16. Interlock all electric service to mixers with fire protection system control to prevent operation of mixers when fire protection system is inoperative.

17. Lock-wire all nuts and bolts on mixers, monorail systems, and other locations so they can not loosen and fall into a mixer.

18. Operators' uniforms shall comply with Chapter 43 of OP 5, Volume 1. Personal items as jewelry, pens, pencils, coins, and rings shall not be permitted in the building.

COMPOSITE PROPELLANT CASTING

The following specification shall be met before processing of castable composite propellants:

CASTING VESSELS.

1. Pressurized casting vessels shall be capable of withstanding at least double their maximum allowable working pressure.

2. Periodic hydrostatic tests of all casting vessels shall be performed at least every 180 days.

3. Tests shall be also made after any alteration to a casting vessel.

4. The periodic tests shall be recorded for each vessel.

5. Similar tests shall be performed on vessels which are evacuated in use.

Design. Casting vessel assemblies shall be designed to avoid internal cracks, crevices, corners, pockets, and any internal mating configurations which could cause propellant to be pinched or rubbed.

Lids.

1. Lids shall be secured to pressurized casting vessels so that they will withstand the vessel's rated pressures.

2. Perform frequent tests to assure that lid-locking devices are working properly.

Line Pressure. Line pressure shall not exceed working pressure. Filters shall be installed in gas lines to remove water and oil.

<u>Blow-out Disk.</u> A blow-out disk designed to blow-out at 120 percent of the vessel's maximum allowable working pressure (but not more than 150 psig) shall be provided. The design shall provide for the potential rapid pressure rise in the vessel due to propellant ignition.

VALVES. Valves through which uncured propellant flows shall be designed to prevent propellant from being compressed between two surfaces (rubberdiaphragm or pinch type). The valves shall be cleaned at regular intervals, and a log of these inspections shall be maintained.

PUMP CASTING. The use of pumps shall be avoided in solids processing; however, where casting is performed by pump, rupture sections shall be installed at the inlet and discharge sides. The sections shall be designed to relieve pressures in case of fire in the pump.

Adequate protection shall be provided for the workers at the propellant casting station. If transfer vessel and pump are located at the propellant casting station, the casting shall be performed remotely with operators adequately protected from exposure.

MANDREL INSERTION. When mandrels are inserted mechanically, the equipment shall be designed to prevent metal-to-metal contact between the mandrel and the motor case below the propellant surface.

COMPOSITE PROPELLANT CURING

The following precautions shall be observed in propellant curing:

TEMPERATURE. The safe curing temperature for the propellant shall be established, and dual heat controls shall be installed to prevent the safe temperature from being exceeded. If the amount of propellant exceeds one pound, a thermistor, thermocouple, or similar adequate temperature sensing device shall be set into the mandrel or around the case to indicate temperature conditions within the grain and core.

HEATING UNITS. Heating units or elements shall be designed to eliminate any direct contact between the heating unit or element and the propellant.

HANDLING GEAR. Loaded motor cases and casting molds shall be handled and secured to prevent overturning. Loaded or partially loaded rocket motors shall be suspended at minimum distances above floor level. If tests or experience indicate that rocket motors may ignite upon impact, restraining stands and/or chocks shall be used.

PRESSURE. Pressure relief valves shall be provided on closed, pressurized vessels which will contain motors during a curing process.

FRICTION. All casting and curing fixtures shall be designed so that thin layers of propellant cannot be pinched or rubbed between metal surfaces.

MOTOR FINISHING AND ASSEMBLY

Observe the following precautions in motor finishing and assembly operations:

MOTOR TIE-DOWNS. Pressure vessels must be secured in fixtures capable of withstanding the rated thrust of the assembly. This is based on its performance as a rocket motor. A safety factor of 2.5 shall be applied to offset shock loads.

THREADS. If a motor case design incorporates internal threads, contamination of the threads by propellant must be prevented. Design and casting of curing fixtures and assemblies shall exclude internal threads, cracks, and crevices whenever possible.

Cleaning and Inspection. All threads shall be inspected and cleaned prior to assembly of component parts.

REMOTE CONTROL. Assembly of threaded components shall be done by remote control. Personnel shall be protected by adequate shielding. In establishing limits, consideration shall be given to the distribution of hazardous materials as well as total quantities, in order that critical dimensions (for instance, depth of propellant powder in a single container) would not be exceeded.

WARNING

Detonable propellants and motors with detonable propellants shall be machined only by remote control.

MANDREL REMOVAL. Mandrel removal shall be by remote control with personnel protected at all times.

CASE-BONDED PROPELLANT MACHINING

The following safety precautions must be observed in case-bonded propellant machining:

FACILITY PREROGATIVE. The local facility shall determine the manner in which a case-bonded propellant can be cut, drilled, or otherwise machined. All operational and safety factors shall be considered.

MACHINERY DESIGN. Machining area and machinery design must take three factors into account: (1) the severe hazards of finely divided propellant, (2) adequate protection for personnel, and (3) minimal damage in case of accident. Propellant dust shall be removed by a vacuum collection system, preferably of the wet type, with intake point as close as possible to the cutter blade. Equipment shall be designed so that cutting tools or blades do not intentionally contact motor cases and other metal objects. The equipment shall not generate excessive heat. The equipment shall afford personal protection and facilitate dust and chip removal.

Design Criteria. Design points to be considered in machinery design are as follows:

- 1. tool configuration
- 2. lineal and rotational speeds
- 3. tool metal
- 4. rate of feed
- 5. safe and effective coolant
- 6. alignment of tool
- 7. control devices
- 8. power sources
- 9. maintenance or replacement
- 10. dust and chip removal.

The individual propellant's characteristics must also be considered in machine design. Initiating characteristics must be borne in mind, because a propellant with elastic properties may require greater rotational speeds and greater rates of feed than a brittle material.

EXHAUST SYSTEMS. All exhaust systems used for dust and chip removal shall comply with Chapter 46 of OP 5, Volume 1.

WASTE PRODUCTS. Waste products may be removed by immersion in a stream of water flowing away from the operation and collected at a location outside the operating room or cubicle.

Other Method. If motor design, grain design, or need to reclaim the propellant waste preclude the use of exhaust systems and immersion, waste products are to be collected in a dry state at the operation.

WARNING

The quantity of waste propellant plus the propellant in processed and unprocessed motors shall not exceed the explosive limit of the room or cubicle.

CUBICLE AND MACHINING ROOM LIMITS

Operator and materials limits for machining rooms and cubicles shall be determined in accordance with the following principles.

MINIMUM PERSONS, EXPOSURE TIME, AND HAZARDOUS MATERIAL. The cardinal principle which must be observed in any location or operation with explosives, ammunition, severe fire hazards, and toxic materials is to limit the exposure to a minimum number of personnel, for a minimum time, to a minimum amount of hazardous material consistant with safe and efficient operations.

All operations shall be scrutinized to devise methods for reducing number of operators, time of exposure, and quantity of material. Determination of personnel limits requires that (1) operations not necessary to a particular hazardous operation not be accomplished in that location, (2) unnecessary operators not be permitted to visit the location, and (3) that a series of consecutive operations not be permitted in the same location without adequate dividing walls, fire walls, or operational shields. Personnel limits must include supervisors and transients. Key employees shall be appointed monitors to assist in enforcing the established limits.

DETERMINATION OF LIMITS. Determination of limits for hazardous materials requires careful analysis of operation timing, transportation method, size of the item, and chemical and physical characteristics of the material. More stringent limits are required for the more sensitive or more hazardous materials.

<u>Bay-to-Bay Propagation.</u> Current studies in this area have not yielded empirical results. However, a CUBE ROOT DISTANCE RULE is noted here. An explosive equivalency is obtained by calculating the cube root of the amount of propellant multiplied by the heat of detonation for TNT (980 calories/ gram). The solution is then multiplied by 1.5 to 1.8,* and the final solution yields a rule-of-thumb distance between bays (in feet).

<u>Magazine-to-Magazine Propagation</u>. Rule-of-thumb distances between magazines may be obtained by calculating the cube root of the weight of the stored propellant multiplied by 1.5 for earth-to-earth direction to 4.5 for earth-to-door direction.

Tests with both donor and acceptor charges at a minimum of three feet from the walls have indicated that the maximum allowable amount of propellant in a single magazine should be between 400 and 500 pounds.

Proceed as follows:

1. Establish limits for each operation so that each operator may be responsible for not exceeding the limit.

2. Establish limits in terms of trays, boxes, or racks, or another unit which can be easily observed.

3. Do not establish explosive limits on basis of quantity-distance tables if lesser quantities of explosives will suffice for the operation.

* For those materials with greater yield than TNT.

4. Establish a quantity limit required for one shift; this should be a minimum quantity. These limits shall then be reviewed by cognizant personnel.

PLACARDS. All rooms, cubicles, and buildings containing hazardous materials shall have a placard stating the maximum amount of material and maximum number of operators allowed in the area at any one time. The placards shall be posted in conspicuous places, and other placards shall set forth local regulations, as required. Placards must be kept current.

EXCESS MATERIALS. The supervisor shall prevent accumulations of excess materials, and he shall remove them immediately. Non-operating personnel should be advised to leave the premises.

Personnel and quantity limits for buildings or process facilities shall be recorded on a plant layout and kept on file.

RECLAMATION. When rejected motors must be drilled to reclaim propellant or case, the operations shall be performed in an isolated area.

NON-CASE BONDED PROPELLANT GRAIN MACHINING

Propellant grains that are not case-bonded must be machined before they are loaded into motors.

Insofar as they are applicable, the safety precautions under <u>CASE-BONDED</u> <u>PROPELLANT MACHINING</u> also shall be met when machining composite propellant grains that are not case-bonded.

IGNITER INSERTION. The following requirements apply where a motor design necessitates inserting the igniter within the manufacturing line:

1. Supply of igniters at the insertion station shall be the minimum consistent with safe and efficient operations.

2. If the process requires removal of the shorting clip, the igniter shall remain shorted until immediately prior to insertion.

3. Storage facilities should be vented to the atmosphere and designed to withstand the effects of an incident involving all igniters in storage.

4. Operators shall wear wrist bracelets and/or semi-conductive safety shoes to dissipate any static charge during igniter insertion.

CONTINUITY TESTS. Electrical continuity tests for igniters installed in motors shall not be performed in the propellant manufacturing and motor loading lines. These tests shall be conducted in a separate facility, by remote control, with adequate protection provided for the operator. MATERIALS HANDLING. If loaded motors with cores must be moved, both motor case and core shall be adequately supported, locked, and tied together to prevent their independent movement. This provision does not apply to a loaded motor with a consumable foam mandrel.

A comprehensive preventive maintenance program shall be set up for all equipment used to move motors loaded with composite propellants.

DISASSEMBLY. The following safety precautions shall be observed in disassembly operations.

1. Use remote control with adequate shielding in disassembly operations with assemblies and fixtures with internal threads, cracks, or crevices.

2. Use common facilities for assembly and disassembly operations, provided they are not performed concurrently.

3. Operators shall be adequately protected during any disassembly operation involving the removal of pyrotechnic-type igniters.

PAINTING AND MARKING. Spray-painting shall be performed in compliance with OP 5, Volume 2.

<u>REWORKING.</u> Loaded component disassembly operations shall be separated from other operations. Reworking of defective loaded components shall be done only by properly trained personnel under the direction of supervisors who are fully aware of the hazards in both assembly and disassembly operations.

COLLECTION AND DISPOSAL OF HAZARDOUS WASTE MATERIALS. The following safety precautions shall apply:

1. Waste collection and disposal operations shall be closely supervised and covered by standard operating procedures. Adequate waste segregation shall be controlled.

2. Remove waste materials from operations areas at regular and frequent intervals.

3. Keep cured propellant scrap under water except in cases in which reactivity of one or more ingredients prevents under-water storage.

4. Use separate, properly labeled containers for different types of waste (combustible waste, waste propellant, waste oxidizer, and oil rags).

5. Disposal of waste materials shall be in accordance with OP 5, Volume 1, Part IV.

PROTECTIVE CLOTHING AND EQUIPMENT

The following personal safety precautions must be observed at all times:

1. Wear the proper eye protection (safety glasses, goggles, and face shields).

2. If clothing becomes excessively contaminated with oxidizer or propellant, change clothing immediately to avert fire hazards.

3. Wear only cotton garments, treated with fire resistant chemicals and equipped with non-metallic fasteners. Pockets shall be skeleton-type and trousers shall not have cuffs.

4. Do not wear silk, wool, rayon, or other garments known to produce static electricity.

5. Practice personal hygiene at all times.

6. If exposed to flammable or toxic materials, take a shower at the end of your shift.

7. Inspect all other employees when leaving change house or entering work area to be sure that each operator is properly clothed and wearing conductive safety shoes.

8. Operators in propellant operating buildings shall wear non-sparking safety shoes. All persons entering an area where conductive flooring is installed shall be equipped with conductive safety shoes. These shoes must meet the requirements in Chapter 43 of OP 5, Volume 1.

9. Persons employed in dusty atmospheres shall be provided with and required to wear respirators approved by U. S. Bureau of Mines for use under specific conditions. See Chapter 3 of this publication. Dust concentration, expressed in milligrams per cubic meter of air, shall determine the necessity for this equipment.

MAINTENANCE

Proper maintenance on a regularly scheduled basis is essential to safe operations. Regular schedules for checking, adjusting, and repairing equipment shall be established.

WARNING

Only trained and competent personnel shall **a**djust, clean, and repair propellant operating equipment.

Chapter 11 HAZARDS

GENERAL

This chapter will delineate the hazards which are not considered in the procedural information contained in other chapters of this publication. The reduction and correction of hazards in explosive, pyrotechnic, and propellant work can only be realized by good housekeeping practices. Secondly, supervisors and operators must question and analyze the information in published reports to determine whether a possible hazardous condition might arise. They shall use every precaution to avert and correct a possible hazardous condition.

Hazards are caused by a chain of events. Keep this chain broken by proceeding in the following manner.

1. Be on the alert and always assume that an operation is hazardous until the problem is alleviated by proper safety precautions.

2. Do not adopt a fatalistic attitude with regard to your work. Yours is a very precise and exacting profession, and has no place for the fatalist.

3. Perform your work only during the first six hours of the working day; if you feel physically tired or emotionally unstable, disqualify yourself from all explosives operations.

4. Do not become a hazard in your self.

PHYSICAL EXPLOSION HAZARDS

Some physical explosions in the laboratory are due to failure of evacuated glass vessels under pressure. Glass in which differences between internal and external pressures can not be used unless adequate precautions are taken against the hazards of flying glass. These equipments include sealed reagent bottles, Dry-Ice containers, and vacuum equipment.

SEALED REAGENT BOTTLES. Long stemmed bottles, such as those used for bromine packaging, are kept thoroughly chilled in an ice bath prior to opening. The stems should protrude above the bath. Open this type of bottle in the following manner:

1. Use a sharp file to make a scratch near the top of the exposed stem.

2. Break the tip off with a sharp tap.

To reseal the bottle, proceed as follows:

1. Keep the bottle cold as above, and heat the neck with a hand torch. Then pull it out and seal it.

2. Call upon the glass blower for necessary help.

<u>DRY-ICE CONTAINERS.</u> Dry-Ice or liquids that boil below room temperature must never be placed in containers with tight-fitting closures. The container must be designed to withstand developed pressures. Containers stored for extended periods and then removed from storage and allowed to warm rapidly at room temperature have been known to develop sufficient pressure to burst the container.

To prevent a dry-ice container from bursting, loosen the stoppers immediately upon removing the containers from cold storage.

VACUUM EQUIPMENT. Most accidents involving vacuum equipment are caused by the implosion of glass equipment. The resultant hazards are flying glass and burns or scalds from the hot or corrosive material in the collapsed vessel.

The inward leakage of incompatible materials (air into hot combustible vapors or water into concentrated sulfuric acid) can also be hazardous. Balloon flasks, vacuum receivers, and tubing are designed symmetrically with outward convex surfaces to withstand pressure from the outside.

Walls are thickened in vacuum flasks and desiccators to provide added strength.

Erlenmeyer flasks, beakers, volumetric flasks, and French square bottles are very likely to collapse under vacuum.

Sealed vacuum units including Dewar flasks and vacuum-jacketed rectifying columns are under vacuum at all times and shall be regarded as potentially hazardous.

As in all hazardous operations, protection against the most hazardous situation is imperative.

<u>Precautions</u>. The following precautions are set forth for protection against physical explosions:

1. When using the above equipment, look out for scratches or flaws in the glassware, mechanical shock, or sudden changes in temperature that might increase the chance of collapse.

2. Wear safety glasses or goggles as necessary to protect from flying glass and liquids.

3. Wear goggles when working with any eye-irritant or hot liquid under vacuum.

4. Place a safety shield in direct line between the apparatus and the operator.

5. When size, shape or location of apparatus prevents the use of a safety shield, wrap the glass vessel in an asbestos blanket, surround it with wire screening, or coat with plastic.

6. Always protect Dewar vessels and vacuum apparatus with permanent casings.

7. Consider remote operations from a protected location.

8. Be sure that the values leading to a vacuum source are OPEN before applying heat to any apparatus believed to be under vacuum.

9. Always bear in mind that pressure differences obtained with a water pump or a stream aspirator are nearly as great and essentially as hazardous as pressure differences obtained with an oil or mercury vapor pump.

ELECTRICAL HAZARDS

The following precautionary measures are to be taken by installers of electrical equipment and laboratory personnel to minimize electrical hazards:

INSPECTION. Inspect all electrical equipment on installation and at periodic intervals. Should a question arise as to a unit's safety or reliability, do not operate any electrical equipment until it has been inspected, repaired, and approved by a competent inspector.

LOCATION. When electrical equipment is moved to a new location, the inspector must ascertain that the provided circuit can carry the additional load.

CONNECTIONS. Make shift connections between non-mating plugs must be strictly forbidden. Do not do anything to nullify the explosion-proof feature of any electrical wiring.

<u>SPECIAL GROUNDING</u>. Oscillographs and vacuum-tube voltmeters shall not be grounded in a standard manner. Identify this equipment, and exercise special care in its use and grounding procedures.

EQUIPMENT IN HAZARDOUS AREAS. Permit only explosion-proof electrical equipment in hazardous areas. Make certain that all electrical equipment, including instruments, meet **the** area requirements before installation.

<u>HOODS.</u> Use stirrer motors or heater thermostats which do not make unconfined sparks in laboratory hoods and on benches where flammables are being handled. Use pneumatic stirrers and steam heat whenever possible. SHOCK. Report all cases of electric shock to the supervisor. Remember that physical contact with as little as twenty volts may result in loss of muscular control. Many fatalities per year result from contact with the standard 115 - 120-volt household power supply.

<u>GLAS-COL HEATING MANTLES.</u> These units are not "liquid-proof" and must be used carefully. Minimize the shock hazard by fastening the ground clip from the mantle cord to metal equipment in contact with the mantle. If a mantle becomes wet, avoid shock by disconnecting the mantle until it has thoroughly dried. Guard against ignition of flammable liquids; ignition can be caused by a hot surface or object.

STATIC CHARGES. Static charges may develop through motion of solids, liquids, and gases. Consider carefully any static hazard in handling of flammable liquids. Use standard antistatic equipment as static dissipating belts, drum fittings with grounding clamps, and grounded steam hoses.

RADIATION HAZARDS FROM RADIO OR RADAR TRANSMITTERS. Tables 7, 8, and 9, respectively, indicate the minimum safe distances at which explosives operations can be performed from FM mobile transmitters, radio transmitters, and radar transmitters.

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MINIMUM DISTANCES VERSUS FM MOBILE TRANSMITTERS

Transmitter Power (Watts)*	Minimum Distance (feet)
$ \begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	5 10 15 30

* Induced current resulting from mobile-type FM radio transmitters up to five watts RF output can be disregarded as a safety hazard.

Table 8

MINIMUM DISTANCES VERSUS RADIO TRANSMITTERS

Transmitter Power (Watts)	Minimum Distance (feet)
$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	100 200 500 1,000 2,000 5,000 10,000

Table 9	ble 9
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MINIMUM DISTANCES VERSUS RADAR TRANSMITTERS

Transmitter Power (Watts)	Minimum Distance (feet)
5 - 25 $25 - 50$ $50 - 100$ $100 - 250$ $250 - 500$	100 150 220 350 450
500 = 1,000 $1,000 = 2,500$ $2,500 = 5,000$ $5,000 = 10,000$ $10,000 = 25,000$ $25,000 = 50,000$ $50,000 = 100,000$ $100,000 and up$	650 1,000 1,500 2,200 3,500 5,000 7,000 7,000

TOXICITY AND SKIN HAZARDS

All chemicals must be regarded as toxic unless widespread usage and toxicity studies have proved them harmless under handling conditions. There are no chemicals that can be ingested intemperately without danger. More than one-half million chemicals are described in available literature, and only a few have been analyzed sufficiently to determine their toxic properties.

Poisonous materials can accidentally enter the human system in three ways. These are: (1) dusts, fumes, vapors, mists, and gases through the respiratory tract; (2) swallowing with food, saliva, or water; and (3) absorption through the skin, or through breaks in the skin. Results of poisoning by any of these routes can be acute or chronic. In acute poisoning, the damage is usually readily apparent; in chronic poisoning, the effects may not show up for years.

WARNING

Avoid ingesting toxic materials through the mouth. Skin afflictions account for the largest number of occupational disease cases. Differences in individual susceptibilities to this type exposure make it very difficult to set up standards for safe handling of any chemical.

WARNING

Wash the hands and face thoroughly and frequently when exposed to chemical dusts or solutions.

WARNING

Avoid long contact with even those chemicals which are supposedly harmless, as many of these are readily absorbed through the skin. A change in solvent may nullify the protective action of skin oils and make even short exposure harmful. For example, a short exposure to an aqueous solution may be harmless, while the same exposure time to the same agent dissolved in methanol or trichloroethylene may produce a major lesion.

WARNING

Wear glasses, aprons, face shields, and protective creams to prevent contact with the body. Report any sign of skin irritation to the doctor. In working with chemicals of known hazardous type, consult another person who is familiar with the materials or classes of materials. The toxic limits in this publication are intended for general guidance. Obtain definite confirmation on the use and handling of certain chemicals.

WARNING

Assume that all chemicals are toxic.

HAZARDS OF TOXIC GASES

The contamination of air in working areas by any mist, vapor, gas, or dust must be considered dangerous, even in cases of low concentrations. In most cases, the degree of contamination in air supposed to be safe for prolonged or repeated exposure is uncertain.

Inhalation of some gases will, in a matter of seconds, cause serious injury or death. In the case of other gases, reaction may develop gradually over a period of years. The result can be irreparable damage.

WARNING

A mild anaesthesia or unsuspected intoxication can impair judgment and cause an accident. An unpleasant odor is not necessarily evidence of the presence of toxic materials. However, it may be sufficiently distracting to become a contributing factor in a seemingly unrelated accident.

An operator must proceed as follows if he notices a doubtful odor.

1. Locate the source and correct the situation immediately.

2. Call upon your supervisor if necessary.

NOXIOUS MATERIALS. From the respiration standpoint, noxious materials may be classed as irritants, asphyxiants, and systemic poisons.

Irritants. Irritants can affect any part of the respiratory system: nose, throat, lower air passages, or lungs. Examples follow in Table 10.

Table 10

Ammonia	Halogen acids
Acetic acid	Hydrogen sulfide
Acetic anhydride	Nitrogen oxides (except nitrous)
Active-halogen organic	Ozone
compounds Acrolein Arsenic compounds Boron hydrides Bromine Chlorine Dimethyl sulfate Formic acid	Phosgene Phosphorus chlorides Sulfuric acid Sulfuryl chloride Sulfur dioxide Sulfur halides Thionyl halides

Asphyxiants. These agents interfere with the supply of oxygen to the body tissues. They may be (1) gases which merely displace the free oxygen in the atmosphere, (2) gases which act on the nervous system to paralyze the respiratory center, and (3) gases which change the blood or body tissue chemically so that oxygen reaching the lungs is not used. They are listed in Table 11.

Table 11

ASPHYXIANTS

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Any gas other than oxygen Hydrogen sulfide Carbon monoxide	The following volatile cyanides: Hydrogen cyanide Cyanogen Cyanogen chloride Acetonitrile Acrylonitrile
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Systemic Poisons. These agents are absorbed through the respiratory system, through the skin, enter the blood stream and affect a site other than the point of contact. They are listed in Table 12.

Table 12

Alcohols	
Aldehydes	Ethers
Antimony and its compounds	Halogenated hydrocarbons
Aromatic amines	Ketene
Arsenic and its compounds	Ketones
Benzene	Lead and its compounds
Boron hydrides	Mercury and its compounds
Butane	Metal carbonyls
Carbon disulfide	Organic nitrocompounds
Carbon tetrachloride	Phosphorus
Cycloparaffins	Selenium and its compounds
Esters	Styrene
	Toluene

SYSTEMIC POISONS

PREVENTIVE SAFETY MEASURES

While toxic gases vary widely in specific effects, the basic essentials of avoiding trouble are basically the same, as follows.

AIR SUPPLY. Supply adequate amounts of fresh air to working areas.

<u>CONTAMINATION</u>. Avoid contamination of air in work areas. Perform experiments with toxic gases in well-ventilated hoods which exhaust so that fumes cannot enter working areas. Check efficiency of hoods frequently. Increase space velocity by partially closing the hood damper.

USE OF BREATHING APPARATUS. Know how to use gas masks and other breathing apparatus and use them whenever there is a danger of inhaling toxic gases. Know the limitations of filter-type masks.

ALARM SYSTEM. Give alarm if toxic gases are released into any working area, and take the proper measures to correct the situation.

ANTIDOTES. Be sure antidotes are at hand before beginning any toxic operation.

FIRST AID FOR VICTIMS OF GASSING. Inhaled gas is usually absorbed instantly by the blood and circulates a moment later to the brain. Injury (anaesthesia, nerve paralysis, or cessation of breathing) is possible within seconds. Man has limited control over his breathing. He can hold his breath only 30- to 60-seconds after inhaling, and considerably less after exhaling. Hence, even with adequate warning, a person may be unable to avoid breathing a poison. If the poison causes breathing to stop, the victim will probably die from lack of oxygen even though the poison in the air were to be removed immediately. The life of a gassed victim is often in the hands of the first person to arrive, and the outcome may depend on his knowledge of proper first-aid procedure. Therefore, the basic principles of first aid treatment should be practiced and understood by all operators.

HAZARDOUS MIXTURES

In carrying out any chemical reaction, do not overlook the possibility that a hazardous mixture may be used or formed. Important considerations shall be rate of reaction, possible catalytic effect of structural materials, heattransfer capacity, stability of products, and possible by-products.

Table 13, as well as other chapters in this publication, shall be consulted to refresh operators' memories about incompatible chemicals before proceeding on any work involving a new chemical reaction.

STORAGE. It must be remembered that hazardous mixtures can result during storage of chemicals. Prevent the mixing of strong acids with alkalies, sodium with water, and carbon disulfide with nitric acid. Spills, fires, and explosions near stored chemicals must be avoided.

WASTE DISPOSAL. The problem of waste disposal in the laboratory must be carefully considered. Organic residues cannot be placed indiscriminately in a waste can unless the material is sufficiently de-activated and unless the waste can contents are known.

FIRE EXTINGUISHERS. Use the right type of extinguisher to prevent secondary reactions.

WARNING

Do not use carbon dioxide extinguishers on sodium or magnesium fires. Use only dry chemical extinguishers approved for use with these materials.

Table 13 lists approximately 72 known hazardous combinations of chemicals and hazardous reactions. The list is not complete, and is included primarily for educational purposes.

Table 13

HAZARDOUS CHEMICAL COMBINATIONS AND THEIR REACTIONS

Chemicals	NOT to be Mixed With	Reaction
Acetylene	chlorine, bromine, fluorine	explosive
Acetylene	air	explosive range from 2.5 percent to 82 percent C ₂ H ₂ by volume
Acetylene	copper, silver, mercury, and metals which form sensitive acetylides	highly sensitive, explosive
Alcohol	nitric acid	highly sensitive, explosive
Alcohol	active chlorine, hypochlorites, alkyl hypochlorites	very unstable
Alcohol	silver salts, & nitric acid (explosive products form)	explosive
Amines or Ammonia	oxygen-containing anions, perchlorate, nitrate, chlorite, chlorate	hot oxidation
Ammonia	nitrous oxide	explosive
Ammonia	mercury	mercuri-ammonia oxide formation
Ammonia or ammonium salts	halogens	violently explosive
Ammonia	silver or silver salts	very unstable
Ammonia	calcium hypochlorite	hot oxidation
Ammonia	hydrogen fluoride	hot oxidation
Ammonium nitrate	acids, metal powders, flammable liquids, chlorates, nitrites, sulfur, finely divided organics, or combustibles	oxidation
Ammonium perchlorate	carbon	exothermic decomposition or explosion

10 34

Table 13 (Cont'd)

Chemical s	NOT to be Mixed With	Reaction
Cyanide	concentrated formaldehyde	a cyanhydrin forms, and may polymerize with violence (the cyanhydrin is stabilized by acids)
Cyanide s	nitric acid	explosive
Furfural and derivatives	strong acids	polymer formation
Formaldehyde	nitric acid	violent oxidation
Hydrazine	ferric oxide	ignition at 23 degrees C.
Hydrogen	chlorine (gas phase)	violent reaction
Hydrogen	air or oxygen mixtures	explosive by spark or flame
Hydrogen peroxide	lead, copper, iron, most metals or their salts (particularly thiocyanate)	decomposition of catalysts
Hydrogen peroxide	flammable liquids, combustible mā- terials, aniline, nitromethane	foaming reaction
Hydrofluoric acid	aqueous ammonia, anhydrous ammonia	heat
Hydrogen sulfide	fuming nitric acid, oxidizing gases	oxidizes; ignites at 150 degrees C.
Hydrocarbons	fluorine, chlorine, bromine, chromic acid	energetic oxidation
Iodine	acetylene, ammonia	sensitivity to impact
Ketones	hydrogen peroxide nitric acid	formation of di- and tricycloketone peroxides
Maleic anhydride	any base with heat	explosive formation of CO_2
Molten cyanides	oxidizing agents	explosive
Mercury	lead or aluminum metals	alloy formation

	rable 15 (Cont	u/
Chemicals	NOT to be Mixed With	Reaction
Aniline	nitric acid, hydrogen peroxide	oxidation
AZO compounds	organic reactants or impurities	explosive decomposition
CALCIUM metal	sodium hydroxide	detonation
Calcium carbide	water	acetylene; considerable heat
Calcium oxide	water	can boil
Carbon-activated	calcium hypochlorite	energetic oxidation
Charcoal	sulfur, sodium nitrate-black powder	explosion
Carbo n tetrachloride	electric arc phosgene in air	explosion
Chlorates, perchlorates	ammonium salts, acids, metal powders, sulfur, finely divided organic compounds, combustibles	violent oxidation, explosive
Chlor ine	ammonia, acetylene, butadiene, butane, other petroleum gases; hydrogen, sodium carbide, turpentine, benzene, finely divided metals	violent reaction
Ch lorine dioxide	ammonia, methane, phosphine, hydrogen sulfide	more violent reaction than above
Chromic acid	acetic acid, napthalene, camphor, glycerine, turpentine, alcohol, and other flammable liquids	violent reaction
Cyanide s	acids or acid salts	HCN formation
Cyanide solutions	chlorine gas	HCN formation C ₂ N ₂ formation CNC1 formation

Table 13 (Cont'd)

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Table 13 (Cont'd)

Chemicals	NOT to be Mixed With	Reaction
Mercury	acetylene	highly sensitive, explosive
	fulminic acid	
	ammonia	Mercuri-ammonia oxide formation; explosion sensitivit
Mercuric nitrate	hydrocarbon oils	violent reaction with unsaturat oils
Nitric acid	acetic acid	
(concentrated)	aniline	
	chromic acid	
	hydrocyanic acid	explosive
	hydrogen sulfide	
	flammable liquids	
	flainmable gases	
	nitratable substances	
Nitroparaffins	inorganic bases	explosive
Organic chlorides	peroxygen compounds	explosive oxidation
Organic chlorides	metals; particularly in presence of dimethylformamide, dimethylacetamide	explosive
Oxygen	oils	
, 0	grease	
	hydrogen	explosive
	flammable liquids	
	solids	
	gases	
Perchloric acid	acetic anhydride	explosive oxidation
	bismuth and its alloys	
	alcohol	
	paper	
	wood	
	grease	
	oils	

Table 1	13 (Cont	'd)
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Chemicals	NOT to be Mixed With	Reaction	
Organic peroxides	acids (avoid friction)	explosive oxidation	
White pho s phorus	air, oxygen	ignition	
Potassium metal	air	K_2O_4 formation	
	water, acids, carbon dioxide, carbon tetrachloride, other chlorinated hydrocarbons		
Potassium tetroxide	organic or combustible material	fire hazard	
Potassium	glycerine	large quantity of heat;	
ber ma ngana t e	ethylene glycol	ignition; possible fire	
	benzaldehyde		
	sulfuric acid		
Silver	acetylene	highly sensitive, explosive	
	oxalic acid		
	tartaric acid		
	fulminic acid		
	ammonium compound s		
Sodamide	moisture and air	explosive compound formation	
Sodium* and other alkaline and alkaline earth metals	carbon dioxide, carbon tetrachloride, other chlorinated hydrocarbons, water	explosive	
Sodium*	sodium peroxide	violent reactivity	
Sodium hydride*	air, water	hydrogen hazard	
Sodium monoxide*	water	fire hazard; very caustic	
Sodium monoxide* Sodium hydroxide*	chlorinated hydrocarbons	formation of chloroacetylenes	
Sodium monoxide*	solid CO_2	violent exothermic reaction	

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Chemicals	NOT to be Mixed With	Reaction
Sodium nitrite*	ammonium nitrate, other ammonium salts	decomposition gives off nitrogen
Sodium peroxide*	ethanol	neutralization or oxidation
	glacial acetic acid	
	acetic anhydride	
	benzaldehyde	
	carbon disulfide	
	chlorinated hydro- carbons	
	glycerine	
	ethyl acetate	
	furfural	
Surfuric acid or	chlorates	ignition and fire
any acid that will release chlorine,	perchlorates	
perchlorine, or permanganic acid	permanganates	
Vinyl esters	alkalies	very exothermic
Vinyl-type polymerizations		heat release

Table 13 (Cont¹d)

* The corresponding potassium compounds are equally or more dangerous.

Chapter 12 FIRE PREVENTION

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GENERAL

Fires account for a tremendous yearly toll of injuries, loss of life, and property destruction. Fires are particularly hazardous in chemical plants and laboratories. Every precaution must be taken to prevent their occurrence. General precautions presented under <u>GENERAL SAFETY PRECAUTIONS FOR</u> CHEMICAL MATERIALS in Chapter 5 apply to the problems of fire prevention.

CONTROL OF FIRE INITIATORS. The following precautions shall be observed by all supervisors, operators, and visitors to any area susceptible to fire:

1. Observe all NO SMOKING rules as posted.

2. Do NOT smoke near benches and laboratory working zones.

3. Do NOT throw matches, cigarette butts, or pipe ashes into waste baskets or other containers. Use ash trays.

4. Keep flames and sparks away from flammable solvent vapors, gases, and dusts.

5. Do not allow carbon bisulfide vapors to come in contact with hot steam pipes or hot plates.

6. Use carbon bisulfide only with special dispensation by supervisors. It is very flammable, the lethal dose is small, and sulfur dioxide, a combustion product, is very lethal.

7. If flammable vapors are present, use only explosion-proof motors, switches, lighting fixtures and electrical devices.

8. Do not place a volatile flammable in an electrically heated oven.

9. Remove all sources of ignition from all refrigerators in which flammable liquids are to be stored.

HOUSEKEEPING. Good housekeeping is an excellent fire precautionary measure. These rules shall be adhered to at all times. Practice good

housekeeping by not allowing combustible wastes, paper, cardboard, wood, and other combustible litter to accumulate.

FIRE SYMBOLS

The user of this publication shall study the material presented on this subject in paragraph 4016 of OP 5, Volume 1.

CHEMICAL AMMUNITION SYMBOLS

The reader of this publication shall study the material presented on this subject in Chapter 26 of OP 5, Volume 1.

MAGAZINE SAFETY PRECAUTIONS

The reader is referred to Chapter 5 of OP 5, Volume 1.

QUANTITY - DISTANCES

The reader shall study the material presented in Chapter 7 of OP 5, Volume 1.

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Appendix B CHECK LIST

GENERAL

The individual should prepare a check list to insure that no references are overlooked. The check list should include the material to be prepared, scale of operation, and amount or number of batches to be produced. The properties of the ingredients shall be listed. These include thermodynamic properties, heat of formation, heat of combustion, and free energy of formation Note also the references from which these properties are obtained. Also record all public abstract literature from the following.

- American Chemical Society Abstracts with subject, formula, and patent indices (1906 -);
- 2. British Chemical Abstracts (1925);
- 3. Chemisches Zentralblatt (1829);
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- 6. Information found in textbooks.

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- OSRD 2014-Compilation of Data on Organic Explosives (available on photocopy or microfilm from the Library of Congress, Washington 25, D.C.) 20390
- 5. Classified reports are available from the libraries of the following facilities:

Naval Ordnance Laboratory, White Oak, Silver Spring, Md. 20910 Picatinny Arsenal, Dover, N.J. 07801

Bureau of Naval Weapons Library, Washington 25, D.C. 20390 Defense Documentation Center, Arlington Hall, Arlington, Va. 22212 Chemical Propulsion Information Agency, Johns Hopkins University,

Silver Spring, Md. 20910

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Naval Ordnance Test Station, China Lake, Calif. 93557 Naval Propellant Plant, Indian Head, Md. 20390

Record pertinent facts from correspondence or conversations with men who have worked with the same materials. Include all opinions about ingredients' compatibilities.

The above check list is suggestive, not intended to be complete, nor should it be suitable for any specific facility. Each facility is urged to make its own check list. Some details of all information should be given, and failure to find information in any source explained. List the subjects sought in the abstract journals. The completeness of the search must result in a reliable conclusion.

Appendix C APPROVAL FOR POTENTIALLY HAZARDOUS OPERATION

CHEMICAL LABORATORIES - Room_	PILOT PLANT AREA - Bldg
Purpose of Operation:	
Materials To Be Used:	
Expected Range of Conditions: Temp.	PressureOthers
Equipment or Apparatus:	
Operation Procedure:	
Safeguards:	
Location:	
Personnel:	
Do you consider the proposed program	
respect?	
Do you consider the above safeguards a	adequate ?
Has a literature search been made of t	he hazards involved?
Submitted by:	
	d additional precautions and safeguards

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THE OWNER

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