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GLOVEBOX FIRE SAFETY

A GUIDE FOR SAFE PRACTICES IN
DESIGN, PROTECTION AND OPERATION

1967

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H. Kinner
Authorizing Official
Date: 7/17/09

Prepared by
FACTORY MUTUAL RESEARCH CORPORATION
for
UNITED STATES ATOMIC ENERGY COMMISSION

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A Guide for Safe Practices in
Design, Protection and Operation

Prepared by the Staff of
Factory Mutual Research Corporation

for

United States Atomic Energy Commission

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GLOVEBOX FIRE SAFETY

Applicability to AEC Facilities

This publication is a guide for improved fire safety of gloveboxes for handling radioactive materials. Its contents are based on an extensive literature review (separately published as TID-3578), visits by the Factory Mutual Research Corporation (FMRC) to major AEC glovebox facilities, analyses of serious glovebox incidents to date, tests, and the suggestions of approximately sixty AEC and AEC contractor personnel knowledgeable in this area.

The report summarizes results of a glovebox fire safety project initiated and sponsored by the AEC Headquarters Division of Operational Safety, funded by the AEC Headquarters Divisions of Production and Military Application, and programmatically administered by the AEC Chicago Operations Office Safety and Technical Services Division.

AEC experience attests to the capability of fire not only to destroy glovebox integrity but also to promote subsequent dispersal of radioactive contamination into areas external to gloveboxes. This document provides a comprehensive summation of the best currently available knowledge applicable to the evaluation and control of glovebox fire risks and is recommended for serious consideration and use by all those responsible for safeguarding personnel and property against fire risks on facilities using gloveboxes containing radioactive materials.

Martin B. Biles, Director
Division of Operational Safety

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PREFACE

With the close cooperation of the Atomic Energy Commission (AEC), the Factory Mutual Research Corporation (FMRC) has prepared this publication as a guide for improved fire and explosion safety in the design, construction, and operation of gloveboxes, primarily as used for handling radioactive materials. The construction of the glovebox is such that its integrity can be easily breached. Although improper techniques in changing gloves and transferring materials in and out of gloveboxes have been the principal reasons for loss of glovebox containment, fires and explosions in gloveboxes have produced the greatest losses. It is the purpose of this publication to present guidelines based on the latest state of the art, for improved fire and explosion safety.

In the guide, the purposes of fire safety have been assumed to be, in order of importance, protection of the general public from radioactive and toxic material, protection of personnel at glovebox facilities, protection against loss of containment within a room or building, and protection against property loss and interruption of operations.

SCOPE

The guide provides a general survey of equipment design, operating techniques, protection systems, and safety procedures. The better practices of various AEC contractors have been included where applicable. Recent developments in fire protection that show promise are also evaluated, including high expansion foam, halogenated agents, an explosion suppression system, and a new automatic sprinkler system which is self-resetting. Findings or tentative conclusions of FMRC research in progress or completed have been included. Where practicable, engineering criteria are provided.

The information is of value to a wide range of readers. For management, it emphasizes the importance of glovebox safety in assuring continued safe operations, and provides guidance in planning new or changing facilities. For engineers, designers, and architects, it points out fire-safety features to consider in the design of new equipment and facilities. The guide will benefit scientists and research personnel in their planning of processes and equipment to assure safe, uninterrupted work. Operating personnel can gain a better understanding of the safety principles underlying their daily work, and new knowledge to make their work even safer. For specialists concerned with fire protection, safety and health, the guide will provide useful information toward achieving and maintaining a satisfactory level of safety in glovebox operations.

Details on specific devices, systems and other particular aspects of glovebox safety are given in the various publications listed in Appendix V, References and Bibliography.

NEW DEVELOPMENTS

Several recent developments in fire protection engineering, described in the guide, offer the promise of improved fire safety for glovebox operations. These include:

1. A new recycling sprinkler system which shuts itself off automatically when fire is under control, thus reducing criticality hazards, decreasing water damage potential, and offering greater reliability against re-kindling fires (Chapter 7).
2. A high expansion foam system. A suitable extinguishing system can be designed from proven existing hardware to control and extinguish fires in large gloveboxes, glovebox lines, and in glovebox rooms (Chapters 7 and 8).
3. Halogenated (Halon 1301) agent systems for gloveboxes and possibly for glovebox rooms. Assuming the development of equipment designed specifically for this agent, its use for extinguishment of fires in flammable liquid fuels appears very effective (considerably more efficient than carbon dioxide), and it also offers high effectiveness against cellulosic fuels (Chapters 7 and 8).
4. An explosion suppression system. An approved commercially available explosion suppression system, using a halogenated agent, can suppress fires and incipient explosions in gloveboxes (Chapter 7).
5. Latest findings on plutonium fires. Information is included on the findings of recent research on plutonium fire behavior and extinguishing mechanisms (Chapter 2).

BACKGROUND

In 1959, the AEC formed an ad hoc committee to establish criteria and guidelines for the design, construction, and operation of safe, economical, and efficient gloveboxes and glovebox facilities. AEC and contractor personnel were appointed to form subcommittees covering: (a) safety and fire protection; (b) construction materials and components; (c) operations; and (d) health physics.

A mail survey, visits to various glovebox facilities, and review of available literature formed the basis for a consolidated report, Report on Gloveboxes and Containment Enclosures, TID-16020, edited by Nelson Garden. This report was recommended as an interim guide. Toward the development of more uniform practices, the committee recommended research on the suitability of various aspects of fire safety, including fire detection, manual and automatic fire extinguishment, explosion suppression, explosion venting, and glovebox structural materials. At the request of the AEC Headquarters, Division of Operational Safety, the AEC-Chicago Operations Office initiated and supervised conduct of the recommended studies.

The Factory Mutual Research Corporation was awarded a contract to undertake a research and test program. The FMRC project objective was to investigate glovebox fire and explosion hazards, and the detection and suppression

of these hazards. Available techniques and equipment were evaluated by tests, by literature search, and by visits to major glovebox sites.

ACKNOWLEDGMENTS

The preparation of this guide reflects the close working relationship between the AEC, its contractors, glovebox operators, health and safety specialists, and the Factory Mutual Research Corporation. To help familiarize the FMRC staff with glovebox facilities and their problems, the AEC-Division of Technical Information Extension, Oak Ridge, Tennessee conducted a thorough glovebox literature search. The published summary of 379 references, Fire and Explosion Protection of Glovebox Facilities, TID-3578, compiled in 1964 by S. F. Lanier, has been a valuable aid. Copies of available literature, references, and AEC accident data have also provided valuable information.

For the preparation of the guide, as well as the conduct of the research work, FMRC gratefully acknowledges the contributions of AEC-contractor personnel, including the opportunity to visit various AEC-contractor glovebox facilities. The first-hand observations and discussions with safety and operating personnel have been of considerable help in analyzing the glovebox fire and explosion problem. Information supplied to FMRC through the liaison established by the AEC-Chicago Operations Office with principal AEC glovebox contractors is also acknowledged.

Special mention of appreciation is given R. B. Smith and L. E. Oldendorf of the Atomic Energy Commission, and to many others at AEC-contractor glovebox facilities for their time and effort in reviewing drafts of the guide and making suggestions and recommendations.

We are especially grateful to the late J. V. McNamara whose assistance, leadership and encouragement, from inception through final draft review of the guide, tangibly reflected his acknowledged competence and sincere interest in promoting glovebox safety at AEC facilities. It was his previously displayed competence and initiative in this area that motivated his selection to administer the AEC contract program under which this guide was prepared.

1. INTRODUCTION

To handle toxic or radioactive materials safely, it is necessary either to enclose the operating personnel or to enclose the hazard. Enclosing the operator requires protective clothing, respirators, etc., which are cumbersome and result in reduced efficiency. Enclosing the hazard is more effective since it permits the operator to work under near normal conditions. A glovebox is a special type of enclosure used in Atomic Energy Commission work and elsewhere. Its purpose is to enclose and control a hazardous operation or process, and to separate it from the environment of operating personnel. For some operations a glovebox may be used to provide a special environment, such as an atmosphere with controlled moisture and oxygen content.

In the AEC program, gloveboxes are used principally to handle toxic, pyrophoric, and radioactive materials. Usually, the radioactive materials are those which emit low-penetrating types of radiation (generally, alpha-emitters). For these, the usual glovebox construction components provide an adequate radiation shield. At times, radioactive materials that emit high-penetrating types of radiation are handled. These materials (except when handled in very small amounts) will generally require special radiation shielding to protect operating personnel.

Glovebox work at AEC facilities varies over a wide range and involves a variety of equipment. It encompasses research, development, production, and weapons fabrication activities. Operations include wet and dry chemistry, metallurgy, nuclear fuel development and preparation, and fuel element fabrication. Metallurgical work includes melting and casting, machining, rolling, blending, sieving, granulating, drying, pelleting, grinding, pressing, heat treating, welding, and brazing. Metals handled include plutonium, uranium, beryllium, and their alloys. Alkali metals include sodium, sodium-potassium alloy, and lithium. Figs. 1-1 through 1-4 are illustrative of the many glovebox types and uses at AEC-contractor facilities.

To avoid the spread of radioactive contamination from the glovebox to the operating area, the atmospheric pressure within a glovebox is usually negative with respect to the pressure in the operating area. Thus, if the glovebox is not gas-tight or if a leak should develop in a gas-tight glovebox, the leakage will be into the glovebox and not into the operating area.

Plutonium is an example of a highly hazardous material handled in glovebox systems in AEC work. It is radioactive; and toxic and pyrophoric in certain forms. If the plutonium handling glovebox system is properly designed; if proper operating procedures are followed; and if glovebox integrity is maintained, this hazardous material can be safely handled and worked, and the hazard will remain within the glovebox. However, if a leak develops, the in-leakage of air from the operating area may change the atmospheric condition within the glovebox sufficiently to set the stage for a plutonium fire.

Certain glovebox construction components are combustible - rubber gloves, plastic pouches, and where used, plastic windows. Thus, the glovebox is vulnerable to involvement in fire, which, in turn, can cause loss of integrity.

Fig. 1-1. Plutonium metallurgy glovebox facility. (Los Alamos Scientific Laboratory)

Fig. 1-2. Metallography glovebox line in plutonium research facility. At right, two glass-fiber-reinforced boxes; at left, aluminum enclosures. (Argonne National Laboratory)

Fig. 1-3. Glovebox facility for recovery and purification of plutonium and uranium isotopes. At right, recovery operations. At left, enclosed evaporator. Note sprinklers at ceiling. (Oak Ridge National Laboratory)

Fig. 1-4. Enclosure, within a hot cell, for confining and handling high-level radioactive material by remote control. (Argonne National Laboratory)

A fire or explosion may cause gradual or sudden pressurization of the glovebox, which can cause its components to fail. Filters or ductwork in the ventilation system are also vulnerable to damage by fire or explosion.

The control and extinguishment of fires in gloveboxes is complicated by the enclosure and the need to apply extinguishing agents without pressurizing the glovebox. Overpressurization could spread radioactive contamination to the operating area if the glovebox is not leak-tight or not strong enough to withstand the pressure. Where plutonium, enriched uranium, or other fissile materials are handled, special care is needed to avoid introducing moderator-type extinguishing agents (such as water) in a form or manner that could create a potential for nuclear criticality. Where chemicals, reactive metals, etc., are handled, care is needed to avoid introducing incompatible extinguishing agents that could cause an adverse chemical reaction (e.g. water and sodium) resulting in overpressure or explosion and loss of glovebox containment.

A glovebox fire that generates heavy smoke and soot can quickly clog high efficiency filters in the exhaust system. If this occurs, the pressure in the glovebox may change from negative to positive, and radioactive contamination may spread into the operating area. Such fires must be quickly and effectively extinguished.

Explosions within gloveboxes can easily breach glovebox containment by blowing out gloves, pouches, and windows. If explosions occur in the ventilation system, they may blow out exhaust filters, thus permitting the spread of radioactive contamination to the environment. In glovebox operations where an explosion potential exists, therefore, it is important that all practical measures, such as the use of inert atmospheres, be taken to prevent explosions, and to minimize and control their consequences.

ANALYSIS OF LOSS EXPERIENCE

Twenty-four fires and nineteen explosions involving gloveboxes have been studied. All occurred at AEC facilities during the ten-year period 1956 through 1965. They are listed and briefly summarized in Appendix I. The more significant ones have been described, with their lessons, in Serious Accidents Bulletins prepared by the AEC and listed in Appendix V, Bibliography.

Total loss from these incidents was \$1,232,000. No loss of disaster proportions has occurred to date, although one fire that lasted only 12 minutes caused a loss of more than \$800,000 because of the spread of contamination. Twenty-four incidents resulted in significant contamination outside the glovebox, and at least seven caused nonfatal injuries to personnel.

Materials involved in the fires and explosions were:

<u>Material</u>	<u>No. of Fires</u>	<u>No. of Explosions</u>
Metals	9	4
Flammable liquids	7	3
Ordinary combustibles	6	2
Reactive chemicals	2	3
Fuel gas, hydrogen	0	2
Overpressures and implosions	$\frac{0}{24}$	$\frac{5}{19}$

Plutonium was involved in nine of the 13 incidents involving metals. In four of these cases the plutonium reacted violently with chlorinated degreasing agents. Flammable liquids included acetone, methanol, kerosene, and various oils. Ordinary combustibles included rags, paper, plastics, wood, and dusts. Solvents and organic resin reacted with nitric acid to cause fires and explosions.

Ignition sources of the fires and explosions were: hot plates and hot surfaces of other equipment (9), reactions of plutonium with chlorinated degreasing solvents (4), reactions of metals with air or moisture (4), chemical reactions (4), open flames, overheated liquids, frictional sparks, combustion sparks, and static electricity (2 each), other miscellaneous (4), and unknown (3). Five additional incidents involved overpressure or implosions of gloveboxes or equipment within them.

Conclusions

1. Both fires and explosions have repeatedly caused loss of glovebox containment, with resultant spread of contamination and/or injury to personnel. Most could have been prevented by following recognized safety procedures.

2. Loss of containment was generally due to either:

- a. burnthrough of combustible glovebox components (especially windows, gloves, and plastic pouches), or
- b. failure of such components to withstand pressures developed by explosions or flash fires.

3. The loss experience and the serious loss potential show the need of continued efforts to reduce the combustibility of glovebox components, and to improve the ability of the enclosure to withstand, suppress, or safely relieve explosion pressures.

4. Provision of inert atmospheres or fixed fire protection systems are usually justified where flammable liquids or pyrophoric metals must be handled in gloveboxes. Where feasible, inert atmospheres are preferred since they greatly reduce the probability of a fire or explosion.

2. GLOVEBOX FIRE AND EXPLOSION BEHAVIOR

A knowledge of the behavior of glovebox fires and explosions is essential to their prevention, detection, and suppression. In the interest of adding to this knowledge, a number of test programs have been conducted. The more important test findings are summarized here, followed by a discussion of combustion explosion behavior in closed containers.

FACTORY MUTUAL RESEARCH CORPORATION TESTS

The Factory Mutual Research Corporation conducted an extensive series of experiments in 1965 with the fuel types and glovebox sizes commonly found in glovebox operations.

Description of Test Gloveboxes

The work was performed in four gloveboxes with volumes of 10 cu. ft., 24 cu. ft., 67 cu. ft. and 180 cu. ft. Proportions, location of gloves and windows, ventilation systems, gloves and glove port assemblies, ventilation and pressure monitoring equipment, and AEC-approved fire resistant filters conformed to existing AEC containment systems. Windows were of 1/4-in. wired glass (to permit repeated fire tests) which remained essentially leak tight during the fire tests.

Description of Fuels

The three classes of fuels used were: flammable liquid, cellulosic, and metallic.

Ethyl alcohol and heptane (representing "clean" and "smoky" flammable liquid fuels respectively) were tested in shallow metal pans. The pans provided specific surface areas from 0.06 to 9.0 sq. ft. Fuel depths (not an important factor) were from 0.2 to 0.35 in.

Paper towels, commonly used in gloveboxes, were selected as a representative cellulosic fuel. A range of fire sizes was developed by increasing the number of towels (from 3 to 48). Towel size was 10 7/16 in. x 14 in. folded. Total fuel weight varied from 16.5 gm to 264 gm.

A standardized mixture of magnesium turnings, chips, granules, powder, and ribbon was used to represent metal fires. The mixture was heaped 1 1/2 in. deep in a shallow metal pan 6 1/4 in. diameter x 3/4 in. deep; fuel weight was 154.9 gm. In a separate evaluation of extinguishing agents for plutonium fires, cerium was used as the best available substitute for plutonium.

Conclusions

1. Flammable Liquid Fires

Flammable liquid fires within air ventilated gloveboxes may cause loss of glovebox integrity by the rapid increase of internal pressure or by the cumu-

lative heat effects on glovebox components (especially rubber and plastic components). Heat-detector-actuated fire control systems function too slowly to assure complete prevention of any positive pressure buildup in boxes used for handling flammable liquids at a temperature above the flashpoint.

The maximum internal pressure caused by flammable liquid combustion in air-ventilated gloveboxes is influenced by numerous variables, particularly fire properties of the specific flammable liquid, fire size, box size, and pressure relief design. However, for any specific set of conditions, the pressure was found related to the fire heat production rate by the equation -

$$P = KH^x$$

where P represents pressure
H represents heat output rate
K and x are constants

The test fires were conducted in gloveboxes ventilated with air at controlled rates to provide initial internal pressures ranging from -0.25 to -0.80 in. H₂O. Under the specified test conditions it was found that:

a. During combustion of the fuels, maximum positive pressure ranged from .02 to 22.8 in. H₂O; time to reach maximum pressure ranged from 2 to 180 sec; the duration of positive pressure during combustion lasted from 0 to 70 sec; fire duration ranged from 15 to 625 sec; maximum temperatures at the glovebox ceiling ranged from 150°F to 945°F while the time above 200°F ranged from 0 to 804 sec.

b. Varying the ventilation rate had little influence on the pressure created by the test fires. Closure of the ventilating air intake, after test fire ignition, led to a marked reduction in fire duration but a slight increase in maximum pressure. Increasing the venting capacity of the exhaust equipment (filter, ducts) tends to decrease pressure rise.

c. In general, flammable liquids cannot sustain combustion in atmospheres containing less than 15% oxygen. Ethyl alcohol fires of a size greater than one-sixth the box floor surface area were found to be self extinguished (through oxygen depletion) in 15 sec. or less. By contrast, cellulosic and metal fires may be sustained at oxygen concentrations under 15%.

d. Duration of combustion is related directly to the amount of fuel present.

2. Cellulosic Fires

Cellulosic fires in air-ventilated gloveboxes were studied in tests that, except for the fuel, paralleled those discussed above. These tests indicated that the rate of fire spread in cellulosic materials was comparatively slow. Neither the rate of pressure rise nor the maximum pressure (about + 1 in. H₂O) were found to warrant serious concern. The test fires did not cause filter

clogging problems to a degree significantly influencing fire behavior. Cellulosic fires may threaten loss of glovebox integrity because of heat effects on glovebox components (especially rubber gloves) or by spread of fire to other combustible materials within the box.

Noncombustible high efficiency particulate filters provided the sole means for relief of pressure built up during glovebox fire tests. No evidence of complete filter failure was noted during these tests which involved exposures of up to 685°F for 10 minutes. Smoke generated during cellulosic test fires did not cause filter clogging to a degree significantly influencing test results.

3. Metallic Fires

Test fires using magnesium chips and turnings caused no significant change of the ambient box pressure or harmful temperature rise. Much greater filter clogging was caused by smoke from magnesium fires than by smoke from cellulosic fires.

Extinguishing agents for plutonium fires were evaluated, using cerium blocks as the best available substitute. Agents were applied by pouring and by placing Teflon or polyvinyl chloride plastic bags containing the agent on the burning cerium. Applications were made once only, following a pre-burn period of 20 minutes. The relative effectiveness of the agents was compared, based on their ability to cool the metal and on the recoverable metal remaining after extinguishment. Under these conditions, copper metal powder (30 to 60 mesh) and magnesium oxide sand (a material now widely used at AEC facilities) proved to be the most effective of the eleven agents tested. For most agents tested, use of plastic bags improved extinguishing efficiency. Note: caution must be used with plutonium fires, since the weight of the extinguishing agent may cause the molten metal to spew (21).

Special tests were also made with carbon dioxide and monobromotri-fluoromethane (Halon 1301). Neither was an effective extinguishant, but under the test conditions, there was no evidence of chemical incompatibility of these gases with the burning metal.

Agents were also evaluated on fires in cerium turnings. Ternary eutectic chloride (TEC) (a mixture of barium, sodium, and potassium chlorides) and ternary eutectic fluoride (TEF) (a mixture of sodium, potassium and lithium fluorides), were found to be the most effective extinguishants when applied by pouring. This conclusion was in agreement with findings previously reported by British workers (11, 38).

Because of physical and chemical differences between cerium and plutonium, cerium turnings cannot be considered an ideal substitute for plutonium turnings. The evaluation of agents on both cerium block fires and cerium turning fires was considered to be a screening evaluation that could serve as the basis for an evaluation to be performed on actual plutonium fires. Note: Ignition and combustion properties of plutonium and its alloys are detailed in Chapter 6 of the Plutonium Handbook, O. J. Wick, Editor (see Bibliography).

ATLANTIC RICHFIELD HANFORD CO. TESTS OF PLUTONIUM METAL

Dr. R. E. Felt, of the Plutonium Chemistry Group, Atlantic Richfield Hanford Co. (formerly Isochem, Inc.) conducted a series of tests to study plutonium fire behavior and determine the most effective extinguishing agent for plutonium fires. The results are available in a publication entitled Burning and Extinguishing Characteristics of Plutonium Metal (21).

For these tests, plutonium was in the form of solid mass, turnings, casting skulls, and hood waste. Quantities varied from about 150 gm to 2,000 gm. Tests indicated:

1. The peak burning temperature is directly related to the ratio of surface area to mass; i.e., the burning temperature is higher for smaller particles.
2. The only effective methods of extinguishing plutonium fires are complete seclusion from oxygen and rapid heat removal to below the ignition temperature.
3. Oxygen exclusion can be attained by the localized application of argon or helium gas, or preferably by complete inerting of the glovebox. Argon extinguishment is only effective when its application reduces the oxygen in the atmosphere below 4%. It is not feasible to attain this by merely flooding the air atmosphere of a glovebox with argon.
4. Complete oxygen exclusion cannot be attained by fusible salts or metal powders.
5. The most effective solid extinguishing agents based on heat removal are copper powder and magnesium oxide sand (MgO).
6. Undiluted Halon 1301 gas applied to plutonium metal fires causes a rapid metal temperature increase.
7. Plutonium metal fires are difficult to maintain on a heat-conducting surface. Stainless steel or aluminum alloy glovebox floors are effective "extinguishing" surfaces for small fires. The heat removal property of metal floors is a significant factor in fire control and extinguishment.
8. No alloying or other undesirable effects resulted from the plutonium burning on stainless steel or aluminum glovebox floors. The burning metal discolored these floors but did not distort them or burn through.
9. A massive piece of plutonium is normally difficult to ignite. When it is ignited and burns, a fragile shell of oxide and metal maintains the shape of the piece while burning, and the plutonium inside the shell liquefies. Slight disturbance causes this low viscosity inner mass of molten plutonium metal to spew from the shell. Rapid extinguishment in such cases can often be obtained by spreading the burning plutonium (i.e., the liquid) on a cool glovebox floor.

10. Containment of a plutonium fire is the primary consideration, in order to prevent: (a) spread of the fire to other combustibles or flammables that may be in the glovebox; and (b) spread of the fire to combustible glovebox construction components, such as rubber gloves or plastic windows.

ARGONNE NATIONAL LABORATORY TESTS

At the Argonne National Laboratory in 1961, a series of fire and explosion tests were made by H. V. Rhude to evaluate fire resistance of a new glovebox design and to obtain information on fire control (62).

The glovebox used for the test had a 1-in.-thick aluminum baseplate, 1/2-in.-thick aluminum panel at one end, sloping 3/8-in.-thick safety glass windows in front and back, and a glass panel on top and at one end, with an enclosed volume of 17.7 cu. ft. A summary of test findings follows.

Flammable Liquid Fires

Nine tests were made using trays containing 50-ml batches of alcohol under varying conditions. The investigators concluded that:

Over 10% oxygen was needed for the combustion of alcohol in the glovebox.

When there is no pressure relief, as when the exhaust ventilation is closed off, the pressure buildup will very quickly burst the gloves.

When there is adequate pressure relief, but essentially a static atmosphere in the glovebox, the fire quickly smothers itself without bursting the gloves.

Metallic Fires

Fifty-gram batches of magnesium chips were used. The chips were heated to the ignition point by a nichrome heater. It was noted that:

The chips will burn with as little as 1% oxygen if additional heat is furnished, but normally will not burn freely until over 5% oxygen is supplied.

The heavy aluminum baseplate absorbs enough heat to greatly retard the burning rate.

Explosions

Two tests were made using a mixture of 10% hydrogen in air. In the first test the gloves were not covered. One glove was blown off intact; the other three were ruptured. In the second test the gloves were covered with steel gloveport covers. In this test the front window was shattered by the explosion.

The investigators concluded that if the glovebox had contained plutonium, contamination would have been widely scattered in both explosions. The explosion without the gloveports covered did much less damage to the glovebox and would have been less injurious to anyone near it. In this case, the gloves acted as explosion vents.

HANFORD LABORATORIES TESTS

At the Hanford Laboratories in 1961, various types of fires and methods of controlling them were investigated by R. R. King, in a test glovebox equipped with standard high efficiency inlet and exhaust filters and connected to the main building glovebox exhaust system (46). The more important findings were:

Oil Fires

Fires in peanut oil, lard oil, and transformer oil were rapidly and safely extinguished with a dry chemical extinguisher. The dry chemical was discharged from an extinguisher through a quick-connect coupling arrangement on the glovebox wall. Similar fires could not be extinguished by carbon dioxide, argon purging, asbestos blankets, or glass fiber blankets. It was concluded that a glovebox floor area up to 18 sq. ft. could be protected with one 5-lb.-dry chemical extinguisher discharged through a single coupling (Fig. 7-4).

Filter Fires

Oil vapors in exhaust gases may rapidly saturate fire resistant filters. A relatively small fire in the glovebox may ignite the saturated filter media, allowing fire to spread to the ductwork and endanger the entire facility. Use of a stainless steel wire mesh pre-filter, preferably coated for corrosion resistance, has two major advantages. First, it acts as a vapor or mist eliminator, and secondly it acts as a fire stop, helping to prevent a glovebox fire from entering the exhaust system.

Metal Fires

In tests using magnesium turnings, magnesium oxide sand was found to be an effective extinguishing agent. Argon purging was unsatisfactory, although it did retard the burning rate.

Glovebox Combustibles

Various paper products, rubber gloves, and plastic bags were ignited in the test box. In all cases the dry chemical extinguishers safely and efficiently extinguished the fire. Tests comparing fire behavior of glovebox panel materials could not be completed because in each case the combustible exhaust filter ignited, requiring that the exhaust be disconnected.

Conclusions and recommendations from these tests were:

1. The glovebox fire hazard cannot be eliminated; but good housekeeping inside the glovebox is the most effective single method to minimize the fire problem. Good housekeeping includes minimum solvent storage and immediate removal of combustible waste.
2. Gloveboxes should be equipped with quick couplings for dry chemical type extinguishers.

3. Stoppered containers of MgO sand should be stored in conspicuous locations inside gloveboxes for use on metal fires.

4. A stainless steel wire mesh pre-filter installed with a glovebox exhaust filter will eliminate oil vapor saturation and act as a fire stop between the glovebox and ductwork.

5. Periodic meetings should be scheduled with firemen and others who are likely to discover and take first action at a glovebox fire to insure familiarity with recommended fire-fighting techniques.

ORNL TESTS

Fire tests were made at Oak Ridge National Laboratory in 1961 by T. S. Mackey, using a low flashpoint solvent (49). The tests demonstrated the need for rapid extinguishment of fire to (1) prevent loss of containment by burn-through of the gloves; and (2) to prevent heat buildup that would accelerate evaporation of the solvent until an explosive concentration of vapor resulted. Such an explosion produced enough pressure (+8 in. H₂O) to knock out an entire panel of the enclosure. It was found that increased ventilation was not a satisfactory means of preventing explosive concentrations, but that the box temperature had to be below the solvent flash-point to avoid flammable vapor concentrations.

After tests of several water spray nozzles, an automatic water spray system was recommended, actuated by a heat detector and supplied from a limited volume (2-3 gal.) of water.

LAWRENCE RADIATION LABORATORY TESTS

Because of the explosion hazard during evaporation of ether from a plutonium solvent-extraction process, J. Young and W. D. Phillips at Lawrence Radiation Laboratory-Berkeley carried out a number of explosion tests with ether-nitrogen and ether-air mixtures in a glovebox of 12 cu. ft. volume (77). Spark plugs were used in the box as the source of ignition while the airflow, ether volume and hot plate temperature were all varied.

Conclusions from the test were:

1. If ether must be used in a glovebox, the following steps are recommended to insure safe conditions:

(a) Do not evaporate ether at a rate greater than 10 to 15 ml per minute with a ventilation rate of 5 CFM.

(b) If a faster rate of evaporation is planned, use an inert atmosphere, or mix the box atmosphere thoroughly during the evaporation to prevent local vapor concentrations from building up.

(c) Eliminate all sources of sparks, or heated surfaces at more than about 150°C (autoignition temperature of ether is 180°C).

(d) Secure the front window by angle brackets to the side of the box. In case of explosion, this will prevent the window from flying off into the faces of operating personnel.

2. Caution should be used in the interpretation of any published LEL value. This value is based upon homogeneous mixing of the solvent vapor and air. In practice, this rarely occurs. Consequently, "calculated safe" levels may build up local concentrations that reach explosive levels.

BRITISH TESTS

J. Holliday and W. A. Conway reported in 1962 on tests made for the United Kingdom Atomic Energy Authority to study the extinguishment of plutonium fires (38), which were summarized by S. F. Lanier as follows:

A fusible ternary eutectic salt mixture of barium, sodium, and potassium chlorides was developed as an extinguishant for metallic fissile material fires. The powder extinguished fires involving massive and finely divided uranium and massive plutonium, but was not successful with fires involving finely divided plutonium; combustion continued under the powder.

Tests indicated that a fusible ternary eutectic of sodium, potassium and lithium fluorides would act as a more efficient extinguishant and would be capable of extinguishing fires involving finely divided plutonium. The mixture was used successfully on fires involving plutonium, in both the massive and finely divided states.

L. H. Cope reported in 1963 on tests made for the United Kingdom Atomic Energy Authority in connection with the development of an agent suitable for use on burning plutonium, uranium, or thorium (11). The report was summarized by S. F. Lanier as follows:

The development of a powder suitable for extinguishing fires of burning uranium, plutonium, or thorium is described. The powder consists of a mixture of powdered inorganic chlorides and/or fluorides inert towards the burning metal.

The mixture has a melting point below the melting point of the burning metal so that fusion of the powder takes place locally where the powder contacts the burning metal. A frit is formed around the locally fused portion of the powder, excluding the ambient atmosphere from the metal. Absorption of latent heat by fusion of the locally fused portion of the powder exerts a chilling effect on the metal.

Examples of preferred powder mixtures are given. The mixture can be prepared by grinding the components together, but preferably the components are fused together before grinding to facilitate melting upon application to a fire. After a fire has been extinguished, uranium or plutonium may be recovered from the residues by leaching with water to remove the soluble chlorides.

COMBUSTION EXPLOSION BEHAVIOR IN CLOSED CONTAINERS

Whenever flammable liquids or combustible gases are present in a glovebox there is the potential for an explosion. It is significant that seven of the ten largest glovebox losses (Appendix I) have resulted from explosions. In all of the seven cases, glovebox containment was lost because of failure of windows or gloves and there was serious spread of contamination. Loss of glovebox integrity can also result from pressures produced by flash fires (as well as from their heat effects). A basic understanding of the behavior of explosions in closed containers is, therefore, of great benefit to the designers and operators of gloveboxes. This knowledge makes it possible to determine how explosive pressures can best be prevented, contained, relieved or suppressed.

Explosions involving mixtures of flammable fluids and air can be classified in three types, depending on the rate of flame propagation and magnitude of the peak pressures produced: flash fires, ordinary explosions (deflagrations), and detonations.

A flash fire develops in seconds and the pressures produced are relatively low (in the order of a few pounds per square inch). The pressures build up so slowly that in most enclosures, such as rooms, they may be relieved through the openings normally present without causing structural damage.

A deflagration develops in milliseconds. The pressures increase so rapidly that unusually strong enclosures or explosion vents are needed if the enclosure is to withstand the pressures without damage.

In an explosion, the time taken to reach a given pressure follows a cube law. For a given explosive mixture, the flame speed is initially constant at atmospheric pressure. Usually, the flame starts from a single point of ignition. It grows as a sphere whose radius is expanding at a constant rate. The pressure at any instant is thus proportional to the sphere's volume or the cube of its radius. From this radial growth of flame, at constant speed, it follows that the larger the container the longer is the time to reach a given pressure. In fact, the time taken to reach a given pressure varies as the cube root of the volume (22).

A detonation is an extremely fast explosion. The flame propagates at supersonic speeds, and pressures starting at atmospheric may reach several thousand pounds per square inch. The flame propagation velocity is so high that explosion vents are not effective. Certain conditions (generally additive) affect the probability of a detonation rather than an explosion.

1. Large ratio of length to diameter of the container.
2. High initial pressures.
3. Large ignition energies.
4. Higher than normal (20.95% by volume) oxygen concentrations.
5. The nature of the combustible gas in the container.

Maximum Pressures From Ordinary Explosions

In any combustion explosion in a closed container, the maximum pressure is directly proportional to the initial pressure.

Expressed as an equation, $P_x = KP_i$, where P_x is the maximum pressure, P_i the initial pressure, and K is a constant.

Regardless of the shape of the container, flammable gases and vapors mixed with air are capable of explosions throughout the range between upper and lower explosion limits. The maximum pressure will occur with "near perfect" (stoichiometric) mixtures.

Only very small amounts of flammable liquid are needed to produce explosive concentrations in enclosures the size of gloveboxes. For example, the evaporation of 0.4 pints of acetone into an air-filled enclosure 100 cu. ft. in size would result in the formation of an explosive mixture.

Actually, it is not necessary to fill the entire volume of an enclosure with an explosive mixture in order to have an explosion; local concentrations may be at or above the LEL. On the other hand, adequate and dependable ventilation can prevent build-up of explosive concentrations from relatively large exposed quantities of liquids. In industrial practice, great dependence is placed on ventilation to prevent formation of explosive mixtures.

A discussion of the pressures developed by explosions in closed containers and the effect of explosion venting is described in Appendix III, based on tests by E. W. Cousins, P. E. Cotton, and J. B. Smith of Factory Mutual. (11b, 66a) Information on the fire hazard properties of materials can be obtained from the Factory Mutual's Handbook of Industrial Loss Prevention (14) and from the NFPA's Fire Protection Handbook (55).

Application to Glovebox Design

The initial point of glovebox failure from excessive over-pressurization due to fire, explosion, or other causes should be determined by design as far as possible, to provide adequate pressure relief while concurrently minimizing the risk of personnel injuries and property damage. Every effort should be made to prevent personnel from being exposed to pressure relief devices designed into a glovebox.

In order to design gloveboxes to accomplish this, one must know:

1. The maximum steady-state pressure that the glovebox can withstand without rupture, with its openings blanked off. Each component (gloves, windows, walls, gaskets, etc.) must be considered.
2. The maximum pressure that can be obtained from an explosion of the materials being handled within the glovebox.
3. The pressure pulse that will result from an explosion of these materials.

These data enable one to determine:

1. Whether the box is so weak that explosion venting is not feasible.
2. What rated bursting pressure and free venting area would be expected to suppress the pressure rise sufficiently to avoid loss of containment.
3. The requirements (design strength, volume, configuration, etc.) of the enclosure or system into which venting would be directed.

FMRC tests, thus far, indicate that, in at least some cases, it would be possible by venting to reduce glovebox explosion pressures to a magnitude that can be withstood by the glovebox. It should be recognized that successful venting of a glovebox is complicated by the need to localize the spread of contamination. Further investigations are under way to determine the practicality of venting into exhaust ventilation systems.

CRITERIA FOR FMRC GLOVEBOX FIRE TESTS

Criteria for standardized glovebox fire tests, based on the test program conducted by Factory Mutual Research, are presented in Appendix IV.

3. ADMINISTRATION AND PLANNING

The overall safety of a glovebox facility depends on a proper administration, planning, and control. Clear lines of communication and authority should be set up and maintained among the many groups of people concerned: designers, fire protection engineers, safety engineers, health physicists, criticality experts, research scientists, technicians, maintenance workers, and fire fighters.

ADMINISTRATION OF THE SAFETY PROGRAM

Administration and organization of the safety program will vary with individual conditions. In general, however, the successful program will provide for:

1. Overall supervision. Defining functions, clarifying responsibilities, and making sure that all procedures are properly carried out.
2. Planning. Anticipating hazards. Formulating plans and procedures for the prevention of fire and for the overall safety of the facility. Pre-planning emergency procedures. Thorough review of plans and specifications by all concerned with the safety of the facility.
3. Education and Training. Making sure that all operators and other personnel concerned are trained in safe procedures, and that all procedures are properly and safely carried out.
4. Assignment. Delegating specific maintenance and emergency duties to employees, including glovebox operators, maintenance men, and guards or watchmen.
5. Coordination. Maintaining liaison with all departments and groups concerned with:
 - a. New construction and layout, new processes, and operational changes. Noncombustible materials should be used wherever possible.
 - b. Installation and maintenance of fire protection equipment.
 - c. Housekeeping and safe storage and handling of hazardous materials. Good housekeeping and safe disposal of waste are essential to fire prevention.
6. Review. Checking periodically the efficiency of the entire program with appropriate reviewing authorities. Keeping the program up-to-date with new developments.

OPERATING PROCEDURES

Proper written instructions designed to promote safe operations, including experimental procedures, should be detailed. These instructions should be originated and kept current by the person responsible for the

operation and they should be reviewed by supervisory and appropriate hazard control groups for all phases of safety. Instructions must be thoroughly understood by the operators and provisions made to insure enforcement of approved operating procedures.

EMERGENCY PROCEDURES

Emergency procedures, including evacuation requirements, should be pre-planned and employees instructed and trained in their application. The instructions should be coordinated with operating procedures and kept current. Further, emergency procedures for glovebox working areas should be carefully coordinated with similar procedures in effect throughout the rest of the site.

INDUSTRIAL SAFETY

A glovebox places certain restrictions on the operator that would not normally be encountered if the work could be performed in an open area. Some of these restrictions or limitations that affect safety are as follows:

1. The operator's movements and position are restricted, creating fatigue.
2. The operator's vision is restricted.
3. The operator's sense of touch is limited by working through gloves.
4. The operator sometimes has to work through gloves at elevated levels.
5. The operator's gloves are susceptible to damage by being punctured, torn, or (in event of fire) burned through.
6. The speed of operations is reduced considerably, so that tension and fatigue may be induced.

Therefore, the design of gloveboxes and equipment and the operating procedures are of concern to the industrial safety specialist.

Some glovebox and equipment design features recommended for minimizing the effects of these restrictions upon industrial safety are as follows:

1. The glovebox design should be such that the operator can reach all parts of the interior of the glovebox with the arm-length glove.
2. The glovebox window material should remain clear with good resistance to abrasion, heat, discoloration, shattering and corrosion.
3. The glovebox window should provide adequate viewing area consistent with other glovebox requirements.
4. Lighting for the gloveboxes and glovebox rooms should be arranged to avoid glare on windows from reflected light. Gloveboxes, particularly, should be well lighted.

5. Platforms should be provided for working through gloves at elevated positions. They should be rigid and immobile when in use. Guard rails may be needed in some cases.

6. Mechanical lifting devices should be provided for movement of equipment, particularly heavy equipment.

7. All sharp edges on the glovebox and equipment within the glovebox should be eliminated to avoid tearing or puncturing the gloves.

8. Mechanical guards should be provided for all moving equipment.

9. Emergency shutoffs for equipment and utilities should be located both inside and outside the glovebox at readily accessible locations. All controls and gauges should be labeled.

10. Remote control of large equipment should be provided where applicable.

11. Heating equipment should be guarded to prevent contact of gloves with hot surfaces, open flames, or electric coils.

General recommendations that apply to the glovebox room and to personnel are as follows:

1. Comfortable uniforms, aprons, goggles, or other protective clothing should be issued and maintained as required. Cotton glove liners have been issued by some facilities to absorb perspiration and prevent allergic reactions to gloves.

2. Cluttered work and floor areas should be avoided to prevent hindering movement of hands and feet. Adequate neat storage space, shelving, files, racks, etc., should be provided for chemical storage, tools, laboratory apparatus, records and books.

3. Adequate air conditioning and room ventilation should be provided for comfortable temperatures and humidity, and to remove irritating fumes, vapors and smoke.

4. Adequate exit facilities should be provided, especially from hazardous areas; all exits should be kept clear. Exits should be clearly marked. Evacuation routes to the exit should also be marked (e.g. painted arrows on floor, signs, etc.).

5. Floors should be non-slip, chemically resistant, and easily decontaminated. More than one floor coating may be required.

6. Smoking should be prohibited where flammable liquids, explosive chemicals, or combustible gases are used or stored, or where contamination is likely.

7. All wiring, gas lines, water lines, and other service lines should be installed according to code and be properly maintained. After installation, flammable gas lines should be leak tested with a halogen or helium leak

detector. Locate utility lines where rupture by impact is unlikely. Leaks, broken insulation, etc., should be promptly repaired.

8. Containers of chemicals and solvents should be labeled. Unbreakable safety containers are preferred.

9. Easily accessible first-aid kits should be provided, unless a first-aid station is nearby.

10. From an operating viewpoint, it is desirable that prolonged continuous work be avoided whenever possible.

The industrial safety engineer has areas of common interest with the fire protection engineer and the health physicist with regard to fire and explosion prevention and control, and the protection of personnel from the harmful effects of radiation. He should be called on for advice and assistance during the design of gloveboxes and glovebox facilities and during operations to assure that safety has been considered, both in design and in operating procedures. Drawings should be reviewed.

HEALTH PHYSICS AND NUCLEAR SAFETY

A health physics (radiation safety) program should be maintained to monitor personnel and areas external to the glovebox system for personnel safety and to prevent the spread of contamination.

A criticality control program should also be established and maintained to control the fissile materials handled in any operation and the movement of such materials within the facility.

Details of these programs are outside the scope of this guide. Standard references on health physics and nuclear safety are available (see H. Blatz, Radiation Hygiene Handbook; AEC Nuclear Safety Guide; and U. S. Public Health Service's Radiological Health Handbook, listed in Bibliography).

4. PRINCIPLES OF GLOVEBOX CONSTRUCTION

Glovebox design is influenced by the toxicity, form and quantity of the materials to be handled, type of operations, radiological risk, safety requirements, and economic considerations. There has been a wide diversity in design and construction. Many gloveboxes have been designed and fabricated to meet special requirements. A sketch of a glovebox used in plutonium metallurgy is shown in Fig. 4-1.

There is a strong trend toward use of commercially built gloveboxes. Although some manufacturers offer standard designs, a larger number of vendors supply gloveboxes to meet the user's requirements and specifications. One source of vendors' names is the Yearbook and Buyers' Guide issue of Industrial Research magazine, under "Laboratory Furniture", "Gloves, Dry Box", etc. Nucleonics, listing more specific glovebox categories in its buyers' guide issue, was discontinued in 1967, but back issues may be available from the publisher, McGraw-Hill.

Most glovebox operations can be performed in boxes of a relatively few standard designs. The benefits of standardization were recognized several years ago by the AEC Ad Hoc Committee (27). Standardized design and fabrication of enclosures and standardization of components result in more efficient operations, savings in time, effort, and materials, and greater safety.

GENERAL DESIGN CONSIDERATIONS

Several factors must be carefully considered in designing the complete glovebox system: size and shape of enclosure; construction materials; air flow patterns; ventilating equipment; filters; atmosphere control; lighting; glove and transfer port size and location; window size, construction and location; penetration requirements, i.e. openings for ducts, piping, filters, etc.; and fire and explosion protective equipment.

Glovebox fire tests and the experience gained from actual fires and explosions indicate that:

1. The initial point of glovebox failure from excessive internal overpressurization due to fire or other causes should be predetermined by design insofar as possible, to afford adequate pressure relief while concurrently minimizing personnel injury and property damage exposure risks (see Chapter 2).
2. The selection of structural, glove, and window materials should be made only after consideration of all safety and operational aspects of the work to be performed. Choice should be based, as much as possible, on the known and anticipated conditions (chemicals, concentrations, temperatures, moisture, type of equipment, combustibility) to which they will be exposed.
3. The use of noncombustible, heat-resistant materials for the construction of gloveboxes is one of the most important steps that can be taken to prevent failure of a containment system due to fire. Fire in a

quantity of combustible material may not offer a serious exposure threat to a glovebox if it is relatively isolated from combustible surfaces (e.g. gloves, plastic pouches, and plastic windows).

4. Proper clamping and gasketing is imperative.

A survey of AEC facilities by the Ad Hoc Committee (27) resulted in several recommendations on standardization of design and construction that are applicable to a majority of glovebox installations:

1. Viewing windows to permit observation of entire interior when operator is in operating position.
2. Window material to resist abrasion, fragmentation, heat or fire, and impairment of visibility from radiation or chemical attack.
3. Glove ports to permit operator access to entire glovebox interior. Circular 8 in. diam. ports, with provision for other sizes as needed.
4. Bag ports of 4 in., 8 in., 12 in., 16 in., and 24 in. diameters.
5. Sealed plastic bags (more desirable than air locks) for transfer operations.
6. Replacement of gloves and transfer bags without violating glovebox integrity.

GLOVEBOX STRUCTURAL MATERIALS

Glovebox designers and users generally have been free to select construction materials according to their own estimates of the hazards involved, the operational requirements expected, and their knowledge of available and suitable materials. There has been extensive experimentation with and use of a wide variety of materials. Because of the diversity of operations, no material has been found ideally suited for all applications.

Glovebox structural materials should preferably be:

- a) resistant to chemical action.
- b) noncombustible.
- c) resistant to heat.
- d) resistant to shock.
- e) resistant to surface abrasion.
- f) non-absorbent and non-adsorbent.
- g) easily decontaminated.
- h) strong mechanically.

Fig. 4-1. Plutonium metallurgy research glovebox, large size, showing service connections. (Argonne National Laboratory)

- i) resistant to permanent distortion, retentive of elasticity.
- j) resistant to radiation damage.

Stainless Steel

Stainless steel is the most commonly used structural material. Several types have been reported: 11-gauge 304-SST, 12-14 gauge 316-ELC, 321, and 347, 403 and 14 gauge 304-ELC (6, 45, 74). Advantages of stainless steel include: resistance to a wide range of solvents, alkalis and most acids (depending on the alloy composition); mechanical strength; hardness; resistance to abrasion; resistance to heat; noncombustibility; ease of fabrication; ease of cleaning and decontamination and minimal maintenance.

Disadvantages are: vulnerability to hydrofluoric, hydrobromic, hydrochloric acids, halogen salts, and sulfuric acid (variable); and high cost. Stainless steel construction is particularly suitable for long term applications; the high initial expenditure may be justified in many cases because of low maintenance compared with other materials requiring coatings.

Mild Steel

Mild steel has had less extensive use than stainless. Coated mild steel has been used where atmospheres are corrosive to stainless steel, or where economy as well as strength are desired in a vacuum box. A suitable coating is required for protection from corrosion and adsorption of activity; however coatings may be broken by impact from heavy equipment. Although mild steel is less expensive than stainless, the initial economy may be lost by additional maintenance and shorter useful life. Stainless steel or stainless clad mild steel may be used as the floor for extra corrosion resistance.

One British standard design, Mark I, is built of 1/8 in. mild steel. Bright mild steel and zinc-coated steel have also been used at British facilities (74).

Aluminum

Aluminum has also been used. It is a better heat conductor than stainless steel, thus dissipating more heat to the surroundings. It is also considerably lighter and less expensive than stainless steel. One facility has reported using aluminum frames, alloy 5456 H112 extrusion (5.25% Mg), and aluminum panels, alloy 6061-T6, with 1-in.-thick aluminum baseplate. Abrasion resistance is satisfactory; where abrasion is a serious problem, stainless steel has been used. Alloys 6061, 6062, and aluminum-magnesium alloys with less than 4% magnesium have been reported as susceptible to fine cracks adjacent to welds (45). Duraluminum, a 95% aluminum alloy with copper, magnesium and manganese, has been used at Los Alamos Scientific Laboratory (74).

Aluminum alloys are widely used for their generally high corrosion resistance. They are generally resistant to organic acids, alcohols, ethers,

esters and phenols. They are generally attacked by the halogens, halogen acids and most salts, inorganic acids (nitric below 82%) hydroxides, and compounds of copper, silver, lead, and nickel (especially in the presence of moisture). (48, 64).

Users of aluminum boxes at Argonne National Laboratory report satisfactory experience in working with plutonium. It was found that plutonium does not form very low-melting eutectics with aluminum; and that the 1 in. thick floor would be expected to dissipate the heat of molten plutonium better than the thinner stainless steel normally used.

Plywood

In past years, plywood had been widely used because of ready availability, ease of fabrication and low cost. The major disadvantage is high combustibility. Suitable coatings are usually needed to increase durability, fire resistance, and ease of decontamination. Fire retardant plywood has become available (71), but its use in gloveboxes has been limited.

Besides coating, rigid materials such as melamine, phenol-formaldehyde laminates (e.g. Formica, Micarta) and asbestos millboard have been used as liners. The Lawrence Radiation Laboratory has developed a special plywood-asbestos laminate, 3/4 in. thick, bonded with sodium silicate, for increased fire resistance (60).

Despite these efforts to improve the qualities of plywood, its use has decreased steadily in favor of other materials having superior inherent properties, suited to more diverse operations, and not requiring the modification or maintenance that plywood requires.

Glass-Fiber-Reinforced Plastics

Glass-reinforced plastic gloveboxes are being increasingly used. These are being used mainly where highly corrosive conditions are considered to preclude any structural metal, or where metallic impurities may contaminate analytical work.

Desirable properties may be obtained in glass-fiber-reinforced materials by selecting the suitable resin and the appropriate form of reinforcing material (57). Heat resistance, flammability and chemical resistance depend primarily on the type of plastic resin used. Various thermosetting and thermoplastic resins have been used, although polyesters account for over 80% of resins currently used. Other resins available include epoxies, silicones, acrylics, polystyrene, Nylon and polycarbonates.

The polyesters have varying degrees of resistance to heat distortion and flammability. The specific resin, the glass-resin proportions, and the processing method affect flammability and heat resistance. Generally, greater glass content decreases the fire hazard. Chlorinated compounds and antimony additives have been used to increase the fire and heat resistance of the resin (24, 39).

The strength of the finished reinforced plastic depends on the amount of glass fiber used and its arrangement. Strength generally increases almost

linearly as the glass fiber content is increased. However, the form of the glass fiber (continuous strand, reinforcing mats, yarn, etc.) controls the maximum amount of glass that can be used. Fiber from special types of glass (e.g. high temperature; high strength) is also available from the manufacturers at greater cost.

Reported advantages of the most commonly used glass-reinforced plastics, the polyesters (non-fire-resistant) when reinforced by glass fiber, include: hard smooth surface; ease of decontamination; high mechanical strength; fair resistance to abrasion; low absorption of radioactivity; good resistance to chemicals; generally good resistance to heat deformation (300-350°F) compared with unreinforced plastics (e.g. PVC); relatively low flammability; seamless construction; and ease of repair and modification. Disadvantages include: poor resistance to alkalis; relatively high cost; and combustibility resulting in toxic, heavy particulate smoke.

Glass-reinforced epoxy laminates are reported to have more resistance to abrasion, alkaline solvents and generally greater heat resistance (330-500°F), but cost more. Limited production of structural material incorporating both polyester and epoxy resins, with the exposed surface of reinforced epoxy, has been reported in Great Britain (74).

At the Argonne National Laboratory, a glass-fiber-reinforced fire resistant polyester was selected as a glovebox structural material for use in an analytical plutonium laboratory (41). The chlorinated resin includes five parts antimony trioxide per hundred parts resin; and is reported by the manufacturer as having a flame spread rating of 20 determined by the Underwriters' Laboratories Tunnel Test (39). The material satisfactorily met certain requirements imposed by the operations, including resistance to solvents and acids, easily decontaminated smooth surfaces, easily cleaned, and easily repaired or modified.

At Brookhaven National Laboratory, a commercially available chemical tank liner of glass-reinforced resin has been used as an enclosure. This is easily cut and drilled to provide windows, glove ports, transfer ports, and utility openings (5). Resin and glass-fiber matting are included for repairs or modifications. The boxes are also supplied with windows molded in place and glove ports already cut out.

Several glass-fiber-reinforced plastics have been tested at one AEC facility for fire and heat resistance: polyesters, polyvinyl, filled cast epoxy, and phenolic resin (24). Glass-fiber-reinforced polyester samples with chlorinated resin molecular structure and/or with chlorinated inhibitor additives supported self sustained combustion. Similar material with antimony trioxide additive would not support flame until after four or five minutes of direct contact with fire. Polyvinyl chloride samples were self extinguishing, but distorted badly. An epoxy resin sample, with inorganic filler (clay, asbestos paper, ferrous wire), was the most fire resistant; very little burning or smoke was observed.

The flammability of the plastic samples was observed to vary, depending on their physical shape. While some flat samples were self extinguishing, the same material, when formed into ducting, supported flame.

In separate large scale fire tests, on a fume hood and exhaust ducts fabricated of 1/8-in.-thick glass-fiber-reinforced chlorinated polyester, with antimony trioxide added, burning resin gave off a dense, sooty smoke. Prefilters were clogged in less than one minute in several tests. Resin was generally burned out of the glass-fiber-reinforcement where fire exposure was most severe.

Compared with other plastics tested, the chlorinated polyester with antimony trioxide was rated as having satisfactory heat resistance and self extinguishing characteristics when removed from an exposing fire. Minor decomposition occurred upon direct exposure to an abutting 600-watt hot plate. A general purpose polyester laminate had lower heat resistance and burned more strongly. Filled epoxy castings were considered excellent in heat resistance and noncombustible, as compared to the better polyesters tested.

Because of the wide variations possible in compounding and manufacturing, fiber-reinforced plastic structural materials should be carefully evaluated for fire resistance and other desired properties before acceptance as a glovebox material. Commonly, changes made to improve one property will affect another desirable property. Special issues of two trade journals, Modern Plastics and Plastics World, listed in the Bibliography, are a good source of manufacturers' names and details on properties of the various resins, additives, etc.

Evaluation of Structural Materials

The following table provides ratings of several structural materials for certain properties of interest in glovebox operations. Ratings apply without coatings, additives or modifications. Ratings are based on FMRC work and information from other sources.

	Stainless Steel	Mild Steel	Aluminum	Plywood	Reinf. Plastics
Corrosion Resis.	Good	Poor	Good	Poor	Exc.
Combustibility	None	None	None	Poor	Fair
Heat Resistance	Good	Good	Fair	Poor	Fair
Abrasion Resis.	Exc.	Good	Fair	Poor	Fair
Decontamination	Exc.	Poor	Good	Fair	Exc.
Strength	Exc.	Exc.	Good	Fair	Good
Workability	Fair	Good	Good	Exc.	Good
Cost	High	Med.	High	Low	Med.

1. Close cooperation with manufacturers and suppliers is recommended to determine the most suitable varieties of aluminum or stainless steel alloys.

2. Generally, stainless steel is recommended for high fire and corrosion resistance, particularly for long term operations. Alternatively, aluminum is recommended as a less expensive choice.

3. Where specific corrosion conditions preclude the use of stainless steel or aluminum, properly formulated glass-fiber-reinforced plastics are recommended. The use of chlorinated resins with antimony additives is generally recommended for improved fire resistance. At greater expense, epoxy resins with inorganic fillers provide even greater fire resistance than modified polyester resins.

4. Smooth, polished interior surfaces are recommended for stainless steel and aluminum gloveboxes to facilitate cleaning and decontamination, and to avoid localized corrosion from trapped moisture or condensates.

PROTECTIVE COATINGS

It is sometimes necessary to coat the interior surface of gloveboxes to provide corrosion protection, a more easily decontaminated surface, or improved retention of transparency. Also, fire retardant mastics and paints are available for wood or metal surfaces (28). Air-dried finishes have been most commonly used. Baked enamel has also been used, mainly on floors. Infrequently, polyethylene and polyvinyl chloride film lining has been used in place of paint. Suitable coatings, carefully applied to a prepared surface can effect considerable economies by allowing the use of inexpensive glovebox materials that otherwise would be unsatisfactory because of undesirable inherent properties.

For facilitating decontamination of the interior glovebox surface and preventing adsorption of contaminants by the surface, removable ("strippable") coatings are often used. Vinyl copolymer lacquer alone, applied over bare metal or wood, has been used (74). For added mechanical strength, a thicker system has been used: two coats of strippable lacquer over two coats of shellac and two coats of titanium-pigmented chlorinated-rubber base paint to cover the strip lacquer. A base of one coat of rubber-base primer and one coat of chlorinated rubber-base paint provides additional protection against mechanical failure and against possible contamination of the wood or metal during stripping (74).

Although conventional air-dried paints have been used, these are susceptible to damage by solvents, heat, mechanical shock, and abrasion. Where strippable or conventional "fixed" coatings are unsatisfactory from the point of view of mechanical strength, impervious coating of high mechanical strength and good decontamination properties are preferable. Titanium-pigmented, chlorinated rubber-base paints have been used in British facilities (74).

Alternatively, cold-catalyzed epoxy resin paints have been successfully used. These have high resistance to alkalis, solvents, non-oxidizing acids, heat, shock, and radiation damage; also, a hard, strongly adhering, non-absorbent surface is provided. Major disadvantages are: fair resistance to oxidizing acids (e.g. nitric acid), difficulty of application, and relatively high cost.

Corrosion protection on mild steel boxes has also been achieved by baked-on phenolic coatings. A commercially available coating of this type,

Bisonite (Bisonite Co., Inc.), has been used in gloveboxes at the Lawrence Radiation Laboratory. This coating, applied in five layers totalling 8 mils in thickness, must be factory applied under controlled conditions.

WINDOWS

The choice of window material is usually governed by the operation proposed for an enclosure. No single material has been found suitable for all glovebox operations. Desirable functional properties of window materials include:

- 1) Satisfactory optical quality; transparency, no loss of transparency over time and minimum of flaws or imperfections.
- 2) Good heat resistance; resist distortion or sagging from exposure to heat.
- 3) Noncombustibility; or at least, non-self-propagation of flame.
- 4) Resistance to explosions.
- 5) Good resistance to chemicals.
- 6) Little or no particulate smoke produced when burning.
- 7) Good resistance to radiation.
- 8) Resistance to impact.
- 9) Resistance to abrasion or scratching.
- 10) Flexibility; can be sealed or gasketed tightly and evenly.
- 11) Easily machined for replacements and modifications on site.
- 12) Moderately priced.
- 13) Easily available in standard sizes and thicknesses.

The primary purpose of a glovebox window is to provide an adequate view of the glovebox interior. Because of safety aspects, fire hazards, costs, strength considerations and other details as applied to presently used window materials, it is usually advisable to restrict window area. Because of inherent weakness of window materials, it is desirable to study viewing requirements before selecting size, number and location of windows.

Laminated Safety Glass

Laminated safety glass remains the preferred window material because of desirable properties inherent to glass. Glass is noncombustible, optically superior to plastics, easy to clean, impermeable to gases and liquids, and resistant to chemical attack and abrasion. Because of the laminated construction, incorporating a plastic (polyvinyl butyrate) layer between and cemented to the two outer glass layers, it does not shatter easily. When

damaged, cracks do not usually extend through the entire laminate. Even when they do, the glass layers generally continue to adhere to the plastic.

Major disadvantages are only fair resistance to impact and low flexibility. In attempting a tight, leak-proof seal between glass, window frame and box, excessive or uneven stress may crack the glass. One user recommends a slow setting rubber potting compound with neoprene window strip. Safety glass is also etched by hydrofluoric acid. A technique has been developed for cutting circular holes in laminated safety glass and lead glass on site so as to avoid breakage or edge cracking⁽⁷⁾, but once the holes are cut, the material reportedly becomes more brittle and fragile.

Tests by the Factory Mutual Research Corporation have shown that laminated safety glass will crack when exposed to high temperatures, and the butyrate layer will burn after exposure of several minutes to an air temperature of 1500°F. As the plastic is consumed, sections of cracked glass will fall away. Use of a 30-mil butyrate layer resulted in no significant difference in fire resistance compared with a 15-mil layer.

It has also been reported that the plastic layer in laminated safety glass tends to lose its transparency over time where much heat is generated⁽⁵³⁾. Furthermore, continued exposure to gamma radiation results in discoloration of the glass to a brownish tinge, unless the glass was manufactured with a cerium additive^(11a)

Tempered Glass

Tempered glass has properties similar to those of laminated safety glass. The most important difference is greater fire and impact resistance. R. F. Malecha⁽⁵⁰⁾ found that a 3-ft. square panel of 1/4-in. tempered glass absorbed 100-ft.-lbs. impact force, while 1/4 in.-laminated glass cracked under 13-ft. lbs. force and 3/8-in. laminated glass cracked under 17 1/2-ft. lbs. force. However, because tempered glass is inherently highly stressed, any cracking from high temperatures, or fracture of the surface by impact or even by scratching can cause an entire panel to disintegrate. Like laminated safety glass, tempered glass will discolor when exposed to gamma radiation over a period of time.

Wired Glass

Wired glass is often used in building construction for windows, including those in door panels, to retard the passage of flame, heat, and smoke. Tests by Factory Mutual Research Corporation have indicated that wired glass is the most fire resistant window material available. However, there may be objection to the restriction on visibility imposed by the wire, and to difficulties, characteristic with glass, in cutting openings. Where visibility requirements are not critical and exposure to hydrofluoric acid is not excessive, wired glass deserves more frequent use. One example is in overhead windows used to transmit light from an external light source, as used at Lawrence Radiation Laboratory. Long term exposure to gamma radiation will result in discoloration of wired glass.

Lucite, Plexiglas

Lucite (DuPont) and Plexiglas G (Rohm and Haas) are two polymerized acrylic plastics that have been widely used for glovebox windows and panels. Their major advantages are good optics (91-92% light transmission), good flexibility for adjusting to slightly curved seating surfaces, excellent resistance to edge cracking, resistance to slight impact and ease of machining. Relative to other window plastics, Lucite and Plexiglas G have relatively good resistance to deformation by heat (maximum service temperature for Plexiglas G is 180° - 200°F (64, 65).

Their disadvantages are that they are flammable, discolor easily, are permeable to hydrogen, helium and moisture, and they rupture easily on heavy impact or explosion. Continuous exposure to high level radiation will cause crazing. Lucite and Plexiglas have only fair resistance to abrasion and variable resistance to chemicals.

Because the standard cast acrylic sheets are flammable, varieties have been introduced with greater fire resistance: Plexiglas SE-3 and 5009B. These two varieties will not support flame, although they deform at lower temperatures (maximum recommended service temperature, SE-3, 160-180°F; 5009B, 140-160°F). Both have improved resistance to abrasion and discoloration. SE-3 has better abrasion and chemical resistance than the 5009B type (65). In tensile, flexural and compressive strength, SE-3, G, and 5009B rank in that order. Frequent cutting, machining and drilling of SE-3 and 5009B should be done with tungsten-carbide tipped tools.

Tests conducted at Rocky Flats indicate that SE-3 is more resistant to HNO₃ and trichloroethylene; absorbs water at half the rate of 5009B; has considerably greater tensile, flexural and compressive strength; and has a maximum recommended continuous service temperature 20° higher than 5009B (63). However, transparency is considerably reduced by exposure to KOH. This is not a problem with 5009B.

Homalite 911 and 911XHT

Two polymerized polyesters, Homalite 911 and 911XHT, have been used in place of Lucite and Plexiglas for improved abrasion resistance, chemical resistance, and flame resistance (6, 8, 53, 74). Both are more difficult to machine, more brittle, and more susceptible to edge cracking. Performance of Homalite in fire tests by Factory Mutual Research Corporation is described later.

Miscellaneous Materials

Sierracin 880-F has been used at Los Alamos for greater heat resistance, and greater shock resistance than Lucite and Plexiglas (53). Rigid polyvinyl chloride (PVC) has also had very limited use. It offers good resistance to abrasion and most chemicals except organic solvents (e.g. ketones, esters). The hard surface can be fairly easy to decontaminate, and the material is easy to fabricate. However, PVC has poor transparency, it deforms at relatively low temperatures (150-175°F) and it will sustain combustion.

PVC in the form of a 20-mil inflatable bag has reportedly been used as a glovebox enclosure (6, 74). Limited space is taken up when not in use, and enclosure can be easily disposed of. Because of the ease of being punctured and its flammability, this enclosure has had limited use for small quantities of low-activity materials only.

Factory Mutual Research

Fire resistance and heat resistance tests have been conducted by Factory Mutual Research Corporation with several types of glass and plastics. Heat resistance tests were performed with a 1500°F radiant heat panel located 2 in. from a vertically mounted 20 in. x 24 in. test sample. Time to ignition, disintegration, or rupture was noted. The fire resistance tests consisted of a 35 in. x 35 in. sample in a frame inclining 18° from the vertical, permitting direct flame impingement from fire in a 1-sq.-ft. pan of alcohol. Failure was considered to be the elapsed time for a hole to appear, or for flame and smoke to penetrate either the material or the frame.

According to the heat resistance test, wired glass and laminated safety glass appear the most desirable from the standpoint of maintaining containment. Plate glass, leaded glass, and semi-tempered glass cracked and fell out of their frames. Fully tempered glass shattered explosively.

Of the plastics, Lexan, Plexiglas SE-3, and Plexiglas G, in that order, were considered the most desirable. While Homalite 911 and Homalite 911XHT resisted ignition considerably longer than Plexiglas G, Homalite 911 and 911XHT shattered like semi-tempered and fully tempered glass, respectively, thus losing containment completely. Lexan, (a General Electric polycarbonate) displayed the greatest resistance to ignition. This material has high impact strength, good heat resistance (to 275°F), contributes much less heat after ignition than Plexiglas G and is self-extinguishing after withdrawal of an ignition source. On the basis of results from these heat resistance tests, only Lexan, Plexiglas G and Plexiglas SE-3, among the plastics, were considered further in fire resistance tests.

In the fire resistance tests, wired glass was far superior to any other material in maintaining containment, followed by laminated safety glass, Lexan displayed the greatest resistance to ignition among the plastics, followed by Plexiglas SE-3 and Plexiglas G. Both Plexiglas varieties continued to burn after withdrawal of the flame source; the Lexan was self-extinguishing.

Both Plexiglas varieties and Lexan softened and eventually sagged badly, continuing to "flow" after withdrawal of the flame source, when CO₂ extinguishing agent was used (on the burning Plexiglas). A water-base agent tended to prevent the "flow" because of the water's cooling effect on the heat-softened plastic.

Evaluation of Window Materials

Ratings in the following table are based on FMRC work and information from other sources.

	Glasses			Plastics			
	<u>Laminated</u>	<u>Tempered</u>	<u>Wired</u>	<u>Plex.G</u>	<u>Plex. SE-3</u>	<u>Homalite</u>	<u>Lexan</u>
Optics	Exc.	Exc.	Fair	Exc.	Exc.	Exc.	Good
Combustibility	Exc.	None	None	Poor	Fair	Fair	Fair
Heat Resis.	Fair	Good	Exc.	Fair	Fair	Fair	Fair
Impact Resis.	Fair	Good	Good	Good	Good	Good	Exc.
Chem. Resis.	Exc. ¹	Exc. ¹	Exc. ¹	Good	Fair	Good	Good
Radiation Resis.	Exc. ²	Exc. ²	Exc. ²	----	----	----	----
Abrasion Resis.	Exc.	Exc.	Exc.	Fair	Fair	Good	Poor
Flexibility	Poor	Fair	Poor	Exc.	Good	Fair	Good
Workability	Poor	Poor	Poor	Exc.	Good	Good	Good
Cost	Med.	Med.	Med.	Low	Med.	High	High

Footnote 1 - except to hydrofluoric acid.

Footnote 2 - if non-browning glass (cerium added).

1. Wired glass is strongly recommended for all windows, where visibility through the wire layer is tolerable. Otherwise, laminated safety glass (from sheet or plate stock) is recommended.

2. Where hydrofluoric acid exposure is serious, where workability of the window material is important, or where the fire hazard is considered not serious, a polycarbonate, such as Lexan, is recommended.

GLOVES

From a fire protection viewpoint, gloves are an inherent weak point in the glovebox containment system. Made of combustible material, they can be damaged or destroyed within one or two minutes by a fire originating inside or outside the glovebox. Inherently weak, they can be ruptured by the pressures of an explosion (Fig. 4-2) and possibly by the pressure resulting from a fire.

The requirements for satisfactory gloves vary with the particular operation. Various properties may be required: physical and mechanical soundness; flexibility; resistance to wear and tearing; resistance to water permeability; resistance to gas diffusion; chemical resistance; and resistance to heat, fire and explosion pressures.

Fig. 4-2. Glovebox following explosion of cleaning solvent. Two gloves were blown off, and the plastic window cracked at the portion taped, upper right.

Glove Material

Neoprene is the most commonly used material. Butyl and polyvinyl chloride (PVC) are less common. Hypalon (a DuPont chlorosulfonated polyethylene) is being increasingly used. Latex has also been used, generally in two-piece construction: latex hands attached to sleeves of neoprene, butyl, or PVC. PVC-coated flannel gloves are occasionally used where air tightness is not essential. Some gloves may be lead-impregnated for protection against gamma rays. Surgeon's latex gloves may be worn as liners to provide a secondary line of defense against leakage or puncture, and to retard moisture penetration by perspiration. Cotton liners have also been worn to absorb perspiration or prevent allergic reactions with the primary gloves. Asbestos or leather gloves, over neoprene inner gloves, have been used for better resistance to heat and chemical attack, but this results in less mobility.

Glove Design

Various hand and sleeve arrangements are available, combining different materials and different thicknesses of the same material. Thickness ranges from 0.008 in. to 0.055 in., with 0.030 in. the most common. Two-piece construction is also used, employing adhesive or mechanical joining of hand to sleeve. One-piece construction may be preferable where radioactive materials are handled because of the hazards of leaks at the joints. Most commonly, individual gloves are designed to be used only on the right hand or on the left hand, although gloves suitable for either hand are commercially available.

Combustibility of Gloves

Involvement of gloves in a fire will likely lead to a burnthrough and possibly contribute to the clogging of high efficiency exhaust filters by the products of glove combustion. With more fire resistant gloves, valuable time can be gained for controlling the fire before the integrity of the containment system is lost.

Recent testing of glove combustibility by the Factory Mutual Research Corporation (Table 4-1), using typical flame and radiant heat exposures, can be summarized as follows:

1. For a given material, the time to ignition and failure increases with glove thickness.

2. Neoprene glove fires tend to be self-extinguishing, if the source of ignition is removed. In close proximity to fire, neoprene will ignite before failure (i.e. appearance of hole caused by softening); under more remote exposure, it will fail by softening before ignition. Butyl, PVC, and latex gloves continue to burn after removal of the ignition source.

3. Radiant heat exposure (e.g. heat lamp) of about $1.2 \text{ cal/cm}^2\text{-sec}$ is unlikely to ignite gloves tested, but decomposition, softening, and failure (appearance of a hole) can occur after continued exposure. Of the materials tested, neoprene is superior to butyl, which in turn is better than PVC.

4. Preliminary tests with silicone rubber gloves indicate considerably greater flame and heat resistance than neoprene. However, weak mechanical properties have been reported based on limited field use.

**TABLE 4-1 FACTORY MUTUAL RESEARCH CORP. TESTS ON
FIRE AND RADIANT HEAT EXPOSURES TO GLOVES**

ELAPSED TIME, FROM INITIAL EXPOSURE TO SMOKING, IGNITION, OR FAILURE, SEC. ¹									
GLOVE		FLAMMABLE LIQUID FIRE EXPOSURE ²				CELLULOSIC FIRE EXPOSURE ⁵		RADIANT HEAT EXPOSURE ⁶	
		POSITION A ³		POSITION B ⁴					
TYPE	THICKNESS, IN.	I	F	I	F	I	F	S	F
N	0.008	14½	16	94	68	7½	19½	-	-
N	0.015	12½	14	93	90	5	8½	12	51
N	0.030	18	24	91	99	12	37	21½	90½
NL	0.030	19	23	94	73	12½	31	27½	70
N	0.055	30	41	97	106	19	42	31½	98½
B	0.015	16	12	64	41	8½	7	8½	43½
B	0.030	26	24	170	50	14	14	13½	72½
PVC	0.015	7½	4½	15	7½	8½	5	9½	15
PVC	0.030	15	11½	15	18	13	13	18½	34½
FRPVC	0.030	12½	9½	86	16	9	21	19	27
PVCF	-	13	5½	49	10½	15	11	6	46
L	0.009	17	9½	104	53	12½	9	10½	30

N = Neoprene
 NL = Neoprene, lead-impregnated
 B = Butyl
 PVC = Polyvinyl Chloride
 FRPVC = Fire-resistant PVC (Prototype)

L = Latex (mechanically jointed
 to 0.015 in. neoprene
 sleeve)
 PVCF = PVC-coated fabric
 I = Ignition
 S = Smoking
 F = Failure

1. Fire times are averages of two trials each. Radiant heat times are averages of 2-4 trials each.
2. 0.24-sq.ft. pan of ethyl alcohol.
3. Pan on glovebox floor, directly under fully extended glove.
4. Pan on glovebox floor, 5 3/4 in. laterally from Position A.
5. 48 paper towels centered directly under glove hand.
6. 1500 ± 10°F as measured at panel face. Center of specimen received 1.2 cal/sq cm/sec.

Rupture of Gloves

Loss experience and FMRC tests show that internal pressure buildup from explosion, fire, and unregulated supply of gas from a high pressure source may cause glove rupture (Fig. 4-2).

Table 4-2 indicates the relative strengths of the various gloves tested by FMRC. It is not intended to show the maximum pressure the gloves can briefly withstand. The following is a summary of the test findings:

1. Glove burst pressure varies with material, thickness, and construction. For one-piece construction, burst pressure is directly related to thickness. PVC gloves resist bursting better than neoprene or butyl, in that order. Limited testing with two-piece gloves, with latex hands and neoprene or natural rubber sleeves, would rank them between neoprene and butyl.
2. Bursting pressures for the gloves range from 0.25 to 2 psig (7-55 in. H₂O). Based on a pressure rise of not more than 0.1 psi/sec, pressures at or above this range will cause failure of gloves unless other box components fail first.
3. The glove hand is inherently stronger than the sleeve. In one-piece and two-piece construction, failure will occur at the sleeve.
4. Gloves have a pressure relieving characteristic because of their elasticity. This effect can help prevent rupture of gloves or damage to other components. The degree of protection provided by stretching has not been determined in any quantitative way.

Chemical and Moisture Resistance

Glovebox work frequently involves materials that are pyrophoric, moisture sensitive, toxic, or radioactive. Since all glove materials are to varying degrees semi-permeable to water, solvents and gases, some care is needed in selecting the best possible material to avoid harm to the operator's hands, excessive deterioration of gloves, or diffusion of undesirable components of the room atmosphere into a special glovebox environment.

Limited testing of various glove materials has been conducted to evaluate resistance to various solvents including water. At one AEC facility, latex, milled neoprene, neoprene-latex, milled Buna-N, milled Butyl, PVA, PVC, and disposable vinyl and polyethylene gloves were examined for resistance against representative solvents: water, acetone (ketone), trichloroethane (chlorinated hydrocarbon), toluene (aromatic hydrocarbon), and two ethers⁽⁵²⁾. Butyl (0.019 in.), followed by PVA (0.029 in.), exhibited the fewest deficiencies. Butyl showed high permeability to trichloroethane and toluene; PVA, to water and acetone. The only significant advantage of neoprene over butyl was that 40% less trichloroethane was lost through it than through a butyl glove of equal thickness. Numerous contradictions were noted between the findings of these tests and claims made by manufacturers.

**TABLE 4-2 FACTORY MUTUAL RESEARCH CORP. TESTS
ON GLOVE BURSTING PRESSURES**

GLOVE MATERIAL	CONSTRUCTION	NOMINAL, THICKNESS, IN.		PRESSURE AT BURST, PSI	FAILURE LOCATION
		HAND	SLEEVE		
Neoprene hand joined to neo. sleeve	adhesive joint	0.008	0.015	0.48	Sleeve
Neoprene	one piece	0.015	0.015	0.56	Sleeve
Neoprene	one piece	0.030	0.030	0.98	Sleeve
Neoprene-leaded	one piece	0.030	0.030	1.09	Sleeve
Neoprene	one piece	0.055	0.055	1.61	Sleeve
Butyl	one piece	0.015	15	0.25	Sleeve
Butyl	one piece	0.030	0.030	0.58	Sleeve
PVC	one piece	0.015	0.015	0.98	Sleeve
PVC	one piece	0.030	0.030	2.09	Sleeve
FRPVC	one piece	0.030	0.030	1.76	Hand/Sleeve
Latex hand joined to neo- prene sleeve	mechanical joint	0.009	0.015	0.28	Hand
Latex hand joined to rubber sleeve	adhesive joint	0.009	0.015	0.34	Hand/Sleeve

NOTE: All values are averages of at least two trials. Most results differed by less than 5% from their average; maximum deviation from average, 12%.

PVC has been reported to have better resistance to nitric acid than neoprene (29). Leaded PVC gloves appear to have better flexibility than equivalent neoprene gloves. However, the plasticizer normally added to the plastic to prevent brittleness tends to be easily extracted during usage.

The diffusion of water vapor through gloves has been considered an important source of in-leakage, and requiring quantitative determination before designing an inert atmosphere and purification system (3). It has been calculated that one 30-mil neoprene (milled) glove will allow 0.224 g of water to enter a glovebox in one day; under the higher temperatures and humidity inside the glove during actual use, this would increase to 2.8 g of water per day.

In studies at Argonne National Laboratory, butyl, milled neoprene, vinyl, and acrylonitrile-butadiene (Hycar) rubber were ranked in that order for resistance to water vapor permeability (4). Increased thickness reduced water permeation almost linearly. Also, the rate of water permeation among several samples of neoprene was found to vary slightly, depending on the manufacturing source.

Hypalon and Hypalon-coated gloves are preferred over neoprene by some users. This synthetic rubber offers good resistance to chemicals, solvents, heat and abrasion; and very high resistance to ozone (64, 74); but has weak mechanical properties. Composite materials have also been investigated with varying degrees of success (74).

Occasionally maximum tactile sensitivity is required. Natural rubber (latex) has been used but it has low chemical resistance to many materials. For work with low-level radioactive materials, the University of Rochester has used thin (0.008 in.) neoprene gloves, with low water porosity, to achieve required sensitivity (75).

Recommended Practices

For the safety of personnel, gloves should provide an effective radiation and toxicity barrier between the hazards in the glovebox and the operator. Within this limitation, they should permit safe and efficient operation within the glovebox. The following recommended practices are intended to help maintain the physical integrity of the gloves, and ultimately, the integrity of the entire containment system.

1. Avoid or limit the use or storage of combustibles near gloves. Maintain maximum practical distances between gloves and combustibles within and outside the glovebox. Within gloveboxes, confine hazardous materials to metal containers. Plastic or glass containers should be placed in secondary metal containers, if possible. Avoid exposure to gloves by heat sources (Bunsen burners, hot plates, heat lamps).

2. Neoprene gloves are generally recommended for satisfactory heat and fire resistance. Where water vapor permeability is critical, butyl gloves are recommended. PVC and neoprene resist bursting from pressurization better than butyl and latex.

3. The use of as thick glove material as possible is recommended to increase resistance to heat, flame, pressurization, chemical attack, permeability, and mechanical failure.

4. Tying two gloves together outside a glovebox, when the gloves are not in use, reduces probability of glovebox fire affecting the gloves. The use of outside metal glove port covers or inside plugs will also lessen fire exposure.

5. Gloves should be firmly attached to glove ports at all times. The use of two expander clamps appears to be the most desirable technique (6). This method has been successfully used at the AEC sites at Oak Ridge, Tennessee and Los Alamos, New Mexico.

6. Design the glovebox interior and glove ports with no sharp edges that can puncture gloves. Handle all equipment having sharp edges with special care.

7. Gloves should be regularly inspected for signs of damage and deterioration, and changed when their condition warrants or is questionable.

8. Depending on the particular operations involved, select the proper type of glove to limit its permeability to water, gases, and chemicals.

UTILITIES AND AUXILIARY EQUIPMENT

A wide variety of auxiliary equipment and services is needed to perform the many operations conducted in gloveboxes. Equipment and components (vacuum pumps, lights, motors, drives, controls) are usually located outside the glovebox.

Careful attention should be paid to sealing glovebox openings through which auxiliary components pass. Corrosion resistant sealing material should be used, and gaskets, seals and clamps inspected regularly for any deterioration or loosening.

Some examples of arrangement and sealing of auxiliary equipment at AEC glovebox facilities are cited below.

At the Los Alamos Scientific Laboratory, the only parts of a rolling mill that extend into the glovebox are the rolls, roll housing, and wobblers (74). Rotating shafts pass through glovebox walls by commercial bearing seals (Garlock closures) or by O-ring grease seals. Closures are press-fitted into the seal housing and mate against the smoothly machined surface of the rotating shaft. Leaking of contaminants has occurred only when alignment or surface finish have been defective. A metal bellows has been used to seal off the entry of a punch press through a glovebox ceiling panel.

At the Argonne National Laboratory Fuel Fabrication Facility, auxiliary equipment such as mechanical drive assemblies and seals and hydraulic systems have been housed below the glovebox in secondary enclosures or cabinets. These enclosures are ventilated with filtered room air drawn through and exhausted through filters to the plant filtration and air cleaning system (66).

A rapidly rotating shaft passing through a glovebox wall is not easily sealed safely and permanently. Various types of seals have been used for such shafts. At Argonne, O-rings, chevron seals, dynamic grease seals, and bellows seals have given good service ⁽⁷⁴⁾. At British facilities, Gaco metal-insert oil seals have been used. These consist of a continuous neoprene knife edge pressed onto the shaft by a circular tension spring ⁽⁷⁴⁾.

Service and utility connections provided in gloveboxes generally include those for hot, cold and distilled water (except in plutonium work); air; vacuum; various gases; electricity; and instrumentation. In some cases, overhead service connections from room headers are installed to provide desirable flexibility; gloveboxes may be disconnected and moved more easily. With overhead connections it is felt that the spread of contaminants in a spill is less likely than with connections made through the floor or lower portions of walls. If there is a criticality problem where plutonium is handled, water is introduced into the glovebox as a batch of limited amount. It is not supplied through lines in the usual manner. Criticality control is exercised by limiting the amounts of plutonium and water in the glovebox.

In the Argonne Fuel Fabrication Facility several utilities are provided as separate system: (a) recirculating water under positive pressure for cooling equipment outside gloveboxes; (b) recirculating water under negative pressure for cooling equipment within gloveboxes; (c) high pressure helium gas, for purging vacuum chambers and plastic transfer pouches; (d) compressed air, for powering pneumatic equipment; (e) control air, for static pressure controllers of building and glovebox ventilation systems; (f) helium gas, for glovebox atmospheres; and (g) argon gas, principally for inert-arc welding, (45, 66, 66b, 74).

These utilities at Argonne are distributed from their source to fabrication areas through a utility tunnel below the working area and up through slots in the floor located at every glovebox line. Shutoff valves are provided at each slot. Piping runs directly to equipment, but also terminates at convenience outlets (valve and quick disconnect coupling) located outside and within gloveboxes. Type "K" copper tubing is used, with silver-soldered joints. Few threaded connections were installed. Where used, joints were either sealed with an "oxygen sealing compound" conforming to MIL-C-5542 or fitted with a Teflon-seated sealing nut.

Utility systems may incorporate numerous electrical interlocks, by-pass valves, solenoid valves, float switches, pneumatic temperature controllers, thermostats, pressure switches, electrical liquid level controls, shutoff valves, pressure reducing valves, pressure relief valves, check valves, and low pressure alarms. A diesel generator can furnish emergency power to critical equipment if normal power fails.

At Los Alamos, utilities have been piped into gloveboxes through 1/2 in. stainless steel pipe ells welded into openings ⁽⁷⁴⁾. For liquids, hose cocks and hose connectors are screwed into ells inside the box. Connections to equipment are made through neoprene or plastic tubing. Because the services are under pressure, little or no contamination is reported to back up into the lines.

In some gloveboxes at Los Alamos, water is drawn through the system from sump pumps under negative pressure, so that the glovebox cannot fill with water if a line ruptures within the enclosure.

Electrical lines for some Los Alamos gloveboxes are run through the welded ells to outlet boxes mounted over the opening. Flexible putty is packed into the ell and the terminal end of the conduit to prevent spread of contamination. At other facilities, outlets have been sealed in resin.

At Mound Laboratory, utilities are piped in through stainless steel couplings welded into the walls and top of the glovebox. Stainless steel tubing is piped continuously through couplings and locked into place with a male adaptor which has pipe thread on one end (which screws into a coupling) and a tubing compression fitting at the other end to swage into tubing. These adaptors are bored to remove shoulders.

Glass-fiber-reinforced gloveboxes at Argonne utilize interior terminals for AC, DC, variac-controlled and regulated power supply voltages (42, 43).

Additional bulkhead fittings may be installed for introducing gas, water and other services over and above the standard equipment. Fittings being installed in contaminated gloveboxes are always introduced from the box exterior. A "Roto-Bor" boring tool is used to cut a smooth clean hole through the plastic, carrying cutting debris to the inside bore of the tool; if high contamination exists, a vacuum bag can be attached to the tool to collect debris.

At some gloveboxes of the Argonne Fuel Fabrication Facility, services are brought into the enclosures through O-ring gasketed aluminum plates set in the top panel of the glovebox (74).

At gloveboxes in the Argonne Fuels Technology Center Plutonium Laboratory, piped services from an overhead manifold above the ceiling are connected to the glovebox by O-ring unions; services are valved both at the glovebox and at the ceiling. To relocate a box, electrical lines are unplugged, two valves on each piped service are closed, and the O-ring unions at the glovebox are disconnected (74).

Gas inlets at plutonium gloveboxes include sintered metal filters, in-line filters, and check valves to avoid backup in the service lines. Electrical cable fed through for power leads is extensively used at one facility.

At British glovebox facilities, piped services are normally connected to overhead mains with flexible hose (74). Deterioration of rubber hose and diffusion of air into it have led to the use of neoprene-lined hose. Electrical power is supplied from a continuous header, above ceiling level, through flexible cable to an amplifier-power supply rack. The cable is vulcanized insulated rubber, taped and varnished cambric fabric, cotton braided, and flexible armored galvanized steel. Glass insulated cable connects the power supply console with the glovebox by multi-pin plugs and sockets at a terminal box mounted externally on the glovebox. From

this terminal box, cables extend to external apparatus, and into the enclosure to an interior terminal box. The point of entry at the glovebox wall is sealed by double rubber gaskets, a rubber bung, and a clamping nut. All switches and terminal boxes are specifically numbered to facilitate servicing.

Recommended Practices

The following recommendations are intended to help maintain the physical integrity of the containment system:

1. Wherever practicable, auxiliary equipment should be located outside the glovebox to avoid contamination, to conserve working space within the enclosure, and to facilitate maintenance and repair.

2. Electrical connections and electrical equipment should be in accordance with the National Electrical Code. If electrical equipment must be located within gloveboxes where combustible concentrations of flammable vapors, gases or dusts can occur, the equipment should be of a type approved for the specific hazard.

3. Overhead service and utility lines may be used to minimize spread of contaminants in case of a spill, and to provide flexibility for glovebox operations.

4. Openings in glovebox walls through which wiring, piping, tubing and equipment pass, should be tightly sealed. Seals should be checked regularly for fit, deterioration, and leakage.

5. Service and utility equipment (hose, switches, terminal boxes) should be resistant to anticipated corrosive conditions, both within and outside of the glovebox.

6. Adequate emergency shutoffs and other safeguards should be provided as outlined in the section on Industrial Safety.

7. Check valves and in-line filters in service lines may be desirable to prevent back-flow and possible entry of contaminants.

8. Auxiliary equipment and service lines and connections within gloveboxes should be noncombustible insofar as possible.

9. Label, color-code, or otherwise identify and differentiate components and equipment (piping, hose, cables, controls, gauges, etc.) to facilitate servicing as well as normal operation.

10. Illuminating and acetylene gas should not be permanently piped into a box.

11. High pressure gas lines should be limited in size to avoid pressurizing the box in case of a break.

12. Precautions should be taken when coolants such as liquid nitrogen are used, since they can rapidly generate gas when spilled and overpressurize the box.

5. VENTILATION

Proper design and control of ventilation is essential to the safety of glovebox operations. Properly designed ventilation systems will assist in preventing spread of contamination from the glovebox to the working area, from the working area to adjacent areas of lesser contamination risk, and from the building to the environment.

Room ventilation should be arranged to establish an airflow pattern from zones of lower to progressively higher contamination risk. Pressure is progressively lower, from "clean" (noncontaminated) areas to "hot" areas. Incoming air is cleaned, and sometimes dried also, as it enters the building. In cases where ventilation is for the building only, it is exhausted from potentially contaminated areas through high efficiency filters to the outside areas. In some cases building air is used to supply the glovebox ventilation system (Fig. 5-1).

Glovebox ventilation systems are of four types: once-through air; once-through inert atmosphere; recirculating air, especially dried air; and recirculating inert atmosphere.

In once-through air systems, an absolute intake filter at the enclosure provides additional cleaning and helps to prevent back-flow of contamination from the box. Airflow through the enclosure is usually adjusted at 1/2 to 3 box volumes per minute. Air is usually exhausted through an exhaust pre-filter of glass fiber, attached to the glovebox or in ductwork near the enclosure. Fire resistant or noncombustible high efficiency (HEPA) filters provide additional filtration of exhaust gases.

These filters are usually located near the glovebox or in ducts within the work area. Additional high efficiency filters may be located further downstream, including the fan loft or room, if highly hazardous materials are being processed.

In many glovebox installations individual exhaust systems are provided with their own ductwork and exhaust fans. Several groups of gloveboxes may be exhausted by a single common main blower assembly, or less commonly, by several individual exhaust fans. At a few installations, large filter banks in separate plenums or rooms provide final filtration of enclosure and room exhausts.

Duplicate fans may be provided for regularly scheduled alternating operation, or for substitute operation in case of malfunction of the on-stream fan. Either automatic or manual fan operation is used. Emergency electric generators are desirable to supply regular or standby blowers. A standby or purge exhaust system is sometimes provided, for actuation in case of sudden rise in box pressure caused by rupture of a glove or window.

INERT ATMOSPHERE SYSTEMS

Continuous inert atmospheres are provided in gloveboxes to prevent contamination of materials being handled and/or to prevent air-dependent fires and explosions.

Fig. 5-1. Example of a combined building and glovebox ventilation system. (Composite illustration. At specific installations, number and location of filters will vary.)

Plutonium, for example, is readily contaminated in a normal air atmosphere. It is also pyrophoric, particularly in finely divided form, and can ignite spontaneously at room temperatures in air containing moisture. Metallic plutonium spontaneous ignition hazards in air are very significantly reduced by keeping moisture levels below 500 to 1000 parts per million. If a supplemental heat source is present, combustion can be sustained in air containing as little as 1% oxygen. For these reasons, inert atmospheres are provided in many gloveboxes handling plutonium. Other pyrophoric metals handled in gloveboxes under inert atmospheres are uranium, thorium, zirconium and alloys of these metals.

Refractory materials containing plutonium in the form of carbides, nitrides, oxides and silicides are also worked in gloveboxes employing an inert atmosphere, as are highly reactive materials such as the alkali metals including NaK.

Chemical operations within gloveboxes often involve the use of flammable liquids. In such gloveboxes, formation of explosive mixtures with air can be eliminated by maintaining an inert atmosphere.

Glovebox gas atmosphere systems commonly utilize argon, helium, or nitrogen. Argon and helium are true inert gases and are usually recirculated, at relatively high flow rates, and also purified. Nitrogen is normally used in once-through systems at low flow rates (Fig. 5-2).

Technical reasons may require use of a gas system of much higher purity than needed for fire safety. In some cases, as when working with ceramic fuel mixtures, impurities must be held to a few parts per million.

Safeguards

When an inert atmosphere system is used:

1. Locate the gas supply (cylinders or tank) in an area separate from the gloveboxes, to reduce the possibility of damage to the gloveboxes from an accident involving the cylinders or tank, and to reduce clutter in the vicinity of the gloveboxes.
2. Provide for prompt indication of over or under-pressurization by suitable pressure switches and alarms. These switches are often used to actuate emergency pressure control systems to correct the abnormal pressure condition.
3. Provide continuous monitoring of the inert atmosphere, as needed, by means of suitable instrumentation to detect impurities, leaks, contaminants (constituents of enclosure atmosphere that may have harmful effects on metal surfaces or other materials), radioactive gases, and abnormal heat (Fig. 5-3).
4. Develop and carry out a preventive maintenance program for all system components, including continuous monitoring of the inert atmosphere to promptly detect leaks.

Fig. 5-2. Schematic diagram of once-through nitrogen system for plutonium gloveboxes.
(Argonne National Laboratory)

Fig. 5-3. Schematic diagram of oxygen analyzer sampling lines for main nitrogen exhaust lines and individual gloveboxes (GB). (Argonne National Laboratory)

5. Provide duplicate arrangement of components critical to continuous operation, such as paired exhaust fans to assure continued exhaust ventilation in case of failure of one of the fans.

6. Consider consequences of power failure. An emergency power source may be needed in some cases, such as to assure continued exhaust ventilation.

EXAMPLE OF VENTILATION SYSTEMS AT AN AEC FACILITY

At the Fuel Fabrication Facility of Argonne National Laboratory, complex ventilation systems are in operation because of numerous contamination risk areas with varying degrees of hazard. Separate building ventilation is provided for the Administrative Area, the Technical Area, and the Fabrication Area (66, 74).

In the Administrative Area, which has the lowest contamination risk, incoming air is drawn by a supply fan through a conditioning system where it is heated or cooled, as required. Air enters the area at 7400 CFM; and is exhausted through wall louvers at 7500 CFM by an exhaust fan and through dampers to a stack. The static pressure in the area is maintained at approximately atmospheric level. All ductwork is galvanized sheet metal.

In the Technical Area, incoming air is filtered through two banks of 8 fire retardant filters. Filter packs are mounted in metal frames. The air is heated or cooled, as required, and introduced to the work area at 6700 CFM. Air is exhausted at 6800 CFM, through ceiling openings, into filtered ducts to the fan room, and then through high efficiency final filters to the exhaust stack.

Static pressure in the Technical Area is maintained at approximately -0.05 in. H₂O. Ducts are galvanized sheet metal (supply air) or welded stainless steel (exhaust air). Exhaust filters are removed, when necessary, by a slip cover method; or, for those servicing the special materials storage vaults, by plastic bag technique.

In the Fabrication Area where glovebox operations are located, intake and exhaust air are more extensively filtered. Incoming room air is drawn by a fan through filters. After heating or cooling as required, the air enters the work area through ceiling diffusers at 24,500 CFM, and circulates through the work area and those gloveboxes where work with clad materials and aqueous solutions is done. Glovebox air is exhausted through glass-fiber exhaust filters. Room air is exhausted separately, through the secondary enclosures described below. Room and glovebox exhaust air enters the building exhaust system, passing through a fan room where it is filtered by two banks of high efficiency filters and drawn by two fans to the stack. A recirculating helium atmosphere is used for the main glovebox line where work with unclad plutonium and plutonium alloys is done. Details of the system are given in the R. M. Mayfield and W. G. Tope article listed in the Bibliography.

Auxiliary equipment that may be contaminated through the failure of gaskets or joints is housed in secondary enclosures. A once-through filtered air system ventilates these enclosures. Air is drawn from the room through inlet filters and exhausted through filters to the building exhaust system.

Static pressure in the Fabrication Area is maintained at -0.125 in. H_2O . Supply ducts are of galvanized sheet metal; room exhaust ducts are of welded stainless steel. Glovebox ductwork is either of welded aluminum or stainless steel.

Two automatic standby or purge systems of 3000 CFM capacity are available to handle any loss of integrity in most of the gloveboxes. Activation of purge air supply at inert-gas gloveboxes results in automatic shutdown of the helium supply. Loss of a 10 sq. ft. window area will result in a minimum air velocity through the opening of 300 ft./min; velocity may be increased up to 900 ft/min. Purge air, when used, would be exhausted through the same high efficiency filters in the fan room as the normal air atmosphere.

FILTERS

Filters used in glovebox and building ventilation systems must have high filtering efficiency as required to prevent the passage of radioactive particles, low pressure drop, fire resistance, and long useful life. Common practice is to provide two exhaust filters at the glovebox: a primary or roughing filter to keep most of the active material from entering the exhaust ducts, and a high efficiency filter to remove any remaining airborne activity. Glovebox inlet filters are intended to prevent escape of contaminants upstream or into the room and to increase the useful life of exhaust filters by removing dust.

The minimal specification recommended for purchase of high efficiency particulate air (HEPA) filters is Military Specification MIL-F-51068A, 23 April 1964, Filter, Particulate, High-Efficiency, Fire Resistant. It is also recommended that the Filters be labelled by Underwriters' Laboratories as meeting the UL Standard High Efficiency Air Filter Units, UL-586, second edition June 1964 (72). Manufacturers of filters conforming to this standard are listed in Underwriters' Laboratories' Building Materials List (71). Information on filter inspection and testing by the Atomic Energy Commission is outlined in AEC Health and Safety Information Bulletin, No. 253, July 10, 1967.

In general, various filter materials are available to meet required degrees of fire resistance, efficiency, strength, or to reduce the cost of fabrication (6, 10, 30, 31, 44). High-efficiency filters can be purchased to meet required moisture and humidity resistance, exposure to heat, and resistance to solvents, alkalis and acids.

Added fire protection may be obtained by wire mesh "prefilters" installed near the face of exhaust filters. These retard buildup of condensed oil on the filters, and act as spark arresters (13, 46). At British facilities, stainless BS40 mesh and, less often, loosely packed glass fibers are used as a spark guard (66c).

VENTILATION DUCTS

Ventilation ducts have been made of various materials, although metal construction is most common at newer facilities. The choice of metal (steel, stainless steel, aluminum) depends on the extent and nature of corrosive fumes exhausted, cost, and decontamination ease. Where corrosive or explosive

fumes and vapors are exhausted, auxiliary equipment such as scrubbers may be installed to remove these substances from the air stream. Metal ductwork has also been coated with phenolic coatings for more economical protection against corrosion or contamination (8).

Various plastics are also available as duct structural material, such as rigid polyvinyl chloride (PVC), polypropylene, and glass-fiber-reinforced plastics. These are usually selected where corrosion problems exist, for lower installation costs, and generally where there is a minimum of fire hazards in the glovebox operations. Poorer resistance to high temperatures and combustibility as compared to metal ducts are the two main objections to these materials. Low combustibility fibrous glass ducts are available (15).

Fire tests of various plastics for ducts and enclosures have been conducted by the Fire Protection Department of Argonne National Laboratory (24). Varying degrees of heat distortion and flammability were noted among glass-fiber-reinforced plastics and unreinforced plastics. The most serious general problem was the heavy particulate smoke emitted by these burning plastics that rapidly clogged exhaust filters.

In small scale tests, glass-fiber-reinforced polyester resin duct units, made of resin with chlorinated additives or having chlorinated molecular structure, supported combustion. With antimony trioxide added, longer continued flame contact was needed to produce self-sustained combustion. Polyvinyl chloride units were self-extinguishing, but flaming was observed at 660°F air temperature without direct contact with the exposure fire. The PVC units also distorted badly. A laminate of phenolic asbestos and phenolic nylon blistered badly so that air flow was greatly reduced through the duct. Epoxy resin with an inorganic filler was the only plastic that did not produce dense, sooty smoke. Little burning and insignificant distortion were observed.

Larger scale tests confirmed that polyester materials, despite fire resistant additives, will emit dense particulate smoke during combustion that will clog even low-efficiency filters rapidly. Noncombustible construction exposed to a relatively smokeless acetone fire will not lead to filter plugging. If a fire takes place in a "standard fuel loading" (a mixture of cellulosic and flammable liquid materials) enough smoke and soot is emitted to clog filters.

At the Y-12 facility of Oak Ridge National Laboratory most ductwork is reportedly of metal (56). Where decontamination is a problem, welded stainless steel is used with angle flanges and neoprene or rubber gaskets. Where cleaning by water is performed, round ductwork is preferred.

Long runs of ducting without seams or joints are desirable for maximum containment. Where joints are necessary, welded joints are preferable to bolted or riveted seams. At disassembly points or disconnects, gasketed and bolted flanged joints are recommended (26).

Smooth straight-through air flow is desirable; sharp bends or elbows encourage accumulation of deposits. Ducts should be accessible for cleaning. Location of access panels where negative pressure is greatest, downstream of filters, is desirable to minimize contamination hazards (26).

Round ductwork is preferred to rectangular shapes. This has several advantages: (1) economical use of metal, by weight; (2) greater strength as a section; (3) maintenance of air velocities at a more uniform level without internal vanes or other devices; (4) internal drainage is more easily handled; and (5) supports can be simpler (26).

Location of exhaust ductwork at a lower level than gloveboxes may be desirable (8). Provisions for drainage should be included, especially where sprinklers are installed or condensation is likely (10). At one facility, where sprinklers are provided in exhaust ducts, the exhaust fans, filters, and ducts are located below operating areas, so that sprinkler systems in the ducts cannot flood gloveboxes. At another facility, a main exhaust tunnel of concrete was poured along with the building foundation.

Where ducts pass through ceilings and walls, the opening should be well-sealed to prevent passage of smoke, fire, and water.

RECOMMENDED CRITERIA FOR SAFE DESIGN AND OPERATION OF VENTILATION SYSTEMS

1. A pressure gradient should be maintained between areas of low contamination risk and areas of high risk. Approximately atmospheric pressure may be maintained in minimum risk areas, such as offices; pressures should be progressively more negative as the contamination hazards increase.
2. Once-through passage of air is desirable in building areas. A recirculated atmosphere in gloveboxes is usually (but not always) desirable where inert gases or dry atmospheres are used.
3. For fire protection purposes, sectionalizing of glovebox lines and their ventilation systems is desirable.
4. Noncombustible corrosion-resistant duct materials, such as stainless steel or aluminum, are desirable for reduced fire hazard. Flexible woven asbestos or material of comparable fire resistance is recommended for flexible duct connections.
5. Metal ducting should be of a gage appropriate to the size of the ducts, for strength and durability. All ducting should be adequately supported.
6. Fire detection systems should be installed in ducts where a fire hazard exists (see Chapter 6).
7. An emergency purge ventilation system is desirable at high-hazard glovebox installations. The emergency system should be capable of maintaining a negative pressure within the glovebox if an accidental breach of integrity occurs such as rupture of a glove or window.
8. Where a high degree of fire risk exists, automatic or manual means of slowing the ventilation system is desirable (31). Sufficient air movement to remove explosive or combustible gases and to provide a cooling effect should be maintained. This should be planned so that a regular procedure can be followed in a fire. In parallel filter installations, segment and isolate with dampers so that fire may be fought in one filter while some ventilation can be continued (10).

9. Exhaust to atmosphere through tall stacks is generally desirable to achieve maximum possible air dilution and minimum settling of particulate matter at ground level downwind.

10. Monitoring of stacks is recommended if not already mandatory, to detect any abnormal radioactivity within the exhaust system.

11. Standby power for the ventilation system and related auxiliary equipment should be available. Duplicate exhaust blowers are also desirable.

12. Fans, blowers and related equipment should be located downstream of filter assemblies to avoid contamination and to facilitate servicing.

13. Noncombustible AEC approved high-efficiency filters should be used. (Note: filters approved on the basis of Underwriters' Laboratories standards, UL-586, will meet this requirement. Vendors are listed in UL's Building Materials List.)

14. Where serious fire and/or contamination hazards exist, automatic spray nozzles or sprinklers are recommended ⁽¹⁰⁾. Water spray ("fog") nozzles provide efficient penetration of filter media without damage. Two lines of spray nozzles are desirable to discharge water on both faces of filters.

15. All filters should have noncombustible or fire-retardant framing, separators, gaskets and sealing compounds. Filters lacking separators are not recommended because of low shock resistance and reduced fire resistance ⁽¹⁰⁾.

16. High-efficiency filters should be carefully inspected upon receipt from suppliers and again before use.

17. Prefilters should be located as close to the operation being ventilated as possible to reduce accumulation of lint and gross radioactive particulate matter in the ducts. However, from a fire-safety point of view, a location exposed by a fire is undesirable.

18. To help retard fire spread, leave as much space as practicable between filter banks, between adjacent filters, and between prefilters and HEPA filters in all installations.

19. Vertical mounting of filters in horizontal ducting is desirable so that the filter pack is properly supported. Where filter units are installed for updraft or downdraft use, a wire mesh screen or metal shield would prevent sagging of the filter pack. Pull-up bolting that compresses the gasket to the sealing face of the frame is reported as a desirable mounting technique ⁽¹⁰⁾.

20. Exhaust filters at the glovebox are more easily changed when installed within the glovebox. Changing of contaminated filters external to the glovebox should be carefully performed, preferably by a sealing-out procedure commonly followed at transfer ports.

21. In-place filters should be periodically inspected for efficiency (DOP) as outlined in Reference 10. Details on tests are given in the proposed USASI standard "Efficiency Testing of Air Cleaning Systems Containing Devices For Removal of Particulates".

6. FIRE DETECTION SYSTEMS

Fire detection systems approved by a nationally recognized testing organization, have wide application at glovebox facilities, within the boxes, in ventilating ducts where a fire hazard exists, and in the room or building. The glovebox is an inherently weak structure. It can withstand only slight fire exposure, and only for a short time. Therefore, the time element in discovery and extinguishment of fire assumes unusual importance. Automatic fire detection systems are used in glovebox work to give prompt warning of fire in its early stages and also to promptly actuate fire extinguishing equipment. The systems consist of fire detecting devices connected through electrical circuits and control panels (Fig. 6-1) to alarms, recorders or equipment-actuating switches (14, 15, 55). Manual alarm systems, independent of detection systems, are also widely used. They enable a person discovering a fire or other incident to call help promptly by operating a manually operated fire alarm box. Automatic detection systems should be equipped for manual as well as automatic operation.

TYPES OF FIRE DETECTORS - GENERAL INFORMATION

Heat-actuated detectors are the type most commonly used at glovebox facilities. They are of two principal types: (1) the fixed temperature type, designed to operate when the temperature reaches a pre-determined value; and (2) the rate-of-rise type, designed to operate when the rapidity of temperature rise exceeds a pre-determined rate (usually 15 to 20°F/min) regardless of the temperature level. Some detectors combine these two characteristics so that actuation occurs at a fixed temperature level in case the temperature rise is too slow to actuate the detector by the rate-of-rise principle.

Heat-actuated devices are further subdivided into (1) the continuous or line type, such as pneumatic tubing or heat-sensitive cable, and (2) the spot type, several of which are installed at specified distances apart. Some devices are capable of repeated operation and others must be replaced after each operation.

Special approved heat detectors are available for use where an explosion hazard exists: atmospheres containing flammable vapors, flammable gases, or combustible dusts (15, 73).

Some heat detectors function when an element fuses or melts; these are typically "one-shot" fixed-temperature devices, requiring replacement after actuation. Other detectors function by the closing or opening of electrical contacts due to thermal expansion of bimetallic elements; this type of actuation is used in both fixed-temperature and rate-of-rise detectors. In another fixed-temperature spot detector, the contents of a bulb "thermostat" expand when heated until the bulb bursts, resulting in the closing of an electric circuit.

One common line detector consists of a pneumatic tube operating on the rate-of-rise principle. Pressure generated by the heating of an enclosed

Fig. 6-1. A main fire control "locator box" for a heat detection system in a glovebox line at a plutonium production building. When a detector is actuated, the location of the glovebox is indicated on the panel, an alarm bell is sounded in the main fire house, and a horn is set off at the locator box. (Dow Chemical Co., Rocky Flats Div.)

gas closes or opens electrical contacts. "Equivalent" air chambers or compartment units may be attached to the tubing to provide more convenient or sensitive detection. For example, where space is restricted, it may be more convenient to install a compartment providing the equivalent detecting capability of 25 ft. of tubing, instead of attempting to loop 25 ft. of actual tubing. In another line detector, operating as a fixed-temperature device, two conductors are electrically insulated from each other by thermoplastic material. When exposed to fire, the plastic melts and the two conductors contact each other thereby closing a circuit.

Still another line detector consists of tubing containing an eutectic salt mixture. The salt mixture, which separates two electrical conductors, becomes electrically conductive at elevated temperatures and closes the circuit. When the temperature drops the salt becomes non-conducting; thus, the detector is self restoring. Eutectic-salt-tubing detectors can be used where ambient temperatures are high. They are used at several glovebox facilities.

Smoke-actuated detectors operate on a photoelectric principle or an ionization principle.

In the photoelectric type, there is a change in photoelectric current that accompanies a change in light intensity on a photoelectric cell or combination of cells. The current change results in a relay operation that produces an alarm signal. In a beam system, the light source and photoelectric cell are located at a distance from each other, in the protected area. In an air-sampling system, a continuous sample of air from the protected area is drawn by a blower in the device into the device's smoke chamber containing both the light source and photoelectric cells.

The ionization type of system consists of individual smoke-detection units that are normally mounted at the ceiling and electrically connected to control and indicating panels. Each detector contains radioactive material that ionizes the air in special chambers within the detector. Smoke particles entering the exposed chamber upset the normal ionization and result in an alarm condition.

Other detectors of the ionization type do not incorporate a radioactive source.

Smoke detectors are particularly suited for fires generating smoke without or before substantial heat emission. To avoid false alarms, these detectors should be used only where smoke is not normally present, or where it is effectively exhausted.

Ionization-type smoke detectors have been reported to be unsatisfactory in glovebox atmospheres whose constituents are changed (59). For example, at one facility, introduction of helium into a normal air atmosphere was found to trigger false alarms because of change in the ionization potential of the atmosphere. Even when the detector was adjusted for a specific atmospheric mixture, any change in proportions of constituents would trigger the detector.

Flame-actuated detectors are less commonly used than heat or smoke detectors. One type reacts to the characteristic pulsing in radiant energy emitted by flames; another type reacts to the radiant infra-red energy of the flames (15, 55). Both are extremely sensitive and fast-acting compared to heat and smoke detectors. Both are receptive to radiant emission from other sources besides flames, and the installation must be carefully planned to avoid false alarms. Normally, the manufacturer will custom-design each installation.

To avoid actuation in the presence of normal sources of heat, a flame detector with specificity has been suggested (42). Specificity may be possible by mounting the detector in a filter open only to frequencies of light known to originate from fire. A detector which responds to the ultra-violet radiation in ordinary flames offers promise for glovebox fire detection. It is insensitive to sunlight, cosmic radiation, infra-red, and ordinary incandescent and fluorescent light sources.

ILLUSTRATIVE DETECTOR INSTALLATIONS IN GLOVEBOXES

Detectors commonly used at AEC contractor glovebox facilities include pneumatic tubing (rate-of-rise), spot heat detectors (rate-of-rise and/or fixed temperature), and eutectic salt tubing (fixed temperature).

In glass-fiber-reinforced gloveboxes at Argonne National Laboratory, 160°F rate-compensated heat detectors have been installed (40). In each enclosure, a single detector is threaded into a pipe reducer which is plastic welded to the center of the box ceiling, about 3 in. from the rear wall. The detector is wired to a squib discharge plug in the discharge head of an externally mounted CO₂ extinguisher, and to a signal board. At 500°F ambient, the detector will operate in about 6 sec.

Because of corrosive conditions, a chemically resistant coating was needed. A two-coat system was selected: a 5-6 mil prime coat of Sarancoat for adhesion and elasticity and a finish coat of modified polyester resin. The slight reduction in sensitivity was not considered objectionable.

At another facility, various requirements were established by which to evaluate detection systems for protecting a specific glovebox line (59):

1. Good sensitivity to combustion of metals and organic material.
2. Good sensitivity to rapid temperature rise, or to temperature rise without combustion.
3. Effectiveness in air-helium atmospheres.
4. Suitability for varying ambient temperatures, including those at high temperature process lines.
5. Adaptability to glovebox design.

6. Components requiring maintenance or adjustment to be outside of enclosure..

7. Installation to be as a zoned system to provide indication of location of fire, and to provide audible warning and visual indication at various monitored locations.

Rate-of-rise pneumatic tubing was selected. To increase sensitivity of the pneumatic tubing, 3/16 in. - I.D. copper tubing was used instead of the normal 0.05 in. size. Sensitivity could also be changed by other adjustments.

Tubing was installed near the ceiling of each glovebox throughout the entire length, passing through the end plate of the glovebox line and extending downward to form a loop in a secondary enclosure housing auxiliary equipment. A section of tubing was also fastened in contact with the underside of the floor of each glovebox module. Instrumentation for calibration and maintenance was located outside of and above the glovebox line.

FMRC INVESTIGATIONS OF DETECTOR PERFORMANCE

In an investigation of fire detectors in gloveboxes, the Factory Mutual Research Corporation concluded that several factors have an important bearing on performance. In addition to the type of detector, other important variables include number of sensing units, location, glovebox size, glovebox ventilation rate, type of fire, location of fire, and size of fire. FMRC evaluated a representative selection of commercially available detectors to determine the nature and importance of these factors (Table 6-1). In the tests, denatured ethyl alcohol was used to represent flammable liquid fires; paper towels, to represent cellulosic fires.

The major findings of FMRC research and testing follow:

1. Response time of heat detectors is dependent on the size of flammable liquid fires. It is not appreciably affected by the size of a cellulosic fuel fire, except where the amount of fuel is too small to actuate the detector at all.

2. Single point location heat detectors are adequate for smaller gloveboxes i.e. dimensions less than 4 ft. x 4 ft. x 4 ft. In the larger sizes within this "small" category (such as the FMRC unit which measured 3.5 ft. x 2.1 ft. x 3.3 ft.), location of the detector is important. Box ceiling center is preferable. The next best location is between the ceiling center and the side from which the exhaust is taken.

For gloveboxes larger than 4 ft. x 4 ft. x 4 ft., several detectors are needed for fast response to small fires. Continuous type detectors may serve equally well, and are preferable in longer boxes or in a long train of enclosures because of less cost and complexity.

3. The temperature rating of fixed temperature heat detectors has a noticeable effect on response time. To minimize the possibility of smaller fires going undetected, a device with the lowest practical temperature rating should be used.

**TABLE 6-1 RESPONSE OF SELECTED DETECTORS IN 67 CU-FT
F. M. R. C. TEST GLOVEBOX**

VENTILATION RATE, CFM	FLAMMABLE LIQUID FIRE SIZE, SQ. FT	DETECTOR RESPONSE TIME, SEC.							
		FENWAL	ROR/FT	ADT FT			KIDDE "ATMO" PNEUMATIC TUBE, ROR		FENWAL
		190°	140°	125°	125°	125°	25-FT	40-FT	SALT
		#	#	#	#	#	EQUIV.	EQUIV.	TUBE, FT 255°
25	0.06	DNO*	DNO*	123*	393*	DNO*	DNO*	DNO*	DNO*
	0.25	41	63	25	43	82	12	21	72
	0.56	16½	24	11	18	30	6	12	29
	1.00	12	17	10	13	22	5	7	21
	2.25	7½	9*	5½	6½	10½	3*	6*	12½
	4.0	5	5	4	4½	6	3	5	9
	9.0	3*		2*	3*	4*	1*	3*	6*
88	0.25	60	96	35	43	100	18½	24	106
	0.56	22	28	18½	22	37	9½	13½	36
	1.00	14½	18½	12½	15½	22	5½	8½	22
	2.25	9½	11	10*	11½	14	4	6	13½
	4.0	6	6*		7	8	3	3½	10½
	9.0	5½		5	2½	6	3½	4	9
	CELLULOSIC FIRE SIZE, NO. TOWELS								
25	3	DNO/56	DNO	50	DNO	DNO	23	31	DNO
	6	32	DNO	34	39	DNO	18½	22	36
	12	26	37	30	29	43	19	23	32
	24	26	32	25	27	36	17½	19½	28
	48	28	31	26	26	34	21	23	29
88	3	DNO	DNO	DNO	DNO*	DNO	40	51	DNO
	6	DNO/36	DNO	35	44*	DNO	20	26	DNO
	12	36	DNO/43	30	35*	59	24	27	40
	24	30	38	27	27*	41	22	24	33
	48	28	36	25	30	40	18	20	32

- Distance from fire differed for each detector.
 * - One trial only
 DNO - Did not operate
 DNO/56 - Two results not averageable.
 ROR - Rate of Rise
 FT - Fixed Temperature

4. Increasing the ventilation rate increases fire detection response time. In small boxes, this effect is important when the ventilation rate is at least 2-3 box volumes per minute; in larger enclosures, when the ventilation rate is at least 1 box volume per minute.

5. Smoke detectors sense small fires in cellulosic materials more quickly than heat detectors. However, smoke detectors cannot react to non-smoky fires, such as those in ethyl alcohol.

6. Limited testing of a quantity (155 gm) of magnesium (mixture of chips, turnings, granules, coarse powder and ribbon) indicates that metal fires of similar size do not produce enough heat at a high enough rate to activate conventionally located heat detectors quickly.

Smoke detectors can be expected to sense smoky metal fires reasonably quickly; but not all combustible metals produce smoke in readily detectable quantities.

Generalized experimentation is not particularly suitable for detector performance in metal fires as no combustible metal has a burning behavior reasonably representative of all the combustible metals. Further, burning rates vary widely, depending on the shape of the metal (powders, granules, chips, solid bulk pieces, etc.).

Where a metal is subject to criticality restrictions, only a limited quantity can be expected to be involved in a glovebox fire. Such quantities of burning metal may be difficult to detect by ordinary heat detectors.

SUGGESTED CRITERIA FOR SELECTING AND INSTALLING FIRE DETECTION EQUIPMENT

General:

1. Only approved detection devices and systems should be installed. Information on suppliers of approved detection and alarm systems is found in the Factory Mutual Approval Guide (15), and Underwriters' Laboratories Fire Protection Equipment List (73).

2. The systems should be installed in accordance with nationally recognized standards for protective signaling systems and with the manufacturer's instructions.

3. The systems should be regularly inspected and tested by trained personnel.

In Gloveboxes:

1. Self-resetting detectors should be used in gloveboxes, to avoid replacement of "one-shot" devices. One common type functions by the closing and opening of electrical contacts due to thermal expansion of bimetallic elements.

2. Conventionally located heat detectors are not suitable for detecting fires in small quantities of combustible metals.

At one AEC facility, a heat detector is connected to a terminal point by a flexible extension cable, permitting the operator to locate the detector near any accumulation of combustible metal (Fig. 6-2).

3. Smoke and heat detectors should generally be mounted at or near the center of the glovebox ceiling. For boxes larger than 4 ft. x 4 ft. x 4 ft., more than one spot detector will be needed to adequately cover all areas of the box. Local tests are recommended for determining locations of detectors for specific fires.

4. Select the lowest practical temperature rating when using fixed temperature detectors. Generally, the rated operating temperature of a heat detector should be at least 25°F higher than the maximum ambient temperature.

5. For detecting fires in cellulose or flammable liquids, pneumatic tube detectors (rate-of-rise) and eutectic salt tube detectors (fixed temperature) offer greater sensitivity than a single spot detector. In long individual gloveboxes and long glovebox trains, one continuous-type detector may be equally effective as and less expensive and complex than multiple spot detectors.

6. If pneumatic tubing is used, a system with 25 ft. to 40 ft. "equivalent" chambers is more sensitive than the tubing alone.

7. Smoke detectors furnish more prompt detection than heat detectors for cellulosic materials. Smoke detectors will require frequent cleaning in dusty or fume-laden atmospheres.

8. To favor most rapid detector response, a low ventilation rate or a static glovebox atmosphere is preferable, other factors permitting.

9. Corrosive, highly humid, or high-temperature atmospheres may cause deterioration of detector components. Where possible, select materials of detector construction suitable for long term use under particular operating conditions. Protective coatings may be helpful as long as sensitivity is not significantly reduced.

10. Where necessary, protect detectors and associated wiring from mechanical damage.

In Glovebox Rooms and Buildings

The proper use, location and spacing of fire-detecting devices should be carefully considered, to assure prompt reliable operation at time of emergency. In laying out a system, ceiling construction, ceiling height location of partitions, normal room temperatures, unusual air currents, and heat or smoke from operations and heating systems should be considered. Where an entire area is protected, the maximum distance between devices, and between ceiling-mounted devices and side walls is normally specified

Fig. 6-2. Heat detector resting on a can containing fine plutonium chips coated with cutting oil, at a lathe glovebox. Another detector is placed directly in the oil of the lathe coolant pan. Both detectors are wired to a central "locator" board. (Dow Chemical Co., Rocky Flats Div.)

by code or by the manufacturer, so that fire or smoke in any part of the area will be promptly detected. Closer than maximum spacing may often be preferable, where determined by construction, occupancy, protection, and values involved.

1. Fire detection systems (heat or smoke actuated) are generally recommended for all glovebox areas, except where automatic fire protection systems with alarms are installed.

2. Automatic detection systems should be installed in all areas protected by manual sprinklers or other manual extinguishing equipment (hose lines, portable extinguishers); and wherever there are unattended operations.

3. The need for, type, and location of fire detectors in ducts will depend on individual conditions, and installations should be custom-designed. General practice has been to install detectors on the downstream side of high-efficiency filters; ionization type smoke detectors, downstream of filters, have been successfully used at Oak Ridge (10). Infra-red flame detectors on the face of filters and smoke detectors downstream may also be suitable in small ventilation systems. Where filters and ductwork are noncombustible and not subject to loading with combustible deposits, there would usually be no need for fire detectors within the ducts in addition to the boxes.

4. The temperature rating of fixed temperature heat detectors should be the lowest practical, compatible with normal operating conditions, to assure fast response and to minimize the possibility of undetected small fires.

5. Where heat detectors are applicable, self-resetting devices should generally be used to avoid the need of replacement after an emergency, and to assure repeated action if a fire rekindles.

6. Detectors are generally mounted high in an area, since most types rely on the convection of heated air or smoke. Except in special cases, partial protection (e.g. detectors on only part of ceiling) is unreliable. Complete protection may require detectors under benches, among shelves, and in closets.

7. The use of an indicating "locator" panel board as part of the detection system is desirable. This permits pinpointing the location of a fire, with more efficient and effective fire fighting response.

8. All detection systems should be regularly inspected and tested. A distinctive trouble-signal system should be incorporated, so that failure of the power supply or a break in a circuit wire will be indicated. The trouble signal should be on a separate circuit. Power should be from a reliable a-c circuit, backed up by storage batteries capable of supplying current continuously for at least 48 hours.

7. FIRE CONTROL WITHIN GLOVEBOXES

Quick and effective control and extinguishment of glovebox fires is essential to prevent loss of containment, with resultant escape of radioactive materials, possible injury to personnel, and other serious consequences. Tests show that some glovebox components, such as gloves, may fail within 20 to 30 seconds under expected fire exposures. The glovebox is also vulnerable to rupture by explosions of flammable vapors, gases, and dusts.

A wide variety of extinguishing agents and application methods are available for glovebox protection. For some hazardous operations, an inert atmosphere system provides the most practical protection. Based on recent tests by the Factory Mutual Research Corporation, explosion suppression systems would have an application in some cases. In many gloveboxes, agents or extinguishers appropriate for the hazard may be located adjacent to or within the box for manual application by the operator.

Table 7-1 compares the suitability of various agents for glovebox interior fires.

EXTINGUISHING SYSTEMS

Extinguishing systems using water, carbon dioxide, dry chemical, halogenated agents (Halon 1301), or high expansion foam are suitable for extinguishing certain types of fires in gloveboxes. Each agent has its advantages and limitations which should be considered in the light of the hazard to be protected and the type of performance desired. Systems suitable for the protection of glovebox rooms are covered in Chapter 8.

Water Systems

Systems using water as an agent are suitable for protection of gloveboxes with contents, such as ordinary combustibles, that are compatible with water (49). At one facility, a fire detection system and a specially designed manually operated sprinkler system with small orifice, open spray heads are installed in a line of gloveboxes. The sprinklers in each box are controlled by a separate manual valve, to permit selective discharge of water spray into one or more boxes as needed. At another facility, a pressurized water extinguisher, located outside the glovebox, is piped to a spray sprinkler within the box (Fig. 7-1). For both of these installations water is compatible with the contents of the gloveboxes, and the limited discharge is sufficient to extinguish the fires which may occur.

Carbon Dioxide Systems

Carbon dioxide, as applied from a fixed cylinder (Fig. 7-2) is capable of extinguishing flammable liquid (alcohols, hydrocarbons) and cellulosic fires in gloveboxes (40, 49, 62). Fires in n-heptane, denatured alcohol, and paper towels have been successfully extinguished in controlled tests by FMRC. The discharged carbon dioxide reduces available oxygen content until

TABLE 7-1 COMPARISON OF AGENTS FOR GLOVEBOX INTERIOR FIRES

	WATER	CO ₂	DRY CHEMICAL ORDINARY MULTIPURPOSE		HIGH EXP. FOAM	HALON 1301	DRY COMPOUND ¹
Suitability for:							
<u>CLASS A</u> (ordinary combustibles)	Yes	No (some surface fires)	No (some surface fires)	Yes	Yes	Yes	-
<u>CLASS B</u> (Flamm. Liq.)	No (unless flashpoint over 150°F)	Yes	Yes	Yes	Yes ²	Yes	-
<u>CLASS C</u> (Electrical)	No	Yes	Yes	Yes	Yes	Yes	-
<u>CLASS D</u> (metals)	No	No	No	No	No	No	Yes ³
CRITICALITY HAZARD	Yes	No	No		Negligible ⁴	No	No
CONTAMINATION SPREAD HAZARD	High	Moderate	Low		Low ⁴	Low	Low
PRESSURIZATION	No	Yes	No		Possible ⁵	Unlikely	No
FILTER CLOGGING	Temporary Partial Plugging	Possible ⁶	Permanent Partial Plugging ⁷		Temporary Partial Plugging	None	-
DAMAGE	Possible	None	To Electrical Parts; General Cleanup Needed		Slight	Slight (decomposition products)	Electrical parts; general cleanup needed

FOOTNOTE:

1. These agents, in order of their relative effectiveness against metals fires, are: copper powder, magnesium oxide sand, lead powder, G-1 powder, ternary eutectic chloride, and ternary eutectic fluoride.
2. Water-soluble fuels (alcohols, esters, ketones) tend to break down foam; but in gloveboxes, the relatively large amounts of foam is expected to extinguish a fire in the small quantity of liquid normally found.
3. Suitability of several proprietary and non-proprietary agents varies, depending on type, amount, and configuration of metals. Proprietary powders are available in large pails for shovel/scoop application or in portable extinguishers for pressurized discharge.
4. The small amount (0.1 to 0.5%) of water in high expansion foam mitigates against valid objections to water on grounds of criticality risk or of contamination spread by water runoff.
5. Pressurization can occur if outside air is used to generate foam.
6. Discharge of carbon dioxide directly at or close to an exhaust filter may result in temporary clogging by CO₂ "snow". This sublimates rapidly.
7. Plugging is reportedly not a problem where atmospheric moisture is very low and, consequently, agent is dry; and where the agent is not discharged directly into the filter.

Fig. 7-1. Glovebox with sprinkler mounted under the glovebox ceiling. The sprinkler is connected by 1/2-in. tubing to an air-pressurized 2 1/2-gal. water-type extinguisher. (Oak Ridge National Laboratory)

Fig. 7-2. A glovebox for reclamation of cutting oil. Because of unattended operation, an automatic CO₂ extinguishing system is installed, actuated by heat detectors. (Dow Chemical Co., Rocky Flats Div.)

combustion is no longer possible. Because carbon dioxide has no permanent blanketing effect, it is not normally recommended for cellulosic fires where combustion becomes deep seated; for such fires the agent must be discharged for a prolonged period or the extinguishing concentration maintained to assure extinguishment of sub-surface burning. However, typical accumulations of paper towels or tissues in gloveboxes present more of a surface burning fire, so carbon dioxide can be successfully used. Carbon dioxide is not suitable for metal fires (68). The agent has been used to cool the exterior of metal gloveboxes during metals fires to improve the heat sink principle. According to FMRC tests with cerium, CO₂ appeared to accelerate the burning rate of the metal slightly.

One major advantage of carbon dioxide is that it does not produce any residue although CO₂ "snow" can plug a filter temporarily if discharged directly at it. No corrosive decomposition products are formed during its use.

The major disadvantage of carbon dioxide is that its use, at unregulated rates, may increase glovebox pressure to a degree threatening glovebox integrity (40). The increase will depend on the rate of discharge; ventilation rate; size, number, and condition of exhaust filters; and the type of fire. For a given glovebox and ventilation rate, there is an optimum rate of carbon dioxide that will extinguish a fire promptly without wasting agent or causing unnecessary additional pressurization.

Dry Chemical Systems

The dry chemical extinguisher (Figs. 7-3 and 7-4) consists of a portable cylinder containing dry chemical treated to maintain free-flowing characteristics and to prevent caking. The agent may be either under constant pressure or pressurized at time of operation; nitrogen, carbon dioxide, or dry air provide the pressure to expel the agent. One model is equipped with a fusible element to permit automatic operation.

Dry chemical is adaptable for use in fixed systems within gloveboxes. Dry chemical systems will provide rapid extinguishment of flammable liquid fires in gloveboxes (62). Potassium-bicarbonate-based agent has proven superior to sodium-bicarbonate based. Multi-purpose agents will extinguish cellulosic as well as flammable liquid fires. Regular dry chemical agents are not reliable for deep-seated fires in cellulosic material, although they can extinguish or control surface fires. Dry chemical agents, applied at proper rates, almost instantaneously reduce any pressure caused by a fire. Little or no pressurization of the glovebox is caused by the agent discharge itself. Dry chemical agents extinguish fires very rapidly, mainly by an interruption of the combustion chain reaction. However, ignition sources must be removed or controlled to prevent re-ignition.

Dry chemical agents may produce filter clogging, thus reducing air flow through the glovebox. Unlike carbon dioxide, considerable dry chemical residue remains after discharge, requiring cleanup.

According to FMRC tests, with 2 3/4-lb. extinguishers piped through the glovebox wall, proper design and location of the nozzle is needed to assure adequate and even distribution of agent. Different shape nozzles are available.

Fig. 7-3. Dry Chemical extinguishers: at the left, with hose and fitting for attachment to quick-coupling connection as shown in Figure 7-4; at right, with glove-piercing nozzle. (Atlantic Richfield Hanford Co., Richland, Wash.)

Fig. 7-4. Manual application of dry chemical extinguisher agent through quick-coupling connection installed on window. (Atlantic Richfield Hanford Co., Richland, Wash.)

Where the use of dry chemical agents is planned, the compatibility of the ingredients with glovebox contents should be determined. Potassium and sodium bicarbonate based agents, as well as "all purpose" powders, are not suitable for metal fire control. The "all purpose" powders are corrosive because of their acid base. The smallest currently approved fixed dry chemical system for industrial occupancies has a 30-lb. agent capacity and is suited for volumes up to 780 cu. ft. and floor areas of 17 1/2 ft. x 10 ft. (15) A scaled down version, designed for gloveboxes, would be expected to provide excellent protection for flammable liquid fires; and for cellulosic fires where a multi-purpose agent was used. Hand portable extinguishers are presently available with up to 30-lb. capacity (21).

Halon 1301 Systems

Halon 1301 (bromotrifluoromethane, CBrF_3) has recently been used in commercial and military aircraft for protection against in-flight engine fires. FMRC tests show that this agent is highly effective against flammable liquid and cellulosic test fires in gloveboxes. However, the agent is not effective against plutonium fires.

The major extinguishing mechanism is believed to be a chemical chain-breaking action (34). Halon 1301 is a liquefied compressed gas stored and handled in cylinders. It has a boiling point of -72°F and a freezing point of -270°F (58). Limited testing has indicated considerable extinguishing superiority over other halogenated agents such as carbon tetrachloride (Halon 104), methyl bromide, and chlorobromomethane (Halon 1011) (34,58); as well as over carbon dioxide (12, 23, 35).

The pyrolytic decomposition products are acidic and toxic (12, 35, 58). However, during a glovebox fire, the effect of acidic corrosion would normally be of little concern because of the very short length of time acidic products are released; and toxic products inside a glovebox would not normally be of concern to personnel. After repetitive discharges (such as occur during a series of fire extinguishing tests), a noticeable milk-colored film forms on surfaces. An extended period of exposure to the acidic decomposition products at elevated temperatures is corrosive to glass, glass fiber and metals (12, 35).

In the Underwriters' Laboratories "Classification of Comparative Life Hazard of Gases and Vapors", undecomposed Halon 1301 has been assigned to Group 6: gases or vapors which in concentration up to about 20% by volume for durations of exposure of the order of 2 hours do not produce injury. In this classification, Halon 1301 ranks as less toxic than carbon dioxide (12, 58, 70). The undecomposed agent, at room temperature, has been rated less corrosive to various gasketing materials (except ethyl cellulose and cellulose acetate butyrate) than carbon tetrachloride and bromochloromethane (12, 35).

High Expansion Foam Systems

High expansion foam is a fluid aggregate of small gas or air-filled bubbles in which the ratio of foam volume to water volume may approach 1000 to 1. The foam volume is usually generated by forcing air (or other gas) through a screen that is continuously supplied with a film of water containing a foaming agent.

Extinguishing action is primarily by decreasing the oxygen supply to the fire, by steam generated at the fire, and by shielding the fire from natural air currents. The water content of the foam also provides a beneficial cooling effect and some wetting action, while minimizing water damage.

High expansion foam was originally developed to fight inaccessible fires in coal mines (47). In recent years, commercial equipment has been designed and successfully used to protect large industrial areas (rooms, buildings).

Experimental Designs. Two experimental high expansion foam systems are shown in Figures 7-5 and 7-6. They were used by the Factory Mutual Research Corporation to evaluate the use of high expansion foam for the protection of gloveboxes. Each system was custom-designed for particular gloveboxes.

The design in Figure 7-5 was developed at the Lawrence Radiation Laboratory, University of California at Livermore (47). Argon or nitrogen aspirates the foam agent solution and projects it against a conical cotton net screen. The generated foam is discharged directly into the glovebox. A fire detector actuates a solenoid valve in the gas supply line, automatically starting the foam production. The foam unit is normally separated from the contaminating glovebox atmosphere. The initial gas flow opens a valve that normally separates the foam generating chamber from the glovebox.

The quantity of foam agent solution is controlled to limit the total foam discharge to a fixed amount (e.g. one box volume), followed by inert gas discharge alone. Since the system supplies a volume of foam and gas to the glovebox interior in addition to the volume added by the fire-gas atmosphere, the foam generation rate must be design limited to avoid excess pressurization.

The other design (Figure 7-6) uses recirculated glovebox atmosphere to generate foam. This arrangement essentially consists of a blower feeding air through a 6-in. duct to a conical screen against which foam agent solution is discharged from a small spray nozzle. Generated foam is discharged directly into the enclosure. The foam generator is not physically separated from the glovebox and would be considered a contaminated part of the entire system.

Current investigations seem to indicate that some products of combustion, such as those produced by certain burning plastics, and certain oil fumes, break down the foam, so that the use of recirculated air appears to be limited in some situations.

Extinguishing Capability. High expansion foam will successfully extinguish fire in cellulosic materials and most flammable liquids and plastics (15, 47). The effectiveness is partly dependent on a controlled generation rate and consistent uniformity of foam quality. High expansion foam has not generally proven effective against metals (magnesium, cerium) fires (47). The use of an inert gas (argon), instead of air, to produce the foam does not improve extinguishing ability against burning metals.

Fig. 7-5. Schematic diagram of high expansion foam system developed at Lawrence Radiation Laboratory, Univ. of California at Livermore, for glovebox protection.

Fig. 7-6. Schematic diagram of high expansion foam system, designed by Factory Mutual Research Corp., using recirculated glovebox atmosphere.

Large Volume Protection. A major advantage of high expansion foam is its ability to protect against fires within a large space by total flooding of the volume, without the exact knowledge of the location of the fire and the need for directional discharge of the agent. In large individual gloveboxes and glovebox production lines, without fire cut-offs, an incipient fire, aided by ventilation, may potentially travel the entire length of the containment system. High expansion foam can be introduced at any point in the system, reaching a fire at any other location and stopping the spread of fire to other portions of the containment system.

Presently no high expansion foam equipment approved by a nationally recognized testing organization is available for the protection of small volume individual gloveboxes commonly found in laboratories and research operations. However, there are commercially available foam generators in the 1000-CFM range that are suited to large glovebox systems (15). Such a generator can be arranged to attach to and discharge into any glove port or other opening. The generator can be designed to automatically start and stop on signal from a detector mounted in the box. In AEC fire tests at Richland, Washington, high expansion foam suppressed a simultaneous cellulosic and flammable liquid test fire in a 20-ft. long glovebox within 20 sec. after receipt of fire detector signals; the 450-cu.-ft. enclosure was completely filled with foam in about 1 minute (61).

Shielding Ability. An advantage of high expansion foam is its ability to shield uninvolved combustible material from ignition. In gloveboxes this would help to prevent strong radiant heat or burning particles from igniting gloves or other combustible components of the box.

Retention of Contaminants. There is evidence that high expansion foam has some ability to capture and retain contaminants that might be spread from the smoke or fumes of a fire (69, 76). In cases where partial loss of containment might occur during an otherwise successful fire suppression by high expansion foam, contaminant retention by the foam would be helpful.

Effect on Glovebox Pressure. Where a foam generating gas is taken from outside the glovebox, the generation rate of foam must be carefully controlled and coordinated with the ventilation rate to avoid excess pressurization. To prevent sustained positive box pressures, the rate of foam generation must not exceed the box ventilation rate. In one series of FMRC tests, a rate of foam input equal to the ventilation rate resulted in a positive glovebox pressure of 1/2 in. of water as long as foam input was continued, despite extinguishment of the fire. To prevent this, a glovebox ventilation rate higher than the foam generating rate is desirable. In several tests with alcohol fires, a ventilation rate twice that of foam generation prevented glovebox pressures from going positive while the fires were being extinguished.

The use of the glovebox atmosphere as a gas source avoids the need for correlating the foam generation rate with the ventilation rate. No additional gas volume is introduced into the glovebox beyond that generated by the fire itself. Since the fire gas atmosphere used will contain less oxygen than outside air, this method improves combustion suppression. Since certain products of combustion may break down the foam, the use of this method is limited, if actuation of the system is excessively delayed.

The glovebox design as described presents two major disadvantages: exposure of the foam equipment to the contaminated atmosphere of the glovebox during normal operations; and certain products of combustion may break down the foam. Appropriate valving of the generating equipment inlet and outlet can prevent contamination, but adds further complications to an already rather complex system.

Effects on Filters. A major disadvantage of high expansion foam is that it can clog high efficiency filters (by wetting) so that air flow will cease within several minutes. Foam can be prevented from reaching the filters, however, by locating them at or above the top of the glovebox and arranging the foam generator to automatically shut off foam production before the box is filled. This can be accomplished by using a heat actuated fire detector to start and stop the generator. In some cases, the fire will be extinguished and the generator automatically shut off before the glovebox is more than half filled with foam. According to FMRC tests, prefilters (demisters) can prevent foam contact with filters, but the prefilters themselves will clog, resulting in pressurization.

Suggested criteria for development of high expansion foam systems for small volume gloveboxes are presented in Appendix II.

MANUAL EXTINGUISHING EQUIPMENT AND METHODS

Extinguishing agents, appropriate for the types of fires to be expected, may be located adjacent to or within the glovebox for manual application by the operator.

For small flammable liquid or cellulosic fires, dry chemical (regular or multi-purpose) in small containers or small extinguishers are suitable. Metal covers can be provided to snuff out fires in some cases, as where flammable liquids are used in small containers.

For fires in pyrophoric metals, materials such as magnesium oxide, graphite, copper powder, sand (8), low melting eutectic salt mixtures (chlorides and fluorides) and proprietary dry-powder agents are used. The powders are kept in the glovebox in metal containers for pouring over the burning metal, or in plastic bags which are placed directly on the metal. In the latter case heat melts the plastic and allows the powder to flow over the burning metal, but may cause molten metal to spew out from under the bag. The relative effectiveness of various extinguishing agents on metal fires is discussed in Chapter 2.

Extinguishing agents in pressurized portable extinguishers can be applied from outside the box through a quick coupling connection installed in the glovebox wall. They can also be applied from outside by piercing a glove with a special piercing nozzle installed on the extinguisher (Figs. 7-3 and 7-4). Where a piercing nozzle is used, its design should be such that the pierced glove will effectively seal the puncture, to prevent escape of contamination.

Small metal fires can sometimes be permitted to burn to the oxide under safe, controlled conditions (8, 53, 62).

EXPLOSION SUPPRESSION SYSTEMS

An explosion suppression system is an assembly of components designed to detect a combustion explosion in its earliest stage, and act to prevent further growth to maximum pressures. The success of this system is based on the comparatively slow rate of pressure rise during the first 15 milliseconds of an explosion (33). Detection and discharge of a halogenated suppressant occur within the first few milliseconds following ignition, suppressing combustion and the build-up of destructive pressures.

Because explosion suppression systems have been successfully used to protect enclosures in aviation and industry, a similar system would seem feasible for gloveboxes. As discussed earlier, the halogenated agents have proven highly effective on glovebox fires in cellulosic material and flammable liquids. An effective dual purpose explosion and fire protection system can be designed, with a pressure-rise explosion detector and a rate-of-temperature rise fire detector wired in parallel so that either can actuate a common extinguishing system of suppressor units. Tests with the FMRC experimental system, discussed later, demonstrated that such a system can successfully suppress an explosion or extinguish a cellulosics or flammable liquid fire in gloveboxes.

Background

Originally, explosion suppression systems were developed during World War II for the protection of aircraft fuel tanks from small arms fire and incendiary bullets (22, 33). Later, systems were engineered for more general protection, to suppress the explosive ignition of fuel vapor attributed to lightning, static electricity, and exposure fires in broken fuel lines. More recently, systems have been installed at industrial properties to protect operating equipment where flammable vapors or heavy dust concentrations exist (22).

Properly engineered explosion protection systems have provided fast, reliable action at various industrial processes as well as in controlled tests. Test explosion of 2 gal. of JP-4 fuel air mixture at 400°F and 2 atmospheres resulted in peak pressures of over 80 psig in 250 msec; with a suppression system operating, peak pressures were only 1.2 psig at about 110 msec (32). A phenolic resin test explosion in a 500 gal. tank reached 12 psi in 100 msec before a rupture disc failed (2). With five 500-cc bromochloromethane suppressors acting, peak pressure was only 2-3 psi in a few msec. At a plastics manufacturing plant, unsuppressed explosions at a hammer mill had commonly peaked at 65-100 psig (1); a suppression system kept maximum pressures at 4-5 psi.

Operating Principle

Detectors in an explosion suppression system operate by detection of pressures (rate-of-rise or fixed pressure level), of temperature, or of radiation. In some types of explosions, radiation intensity increases more rapidly than pressure (33). For example, in aircraft protection systems, radiation detectors are commonly installed to sense visible and near infrared radiation from explosions of hydrocarbon fuel (33, 37).

A detector generates a signal to an amplifier power supply unit which discharges a capacitor through the suppressor units. At the suppressor, a small blasting cap operates to start the rapid discharge of suppressant, commonly a halogenated fluorocarbon (Halon group), at over 200 ft./sec. (51). Suppressant may be discharged at speeds up to 600 ft./sec. depending on the type of suppressor (36). Rapid discharge permits high extinguishing capability. For example, 2 cc of bromochloromethane can suppress an explosion in a 1 gal. volume.

At industrial installations, an explosion protection system is usually engineered to take other counter-actions against explosions, in addition to suppression (22, 33). These are: explosion venting by burst discs or similar devices; advance suppression or inertion further downstream in equipment to prevent chain explosions or spread of fire; shutdown of machinery; isolation of equipment or plant areas; and sounding of alarms. Experimental work has also been done in enrichment, i.e. suppression of a flammable vapor explosion by rapid discharge of the same flammable vapor to make the vapor-air mixture too rich for continued explosive combustion.

Advance inerting has been applied in processes where explosive dusts, burning material, or flammable vapors pass through ducts so that chain explosions or flame propagation is prevented (22, 33). A similar installation may be feasible for the protection of a glovebox and exhaust ventilation ductwork.

FMRC Tests

For FMRC's explosion suppression project, a homogenous 5% propane-air mixture was established in a 24-cu.-ft. glovebox (Fig. 7-7). This slightly rich air-propane mixture is easily ignitable, has the highest flame spread of any air-propane mixture and produces the highest pressures of any propane-air mixture. Three suppressors were installed, each containing 500 cc of halogenated agent (dibromotetrafluoroethane). The pressure detector was set at 0.25 psig.

Results of two tests follow:

	<u>Test No. 1</u>	<u>Test No. 2</u>
Time from Ignition to Reach 0.25 psig (milliseconds)	160	132
Time from Ignition to Suppressor Actuation (ms)	170	142
Maximum Pressure (psig)*	1.04	1.0
Time (ms), Pressure over 0.25 psig	34	34
Time (ms), Pressure over 0.5 psig	10	7
Time (ms), Pressure over 1.0 psig	1	0

*Although the indicated maximum pressure is high enough to fail some glovebox components, the pulse is of such short duration that, in fact, no important stress occurs.

Fig. 7-7. Schematic diagram of Fenwal explosion suppression system installed in Factory Mutual Research test glovebox.

Fire extinguishment capabilities of the explosion protection system were also tested. Using a fixed temperature (125°F) detector and one 500-cc suppressor in a 180 cu.-ft.-enclosure, a fire in a standard 48-paper-towel fuel accumulation was extinguished within 34 sec. of ignition. Maximum temperature recorded was 140°F, while in the unprotected glovebox, temperatures reached 510°F.

Maximum pressure increase in unprotected glovebox averaged 0.85 in. H₂O as compared to 0.45 in. H₂O in the protected enclosure. Duration of pressure above atmosphere averaged 43.5 sec. in the unprotected glovebox, while in the protected enclosure, the duration was only 12 sec.

Application of Explosion Suppression to Gloveboxes

The application of explosion suppression systems to gloveboxes remains largely on an experimental basis. Based mainly on the tests performed by the Factory Mutual Research Corporation, some general considerations may be listed regarding the possible use of explosion protection systems in gloveboxes.

1. The provision of an explosion suppression system in a glovebox - a relatively weak structure - would probably enable a glovebox to withstand the maximum pressure pulse expected from a suppressed explosion. Ventilation inlets and outlets, the resilience of the gloves, and the mixture homogeneity throughout the enclosed volume are factors that favor successful suppression.
2. Explosion suppression systems can protect against explosions involving mixtures of solid particles and vapors with air; and fires involving cellulose or flammable liquids. They are not recommended for protection against non-air-dependent fires and explosions.
3. Although explosion suppression components are standard hardware, each system must be custom designed for the specific installation. Explosive and reactive characteristics of the hazardous materials should be determined; ambient temperatures and pressures should be considered; and the strength, volume, size, and configuration of the enclosure system must also be considered.
4. Some glovebox structures may require strengthening to withstand the dynamic loading of suppressed explosions and the loading caused by the reaction of activated suppressors. Particular attention should be directed to window fastenings, glove attachments, filters, ducts, and transfer ports.
5. Explosion suppression systems require the same adequate monitoring and maintenance as required for automatic fire protection systems. A reliable power supply, backed up by storage batteries, should be provided.
6. Alternate methods can be used to eliminate a combustion explosion hazard. Non-hazardous materials may be substituted in some processes; or an inert atmosphere system can be provided. Adequate "once-through" ventilation can help avoid the accumulation of flammable vapors. Gas analyzers can be used to monitor the glovebox atmosphere to detect dangerous concentrations of combustible vapors.

8. FIRE PROTECTION OF ROOMS AND BUILDINGS HOUSING GLOVEBOXES

Glovebox integrity can be lost through exposure to fire starting outside the box, as well as to one starting inside. And if the fire involves both the room and the glovebox, contamination may be spread over wide areas, with severe consequences to life and property. The fire safety of the glovebox environment (room or building) therefore demands as much or more attention as the safety of the glovebox itself. The fundamental considerations for fire safety of rooms and buildings housing gloveboxes are covered in this chapter.

CONSTRUCTION AND ARRANGEMENT

The preferred location for a glovebox installation is in a fire resistive building or area specially designed for the purpose and well separated or cut off from other areas. Noncombustible materials conforming to recognized standards and codes should be used to the greatest practical extent for all components of the building construction, finish, and furnishings (14, 15, 55, 71).

The building should be so arranged that gloveboxes are not unnecessarily exposed to hazardous processes or combustible contents. Combustibles within the room should be limited to the lowest practical amount. Normally closed metal cabinets should be used for storing combustibles that cannot be eliminated.

Containment of potential contamination is a vital consideration. Fires and explosions that rupture gloveboxes can result in serious release of radioactive contaminants into the surroundings. Contamination can also be released through leakage, glove failure, and improper operating procedures. It is necessary therefore, to set up secondary controls if contamination is to be confined to as small a building area as possible.

Operations within the building should be arranged in separate zones according to their contamination release potential, and in a manner compatible with operating efficiency. For example, the building might be divided into a glovebox operations zone, a technical and service zone, and an administration or office zone (66).

Ventilation can be arranged to establish an air flow pattern from zones of lower to progressively higher contamination risk, as described in Chapter 5. In order to accomplish this, the glovebox operations zone must be constructed sufficiently tight so it can be maintained at a lower pressure than adjacent zones without excessive air flow rate. Weatherstripping of doors is one useful technique.

Gloveboxes and glovebox lines should be arranged to prevent spread of fire or contamination from one to another. Lines of boxes can sometimes be arranged with access corridors behind them so they can be serviced without the necessity of shutting down the work corridors.

PORTABLE EXTINGUISHERS

Portable extinguishers for glovebox rooms should be selected on the same general basis as those for any industrial occupancy; that is, the type or types most suitable for the fires that may occur. Several types of extinguishers are available: dry chemical, dry powder (for metals fires), carbon dioxide, foam, and plain water (14, 15, 55, 73). The "all purpose" dry chemical extinguisher is suitable for ordinary combustibles, flammable liquids, and electrical equipment. The carbon dioxide extinguisher is preferable to dry chemical for delicate electrical and mechanical parts as no residue is left for cleanup. Dry powder agents, in pressurized extinguishers or pails, should be provided as applicable for fires in metals such as uranium, plutonium, magnesium, aluminum, sodium, potassium and their alloys. Usually more than one type of extinguisher is provided for glovebox rooms.

Consideration should be given to the extinguisher being used on a fire starting within a glovebox as well as outside, in the room. A quick coupling connection as shown in Figs. 7-4 and 8-1 has been used to facilitate such use.

Details on approved extinguisher types and sizes, and operating and maintenance instructions are available from a variety of sources including the National Fire Protection Association (55), the Factory Mutual Engineering Corporation (14, 15), the Underwriters' Laboratories (73), and the extinguisher manufacturers.

Some drawbacks in relying on portable equipment alone are the limited range, quantity of agent, and duration of discharge. Only small fires are within the capabilities of these extinguishers. Great reliance must be placed on the human element. All personnel working in an area should be familiar with the operation of portable extinguishers, and their suitability for fires of different types.

WATER - General Considerations

At industrial properties generally, main dependence is placed on water agent systems for the suppression of fire. At many AEC glovebox facilities where there exists the danger of criticality, reactivity with certain materials, and contamination spread by run-off, water based systems, if available, are usually regarded as "last resort" protection⁽⁶⁹⁾. The goal of the fire safety program at such locations is to so limit combustibles and provide other safeguards that the need for application of water will be extremely remote. Nevertheless there are some locations where water will be required for the protection of the building or contents, even though radioactive materials are present. Special precautions are needed in such cases, in the installation and use of water-based extinguishing systems and equipment⁽⁶⁹⁾.

Control of the Spread of Water

There are several methods for confining water run-off to limit contamination spread and water damage (14). Curbed or ramped doorways, curbing around utility openings in floors, floor drains leading to holding areas or tanks, and sealing of floors in upper stories can be employed. Equipment and materials can be located in an elevated position, several inches above the floor, out of reach of water spreading or accumulating on the floor.

Fig. 8-1. Actinide processing glovebox in a plutonium chemistry laboratory, showing glove port covers, emergency alarm bar against which an operator can press, and quick-coupling connections for a dry chemical extinguisher. (Atlantic Richfield Hanford Co., Richland, Wash.)

When radioactive materials are being carried off in the water run-off, limited quantities of run-off may be drained to (1) a holding tank located outside the building or in the basement or to (2) a portion of the room itself designed on a lower level to serve as open storage. Larger quantities of water run-off may be drained to a diked reservoir, lined with impervious material to prevent seepage into ground water. Where acceptable, large quantities of water may be routed to a remote spill area where time and natural earth filtration would gradually reduce the contamination level.

STANDPIPES AND HOSE

Standpipes equipped with small hose and water spray nozzles should generally be provided. Their use should be restricted to persons trained in the use of water where radioactive materials may be present. Some AEC facilities have standpipes widely installed, but with hose and spray nozzles omitted in those areas where plutonium is present, thus restricting the use of hose streams to members of the plant fire department (53, 54).

AUTOMATIC SPRINKLERS

Ordinary combustibles can and should be eliminated from areas external to the glovebox. Where this goal is not achieved, and criticality is not a problem, automatic sprinklers can furnish the best fire protection for the glovebox room. An example of such an installation is shown in Fig. 1-3. At some AEC glovebox facilities (Rocky Flats, Los Alamos, and Richland) automatic sprinklers have been installed in loft areas under metal deck roofs having combustible vapor seals (53, 54). Sprinklers have also been installed in other areas at glovebox facilities, including storage rooms for waste, paint, chemicals and other combustibles. Where criticality is possible, limited volume systems or remotely controlled manual systems have been recommended (69). To date, AEC has not suffered a single major fire loss on any facility protected by automatic sprinklers.

Sprinklers where needed may help reduce contamination spread of airborne materials by putting out a fire that otherwise could spread out of control.

New Recycling Sprinkler System

A new type of automatic sprinkler system became available in 1967, designed to discharge enough water to suppress a fire, then shut down and reset itself, ready to cope with a rekindling blaze (16, 15). This new "on-off" system, designed by the Viking Corporation with Factory Mutual Engineering Corporation, not only detects and attacks a fire, but also automatically shuts itself off when the fire is extinguished or suppressed. Such an arrangement offers a new approach to protecting rooms containing gloveboxes where building construction is combustible, or where the contents of the glovebox rooms are sufficiently combustible to present a serious loss potential.

The heart of this recycling system is a hydraulically operated valve which is normally kept closed by water pressure immediately over the valve. This flow control valve is controlled by two solenoid valves installed in

parallel for increased reliability. When a fire occurs, thermal sensors, mounted throughout the protected area, break electrical contacts at 140°F and cause the solenoid valves to open, in addition to actuating an alarm. These valves, in parallel discharge pipes, drain the water over the flow control valve, which then opens allowing water to enter the entire system well before the sprinklers fuse.

When the temperature at the sensors drops below 140°F, their contacts close; an automatic timer allows the system to continue discharging water for an additional regulated interval as a safety factor. Then the solenoid valves close, and water begins to build up over the flow control valve, closing it in about one minute. Water remains in the system above the valve.

If a fire rekindles after the valve has closed, the sensors reactuate the contacts; again, the solenoid valves open, and the flow valve allows more water into the system.

Some advantages of the recycling system for protection of glovebox rooms are:

1. Water is used more efficiently than in conventional sprinkler systems; and chances of criticality or unnecessary water damage are reduced. The system automatically shuts off when a fire is suppressed. If sprinkler piping is broken by careless operation of a fork lift truck, freezing, roof collapse, or other reason, no water will discharge from the normally dry piping. In case of breakage, a pressure actuated switch will trip an alarm, indicating a drop in the air pressure normally maintained on the system. After a system has operated and shut down, it is unlikely that additional sprinklers will open if a fire rekindles, because the sensors are more sensitive and operate at lower temperatures than the sprinklers.

2. Uninterrupted fire protection is assured. After the system has operated, sprinklers may be replaced without shutting off the water supply valve. At all times, repairs or changes may be made to sprinkler piping without shutting off the water supply.

Battery power automatically becomes available when electric power is lost. If the batteries fail, the solenoid valves open automatically and the system becomes a conventional automatic wet-pipe system (i.e. water in all piping). If electrical repairs are needed, a manual adjustment makes the system a conventional dry pipe system (i.e. no water in piping). During these emergency wet pipe or dry pipe conditions, the main flow control valve will open automatically in case of fire, but will not close automatically. In general, the threats of a manual valve prematurely closed during a fire, or of a forgotten closed valve are removed.

HIGH EXPANSION FOAM SYSTEMS

Extensive test experience and growing, although still limited, field experience indicate that high expansion foam (see Chapter 7) can provide excellent protection for buildings and rooms (17, 18, 19, 20). Where criticality probability is so high in a glovebox facility that the use of

water is severely limited, high expansion foam has practical application (69). Because of limited reliability experience, high expansion foam protection is considered as a substitute and not an equivalent to sprinklers.

Approved commercially available high expansion foam equipment includes a range of portable unit sizes having generation rates from 1000 CFM to 12,500 CFM, or fixed automatically controlled systems with generating rates designed for a specific hazard and essentially unlimited in capacity (15, 73).

A major advantage of high expansion foam is that it can be applied at relatively safe distances without accurate directional flow. The agent can be introduced through a door, window or other opening, and it can be expected to reach and isolate the fire by its mobility and volume flooding capability. Personnel need not be exposed by close approach to those areas where airborne contamination, heat, smoke, and fumes would be hazardous. High expansion foam can be introduced into inaccessible glovebox ventilating systems to isolate the fire source, stop fire spread, and prevent breaching of tubing or ducts (61).

Another advantage of high expansion foam is the reduced water damage, as compared to damage by sprinklers or hose. Less water is used for equivalent extinguishing action. Electrical and electronic equipment suffer relatively minor damage (18, 47, 67). Cellulosic materials will absorb some moisture, but after dissipation of the foam and evaporation of the water content, most materials are usable (9). Little residue remains after evaporation, so that room cleanup is no problem.

HALOGENATED AGENT (HALON 1301) SYSTEMS

Extinguishing systems using Halon 1301, described in Chapter 7 for the protection of gloveboxes, offer promise for the protection of glovebox rooms as well. The agent is highly effective against cellulosic fires, flammable liquid fires, and electrical fires (12, 19, 35, 58). Also, Halon 1301 is not expected to increase the criticality hazard, result in the spread of radioactive contamination, or clog exhaust filters.

Holding a concentration of 5 to 6% of volume in air for the proper time, (seconds for flammable liquids and minutes for deeply charred wood) is sufficient to extinguish fires in most fuels on contact. These same concentrations make it impossible for combustible atmospheres to ignite explosively (12). Since the air flow in glovebox rooms is constantly changing, an additional flow of agent is required to maintain an inerted atmosphere. A recycling Halon 1301 extinguishing system designed to provide sufficient "soaking time" would require no clean-up work resulting from its use to extinguish a fire or after an accidental activation of the system.

In a pound to pound cost comparison with other fire extinguishing agents (water, foam, carbon dioxide), Halon 1301 is extremely expensive. However, in comparing complete system costs for other agents (piping and drainage for water, foam generating machinery, and cylinder hardware for CO₂), installed cost for a Halon 1301 system may be very competitive. The principal disadvantage is the lack of commercially available systems approved by a nationally recognized testing organization. In general, carbon dioxide system hardware

is employed at the present time, using up to one-tenth the number of cylinders necessary for equivalent protection by CO₂. Although fixed automatic systems utilizing Halon 1301 have not yet been fully developed, such work is now in progress. In the meantime, experimental systems are justified where exposure to personnel is minimized (see Reference 25).

Halon 1301 is an invisible and odorless gas in the undecomposed state. Undecomposed agent has been shown to be practically innocuous in ordinary inhalation (12, 35, 58). Halon 1301 gas can apparently cause death only by displacement of oxygen in the atmosphere to below safe limits; concentrations must exceed 50% to cause rapid death. In comparison, about 10% carbon dioxide may cause death if inhaled for a few minutes; while 1.28% concentration of carbon monoxide causes unconsciousness after two or three inhalations and likely death after two or three minutes (55). Halon 1301 systems are being designed for extinguishing concentrations of only about 5% or less, for Class A fires (ordinary or cellulosic combustibles) so that toxicity of undecomposed agent is apparently of no concern.

The disadvantage of toxicity of the pyrolysis products may be significantly offset by fast detection systems combined with the rapid extinguishing capability of this agent. Relatively small quantities of agent may be needed (19); and contact with hot surfaces or combustion would be expected to be minimized.

APPENDIX I

GLOVEBOX FIRES AND EXPLOSIONS AT AEC FACILITIES (1956-1965)

<u>Date</u>	<u>Loss</u>	<u>Remarks</u>
9-11-57 Serious Accidents Bulletin 130	\$818,600	Fire in plutonium production line. Plutonium ignited spontaneously. Combustible filter at box burned. Fire spread to flammable plastic window and through ventilation system to ignite and destroy main filter bank. Serious spread of contamination.
11-10-60 Bulletin 175	101,000	Fire in glovebox in curium processing cave. Oil bath overheated. Combustible glovebox construction ignited. Equipment destroyed. Contamination released to room.
8-23-65 Bulletin 261	76,800	Explosion in acetone vapors while cleaning wax-like deposits from furnace. Vapors ignited probably by static spark. Three gloves ruptured. Fire controlled by 20-lb. dry chemical extinguisher applied through quick coupling connection. Decontamination costs \$76,200.
6-12-64 Bulletin 246	56,500	Explosion while degreasing plutonium chips. Chips ignited and accidentally fell into carbon tetrachloride bath, resulting in non-air-dependent chemical reaction type explosion. Window shattered. Employee injured. Decontamination costs \$43,000.
10-27-64 Bulletin 242	34,900	Explosion from undetermined cause in methanol vapors while evaporating plutonium oxide solution. Six gloves torn from their ports. Plutonium oxide discharged through opened ports and spread throughout operating areas.
7-6-60	31,300	Explosion (overpressure) of glovebox while purging equipment with argon. Malfunction of solenoid valves caused pressure to build up and blow off gloves. Contamination spread into several areas.
6-14-57	31,000	Explosion in vessel in glovebox. Gloves and window damaged. Decontamination costs \$11,000.
11-9-65	23,300	Explosion in acetone vapors ignited by muffle furnace in adjoining box. Paint containing 50% acetone being used for stripping and masking. Lack of communication between maintenance and technical groups.

<u>Date</u>	<u>Loss</u>	<u>Remarks</u>
10-15-65 Bulletin 262	\$17,000	Fire in plutonium chips in plugged drain. Plutonium carbon tetrachloride reaction initiated during forcible attempts to unplug line. Spread of contamination accounted for \$17,000 loss.
8-27-65	4,700	Explosion due to decomposition of nitrated resin being dried on hot plate in glovebox. Window blew out. Room required decontamination.
7-19-63	4,500	Fire caused by resin-nitric acid reaction in plutonium production line. Seven gloves burned through. CO ₂ extinguishers used on flames. MgO sand used on glowing embers in box.
9-20-58	4,500	Steam explosion in water jacket of graphite incinerator within glovebox. Valves to cooling water left closed by mistake. Interlocks to be provided.
3-16-56	4,400	Fire in vapors given off from ether dissolved in water, ignited by hot plate. Plastic glovebox window burned. Floor, walls, ceiling and ventilating ductwork contaminated.
6-28-65	4,400	Explosion while machining plutonium. Plutonium chips burned and reacted with trichloroethane. Window cracked, plastic pouch blew out. Area contaminated. Inspection equipment corroded.
2-16-63	2,700	Overpressure of glovebox used for hydroform press when carbon tetrachloride liquid filled box. Glove burst and contaminated carbon tetrachloride spilled on floor of room.
8-16-57 Bulletin 129	2,000	Explosion in flammable solvent being used to clean rust from lathe. Sparks from brush ignited vapors. 18 gloves blown out, 3 safety glass windows cracked.
8-6-59 Bulletin 148	1,900	Explosive chemical reaction of alpha emitting solution involving acetone and nitric acid. Gloves blown off. Safety glass window cracked. Most of loss due to decontamination costs.
3-31-59	1,700	Explosion from decomposition of trichloroethane inadvertently substituted for trichloroethylene at plutonium machining operation. Glovebox window blown off. Room contaminated.
11-4-63	1,700	Fire in glovebox caused by rag in contact with hot leach tub. Improper use of dry chemical extinguisher pressurized box and resulted in gross contamination of room.

<u>Date</u>	<u>Loss</u>	<u>Remarks</u>
11-22-58	\$1,100	Fire in improperly fused motor in shelf under chemistry glovebox. Promptly extinguished before fire got inside box.
10-8-63	950	Fire at pressure vessel for heating plutonium fluoride and calcium by induction. Seal ring failed. Molten metal spread into box and burned glove. Similar fire 5 hours earlier.
1-11-63	850	Fire at thorium melting operation. Plastic bags of thorium pellets being opened under argon atmosphere. In-leakage of air set stage for fire, which was probably caused by a static spark. Oxygen monitors will be installed.
9-14-65	750	Plastic dome imploded during pump-down phase of vacuum test. Vendor being sought for metal dome with plastic ports.
7-10-64 Bulletin 241	750	Explosion (disintegration) of plutonium being pressed. Gloves perforated. Operators had eliminated intermediate step in pressing.
10-24-60	650	Explosion in water solution of hydrazine nitrate being evaporated. A thermal ignition reaction occurred. Explosion blew out entire front of glovebox including frame.
5-3-65	650	Fire possibly caused by spark igniting oily deposits in flexible duct from portable glovebox used for burning plutonium chips. Combustible flexible hose burned through and ignited temporary plastic enclosure around casting furnace.
5-4-59	600	Fire in mixture of lithium, aluminum hydride, and anhydrous carbitol, due to chemical reaction when moisture entered nitrogen atmosphere. Plastic glovebox (wet chemistry) destroyed.
5-21-63	400	Fire in oil condensate dripping onto hot plate. Lithium being melted under oil. Process left unattended.
10-5-59 Bulletin 152	350	Fire in plastic tubing too close to furnace spread to plastic doors of enclosed walk-in hood. One sprinkler in room controlled fire and sounded alarm.
2-2-62	300	Fire at reduction furnace. Burning metal and slag ignited glove and burned through plastic window.

<u>Date</u>	<u>Loss</u>	<u>Remarks</u>
4-2-65	\$250	Fire in sodium-potassium alloy (NaK) chemically reacting with piece of teflon being used to skim oxides from surface of NaK. Glovebox not ruptured.
12-21-64	216	Fire in deep-well pump assembly, caused by electric spot light igniting rubber glove. Slight contamination was involved.
4-22-64	200	Fire in rag incinerator ignited gloves and plastic window and carried burning rags into adjacent box. Fire door between boxes was recommended.
9-22-59	200	Fire involved hydrogen leaking from an off-gas line at a hydrogen sintering furnace caused failure of snap rings on glove ports. Ignition was by the hot furnace or a spark from electrical equipment.
1-23-62	192	Fire in a research operation of heating metal rings. Torch ignited adhesive tape and involved paper tissue, absorbent paper, rubber gloves and plastic windows.
1-28-60	150	Explosion of dust in a rag incinerator. Two windows, broken and two gloves ruptured.
10-5-59	150	Fire in beryllium machining operation involved kerosene coolant which was ignited by a spark from cutting tool.
11-6-57	104	Fire in a wet chemistry glovebox resulted from a chemical reaction between butyl ethyl alcohol and NaK. Windows were cracked.
4-23-56	100	Fire in a wet chemistry operation resulted from an acid solution chemically reacting with nitride solvent.
7-21-58	75	Explosion in a graphite incinerator caused by clogged filters permitting oxygen build-up in glovebox atmosphere. Graphite dust ignited by match. Two windows blew out and one buckled.
6-9-59	50	Fire in plutonium briquetting glovebox occurred when plutonium briquettes ignited spontaneously after air leaked into a helium atmosphere box.
3-16-59	50	Fire involved peanut oil used for heating plutonium when oil was heated above its flashpoint. Operator plugged in heater instead of another electrical device.
10-30-58	50	Explosion in a wet chemistry glovebox involved propane-air mixture coming from loose hose connection. Ignition was from the torch. Flying glass from the safety glass front panel injured an employee.

APPENDIX II

CRITERIA FOR DEVELOPMENT OF HIGH EXPANSION FOAM GENERATORS
FOR SMALL GLOVEBOXES

The following criteria for high expansion foam generators adaptable to small gloveboxes are presented as an accompaniment to the recommendation that such generators should be developed. They apply to generators that would be used as fixed system units employed as integral parts of the glovebox protection system.

1. Capacity Range

a. The term "small gloveboxes" refers to all boxes having volumes less than could be suitably protected by adaptation of presently available foam generators, which are generally in the 1000 CFM and higher rate class. Development of a new class of generators should be in the 10-CFM to 500-CFM range.

b. These units should be made available

(1) either in two or three sub-ranges which together would span the overall 10-to 500-CFM range, providing for selection of a specific rate within a sub-range.

(2) or in several specific sizes in the 10-to 500-CFM range.

2. Control

The units should be equipped with all needed automatic mechanisms to enable arranging them for automatic start, stop, and repeat cycles on command from detection devices monitoring the protected space. At the same time, there should be a provision for reverting to manual start, stop, and recycle at any point in the automatically scheduled program.

3. Agent

Units should have storage capacity for enough foam concentrate to produce a foam volume based on the exhaust rate and box volume to prevent clogging the filters. This is usually one box volume or less.

4. Special Features

a. The units should be equipped with a barrier that would separate the equipment from the protected space during all normal (no-fire) conditions. This barrier should:

(1) Automatically open to provide for discharge of foam into the protected space on receipt of the first automatic or manual signal of a fire event.

- (2) Provide separation of the generator from the protected (contaminated) space equivalent to that achieved by the glovebox itself.
- (3) Automatically close at the end of the operation in a manner to permit removal and subsequent decontamination of the generator unit.

b. All surfaces of the generator that would be exposed to contamination during an operation should be minimized and so arranged as to facilitate decontamination.

c. Design of the generators should enable users to adapt them readily to any of three operation modes, namely:

- (1) Use of ambient atmosphere as a source of bubble-forming gas.
- (2) Use of an inert gas source for the bubble-forming gas.
- (3) Recirculation of the glovebox atmosphere as a source of bubble-forming gas.

d. Discharge ports of the generators should be arranged to adapt readily to glove port size openings of existing gloveboxes.

5. Maintenance Features

The generators should be designed to enable the following fire protection system monitoring procedures:

a. A weekly operational check of all functions, up to but not including actual formation and discharge of foam.

b. A monthly operational check of all functions. This should include formation of foam, but with arrangements for discharge outside the protected space to enable a check of foam generation rate and foam quality. Special means should be provided to maintain box integrity during operation of the barrier separating the generator from the protected spaces.

APPENDIX III

FACTORY MUTUAL RESEARCH ON PROTECTION OF
CLOSED CONTAINERS AGAINST INTERNAL EXPLOSIONS

Much of the early research on explosion behavior in closed containers was conducted by E. W. Cousins, P. E. Cotton and J. B. Smith of the Factory Mutual Laboratories. Research results were presented by E. W. Cousins and P. E. Cotton to the Second Process Industries Conference, ASME, and by J. B. Smith to the International Acetylene Association. They were also summarized in an article in Chemical Engineering, August, 1951.

The article is reprinted here in essentially complete form. The charts and accompanying data furnish basic information for evaluating and controlling maximum pressure effects of accidental flammable fluid combustion within gloveboxes.

* * * * *

Many pieces of industrial equipment - mixers, churns, reactors, autoclaves - may unavoidably contain mixtures of combustible gas or vapors. These gases may cause an internal explosion. To design a pressure vessel thick enough to withstand such a blast would be impractical (a tank normally operating at 25 psig would have to have its thickness increased by a factor of 10 to withstand the rapid stresses of an internal explosion).

The economical way to make these vessels safe is to reduce the pressure. In some cases, inert gas can be added to make the gas or vapor mixture non-flammable or to greatly reduce the flammability. An alternate method is through the use of bursting disks. The difficulty of this latter method is that very little data have been available with regard to the effect of disk strength and vent area on the maximum explosion pressure.

Except for some data on open vents for containers operating at atmospheric pressure, and a paper by Merl D. Creech entitled "Combustion Explosions in Pressure Vessels Protected with Rupture Disks" published in Vo. 63, No. 7 of the ASME Transactions, most of the data available are for closed vessels of 1-liter capacity.

Tests were conducted by Factory Mutual Laboratories with mixtures (at maximum explosiveness) of hydrogen and air and of propane and air. Initial pressures were 0, 15 and 45 psig. Ratio of venting area to volume was made variable in vessels of 1.1 to 20 cu. ft. capacity. Several strengths of disk were used. Results of these tests are shown in Figs. III-1 through III-10.

The hydrogen air mixture was intended to represent a relatively rapid-rate burning material; the propane air mixture, a medium rate burning material comparable to many solvent vapors. The particular concentration ratios of hydrogen to air and propane to air were chosen to give maximum pressure of explosion, maximum rate of burning, and reliability of ignition. The gases were thoroughly mixed and lighted by an electric match.

Fig. III-1. Test vessels used in explosion venting tests. First three vessels, left to right were used at 0 psig initial pressure; vessel at extreme right was used at 15 and 45 psig. L/D is ratio of length to diameter.

Fig. III-2. Results of explosions initiated at atmospheric pressure (0 psig) with open vents. Effect of L/D ratio is slight because container volumes do not vary in the same order as L/D. Because of small difference between explosion pressure in tank and that in drum, all later tests at 0 psig were run in tank.

Fig. III-3. Results of propane-air explosions at atmospheric pressure with open vents. Curve shows slower burning propane-air mixture is vented more easily than hydrogen-air mixture.

Fig. III-4. Effect of rupture disk strength and vent ratio on maximum explosion pressure of hydrogen-air mixture, initial pressure 15 psig. These maximum internal pressures can be applied to a vessel of similar L/D exposed by explosion of gases of similar burning rates and same initial conditions.

Fig. III-5. Effect of rupture disk strength and vent ratio on maximum explosion pressure of propane-air mixture, initial pressure of 15 psig.

Fig. III-6. Higher maximum explosion pressures are recorded for 40% hydrogen-air mixture when higher initial pressure (45 psig) than in Fig. III-4 is used.

Fig. III-7. Higher maximum explosion pressures are recorded for 5% propane-air mixture with higher initial pressure (45 psig) than in Fig. III-5.

Fig. III-8. Effect of vent ratio on maximum explosion pressures of hydrogen-air and propane-air mixtures, using 30-lb. and 100-lb. rupture disks.

Fig. III-9. Similar to Fig. III-8, but with 60-lb. and 200-lb. rupture disks.

Fig. III-10. Relation of maximum rate-of-pressure rise to initial pressure. Points for 0 psig initial pressure from Eastman Kodak work. Data for propane actually for acetone and toluene which have similar burning rates.

Disks, used for tests at initial pressures above atmospheric, were 2S-0 aluminum of various thicknesses. Disks of hard Monel, spring-temper brass, and soft copper were also used. All of the disk material (in the size and thickness to be used) was burst by subjecting each to a slow increase of air or nitrogen pressure.

Explosion pressures were measured with an electronic apparatus especially designed for the measurement and recording of explosion pressure. The pressure sensitive element is a condenser, one side of which is a diaphragm exposed to the pressure to be measured. Increasing pressure decreases the spacing between the plates of the condenser thereby increasing the capacitance. The remainder of the apparatus is a bridge circuit for measuring the change in capacitance of the sensitive element. The change is displayed on a cathode ray tube where it is recorded as an image of the pressure-time diagram shown by an oscilloscope. Timing marks are made at intervals of 1/60th of a second.

PRACTICAL APPLICATION OF TEST RESULTS

For practical use in design, results of the explosion research are covered in Figs. III-11 through III-14. Each graph covers all data for one gas and one initial pressure and consists of curves showing the relationship between explosion pressure and vent ratio for various disk strengths.

For example, the graph for a hydrogen-air mixture at initial pressure of 15 psig shows that the maximum explosion pressure that will occur in a vessel with no vent ratio (0.0) is 198 psig. If a vent ratio of 5 sq ft for each 100 cu ft of vessel volume is provided and equipped with a disk having a static burst strength of 25 psig, the explosion pressure will not exceed 90 psig.

When it is not practical to use a single large disk because of space limitations or because of the impracticability of obtaining the required strength in a large diameter disk, a number of smaller disks of the required strength totaling the required area can be used. This is because, while for any given volume and disk strength, the explosion pressure varies with the disk area, tests with varying volumes show that, in general, it is not the area alone that determines the explosion pressure but the ratio of vent area to volume.

Since the graphs were derived from tests of two gases at two initial pressures, good judgment will be required in their application to actual protection problems. Few such problems will involve materials or conditions exactly equivalent to the test conditions. However, it is possible, by proper selection of one graph or a combination of two graphs, to apply the derived data to most protection problems that may be presented. One general rule should be noted in determining whether to apply the data for hydrogen or propane to the problem at hand. Since hydrogen's rapid burning rate is not widely met in other gases, the hydrogen data should be used for problems involving hydrogen or any mixture of combustible gases containing more than 20% hydrogen. The data for propane should be applied to all materials having moderate or slower burning rates such as organic solvent vapors. If there is doubt of the characteristics of the materials involved and no data are available to indicate the proper selection, the safest course is to apply the data for hydrogen.

Fig. III-11. Maximum explosion pressures of 5% propane-air mixture, showing relationship between explosion and vent ratio, for various disk strengths. Initial pressure, 15 psig.

Fig. III-12. Maximum explosion pressures of 5% propane-air mixture, similar to Fig. III-11, showing higher pressures with higher initial pressure (45 psig).

Fig. III-13. Maximum explosion pressures of 40% hydrogen-air mixture, showing relationship between explosion and vent ratio, for various disk strengths. Initial pressure, 15 psig.

Fig. III-14. Maximum explosion pressure of 40% hydrogen-air mixture, similar to Fig. III-13, showing effect of higher initial pressure (45 psig).

The application of the explosion pressure vent ratios relationship has certain limitations determined by the scope of the investigation from which it was derived, and the following specific instances are given to govern the use of the data developed.

1. These design graphs are not applicable to pipelines or long tanks. In such containers, a "detonation" can occur for which the maximum pressures are thousands of pounds per square inch and venting is totally ineffective. At atmospheric initial pressure, the critical L/D ratio is about 60 which reduces to 30 at an initial pressure of 1 psig. Critical lengths for higher initial pressures have not been established.

2. Initial operating pressures above the scope of the investigation are not covered by it, although straight line extrapolation of the data up to 60 psig is probably safe. For initial pressures below 15 psig, straight line extrapolation to 0 psig can be made with safety.

3. For temperatures much higher than the range of the investigation (50°F to 90°F), the data should be applied with caution. As the temperature increases, the quantities, by weight, of combustible decrease, other things being equal. At the same time, the initial energy level increases with the result that it is not known specifically how extreme temperatures affect explosion pressure or ventability.

4. The data apply to vents having an unobstructed discharge to atmosphere (after the disk bursts). That is, they are not applicable to vents discharging into ducts or other confining spaces.

5. Since an explosion vent is a method of spreading out a quantity of energy which would be harmful if confined, caution should be employed in determining where that energy can be spent safely. The placement of vents should be such that their discharge would not be directed at flimsy construction such as thin partitions, glass windows, etc. Nor should their discharge be directed in a manner such that personnel would normally be within the discharge path while the hazard exists.

APPENDIX IV

CRITERIA FOR FMRC GLOVEBOX FIRE TESTS

The design of valid fire tests in gloveboxes is complicated by the many possible behavior patterns of accidental fires, and the variety of glovebox designs, ventilation characteristics, and specific operations. Some simplification of design and substitution of materials is usually necessary to permit repetition of tests and savings in time, labor, and material. At the same time, care must be taken not to sacrifice the validity of the tests. One favorable aspect of glovebox fire testing is that full scale tests are feasible.

The following criteria for tests were found useful by FMRC in their studies of glovebox fire behavior. They are offered as a guide to those doing further research in the field. Other useful information on test methods used by FMRC and others is summarized and referenced in Chapter 2.

1. GLOVEBOX

A. General Requirements

Design to conform to gloveboxes being used or to be used.
Design to withstand repetitive fire testing (e.g. wired glass windows.)
Design to facilitate field fabrication and modification.

B. Dimensions and Volume

Rectangular shape.
Size and volume to conform to commonly used enclosures.
Examples:
34 1/2 in. x 18 in. x 28 3/8 in. high - (10 cu. ft.)
41 1/2 in. x 24 3/4 in. x 40 in. high - (24 cu. ft.)
71 1/2 in. x 40 1/4 in. x 40 1/4 in. high - (67 cu. ft.)
95 1/4 in. x 46 1/4 in. x 70 1/4 in. high - (180 cu. ft.)

C. Assembly Details

1. Box: glovebox panels (except end panels) to be joined with permanent joints (e.g. 3/4 in. plywood rabbetted, glued, and screwed; interior surface lined with asbestos millboard).

Extra bracing to resist pressure rises.

End panels removable for access (e.g. join to box against gasketed flanges, fasten by wing nuts on lugs in the flanges).
2. Window: permanently clamped to front panel with gasketed joint, large enough to permit observation of test fires.

3. Glove Ports: two standard 8 in. fittings (e.g. Zollinger SN-101, 8 in.); to accommodate gloves or cover plates.
4. Access Ports: simple hinged doors with airtight closure, if no radioactive materials used.
5. Miscellaneous: enclosure to include leak-tight provisions for entry of thermocouples, electricity, gas and water lines, instrument probes, detectors, and quick-coupling connections for application of agents.

II VENTILATION

- A. Design and operating characteristics to conform to actual gloveboxes.

Example:

1. Range of airflow rate from less than one box volume per minute to slightly more than one box volume per minute; 0 to 110 CFM or rated flow of filter used.
 2. Range of glovebox pressures - generally negative - to conform to commonly used pressures (e.g. - 0.25 in. H₂O to - 0.80 in. H₂O).
- B. Size and location of inlet and outlet vent openings and filters to conform to most prevalent designs in use. (e.g. inlet at bottom rear of glovebox; outlet on top).

III TEST FUELS

- A. General Requirements

1. Fuels to be representative of or the same as those fuels commonly found in gloveboxes.
2. Fuels to be of standard composition, size, and heat output; permit variation in fire size.

- B. Fuel Types

1. Cellulosic (Class A Fires)

Example:

Paper hand towels, Scott, (C-fold) #150, wet strength ultra-high absorpency, weight of each towel 5.5 gm.

2. Flammable Liquid (Class B Fires)

Example:

Industrial ethyl alcohol - completely denatured, clean burning to minimize build-up of soot.

Heptane - to represent those flammable liquids that burn with heavy sooty smoke and with higher heat output; and to permit study of smoke detectors and filter clogging.

Other possible fuels - acetone, lard oil, transformer oil, peanut oil.

3. Metal Fires (Class D Fires)

Example:

a. Magnesium-turnings, chips, granules, powder, ribbon, or, any type and configuration of interest.

b. Plutonium (if available; otherwise cerium) - solid mass, turnings, casting skulls, etc., as applicable.

C. Fuel Arrangement and Size

1. Cellulosic - arrange and pack in a manner to minimize variation in fire spread characteristic of cellulosics fires.

Example:

Crumple paper towels individually and pack in standardized manner; pack in wire basket, 3/8-in.-mesh galvanized hardware cloth; basket height: 4-1/2-in.; basket diameter varied according to amount of fuel.

2. Flammable liquid - provide range of fuel areas in a noncombustible container.

Maintain approximately same depth (not a critical factor as flammable liquid fires typically occur at the plane, horizontal surface of the liquid whether in a container or as a random spill).

Example:

0.25-in. deep fuel in 2-in. deep metal pans, 0.06-sq.-ft. to 9.0-sq.-ft. areas.

3. Metals - one or several sizes - accumulation typically found, or permitted to accumulate where high standards of order and neatness prevail.

Example:

35 gm. magnesium heaped to 1 1/2-in. deep in 3/4-in. deep by 6 1/4-in. diameter metal pan;
Plutonium: block, 6 cm x 4 cm x 3 cm.

D. Ignition Procedure

1. Cellulosic fire - match; Bunsen burner; cigarette lighter; ignition time considered to be when first flame observed.

2. Flammable Liquid - match,
other sources: mantel heater; immersion heater; tesla spark
coil, depending on quantity of fuel and size and shape of
container.
3. Metals - black gunpowder placed in depression on top of metal
pile; ignited by match. 2.7 gm gunpowder needed to ignite
35-gm pile of magnesium mixture in III-C3. Metal blocks
ignited by gas-air torch.

IV SPECIFIC TEST PROCEDURES

A. Fire Temperatures and Pressures

1. Variables - Air flow rate, box size, vent capacity, (i.e. filter
size), fuel quantity.
2. Test Procedure - Establish ventilation rate, vent capacity,
and box size. Ignite cellulose, flammable liquid, and metals
fires.
3. Observations - Note influence of ventilation, vent capacity,
box size, and fuel quantity on box pressure, box temperatures,
temperature on upstream face of filters, pressure drop across
exhaust filter, duration of fire.

B. Evaluation of Detectors

1. Variables - Detector type and principle of operation, detector
locations, enclosure size, fire type, fire size, fire locations,
ventilation rate.
2. Detectors mounted at various locations (especially ceiling).
Detectors connected to individual timers (second counters).
Fire permitted to continue until all detectors respond, or
until fuel consumed. Response time noted as affected by dif-
ferent variables, such as fire type, fire location, and
ventilation.

C. Evaluation of Extinguishing Agents

1. Variables - Agent, agent discharge rate, fire type, fire
location, fire size, enclosure size, location of agent discharge,
duration of free-burning before agent discharge.
2. Observations - Time of extinguishment; quantity of agent
consumed; filter clogging (change in air flow and pressure
drop); change in glovebox pressure; glovebox temperatures;
residual effects.

D. Evaluation of Explosion Suppression

1. Variables - Suppressor location, number of suppressor units; type of fuel (dust, flammable vapor, flammable gas); concentration of fuel by volume in glovebox atmosphere; ventilation rate; enclosure size.
2. Explosion suppression - Establish homogenous explosive mix in enclosure; test various concentrations including that with highest flame speed and highest explosion pressures.

Record time and pressure readings during tests for varying number of suppressor units.

Test with ventilation openings blanked off and no air flow, as well as with commonly used ventilation rate(s).

Note effects on filters, gloves, and windows; and effects with filter openings and glove ports blanked off.

3. Fire Suppression - Test various fuels and fuel quantities, with varying number of suppressor units. Set heat detector at 125°F. Note any effects of varying ventilation rate.

E. Glove Combustibility and Pressure Resistance

1. Variables - Glove material; glove thickness; construction.
2. Fire Test - Establish glovebox size, ventilation rate, and glovebox atmosphere pressure.

Attach gloves in conventional manner, fully extending into glovebox.

Locate cellulosic and flammable liquid fuels directly under glove and at alternate exposing location.

Measure glove ignition time: elapsed time from ignition of fuel to ignition of glove (observed visually) or appearance of hole.

Gloves pressurized with inert gas (e.g. nitrogen, 2 in. H₂O); provide pressure switch to indicate pressure drop upon glove failure.

3. Radiant Heat Exposure - Support sample of glove material (e.g. 6 in. square) vertically.

Expose to gas-fired radiant heat panel (e.g. 12 in. x 18 in.) supported vertically parallel to glove material sample at fixed distance (e.g. 6 in.).

Control radiant panel temperature as measured by a 20-ga. thermocouple located at panel face (e.g. $1500^{\circ}\text{F} \pm 10^{\circ}\text{F}$).

Measure amount of heat received at center of sample (e.g. $1.2 \text{ cal/cm}^2/\text{sec}$).

Measure failure time i.e. from initiation of exposure to appearance of hole in glove.

Measure time to beginning of glove decomposition, i.e. elapsed time from initiation of exposure to appearance of smoke or fumes.

Measure time to ignition.

F. Glove Pressure Resistance

Attach sample glove firmly to conventional port in vertically oriented panel, with port cover in place. Air supplied to glove from fixed pressure source through fixed valve. Pressure at glove continuously indicated on manometer separately connected to glove port. Note bursting pressure.

G. Window Materials - Heat Resistance

1. Test sample size - to be slightly larger than radiant panel to avoid edge heating (e.g.)

2. Test Apparatus

Commercial radiant heat panel, 12 in. x 18 in. vertically mounted. Combustion of gas-air mixture adjusted to produce glow without visible flame.

Temperature adjusted to $1500^{\circ}\text{F} \pm 40^{\circ}\text{F}$ as recorded by thermocouple on face of panel (e.g. 1 in. up and 1/2 in. in from lower corner).

Samples mounted on dolly to permit rolling into position before radiant panel. Sample to be parallel to radiant panel, 2 in. distant.

All plastics and some glass samples fitted with rigid frame to prevent deflection away from heat panel when heated.

3. Test Procedure - Sample mounted on dolly, shielded from heat source by hand-held sheet of asbestos-cement board, while being rolled into test position.

Test is initiated when asbestos-cement board pulled away rapidly, exposing sample to radiant heat source.

Test terminated normally by moving sample away from heat source.

4. Observations - Record temperature at face of sample during test, if possible. For glass products, note time of cracking, and falling away or shattering. For laminated safety glass, note time to smoking and burning of plastic layer.

For plastics, note time to smoking, burning, appearance of hole, sagging, etc. Note relative quantities of smoke emitted.

H. Window Materials - Fire Resistance

1. Test Apparatus

Simplified Test Enclosure - rigid metal frame with bottom, sides, upper half of rear, and forward half of top consisting of hard asbestos-cement board. Lower half of rear and rear portion of top left open for natural draft and access.

Front to consist entirely of window sample (e.g. 35 in. x 35 in.) clamped to frame, forming sloping face inclined at 18°.

(Alternately, standard glovebox, as designed for repeated tests on fire behavior, gloves and agents, with standardization ventilation rate. Windows may be vertically mounted or sloped, conforming to actual gloveboxes).

Chrome-alumel thermocouples placed at fixed locations (e.g. through top panel and inside enclosure close to sample center).

2. Test Procedure - Plastic sample to be clamped tightly into position, (e.g. welded angle frame, 1 x 1 x 1/8 in. against unexposed face, and three 2-in. C-clamps on each side, total 12). Glass samples similarly clamped, with gasketing at both faces (e.g. 3/8-in. silicone rubber, 50 durometer).

Exposure to be 1-sq.-ft. pan of clean-burning flammable liquid (e.g. ethyl alcohol, acetone) placed inside enclosure on floor, centered to allow flame impingement on window.

Fuel ignited. Temperature and time recording started at ignition.

3. Observations - For glass, note time to cracking, falling away or shattering. For laminated glass, note time to cracking, separation of layers, smoking and burning of plastic layer on the outside, and falling away of glass. For plastics, note time to smoking, burning, and burnthrough, pulling out of the frame, shattering.

APPENDIX V

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