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FIRE AND EXPLOSION TESTS OF PLUTONIUM GLOVEBOXES

by

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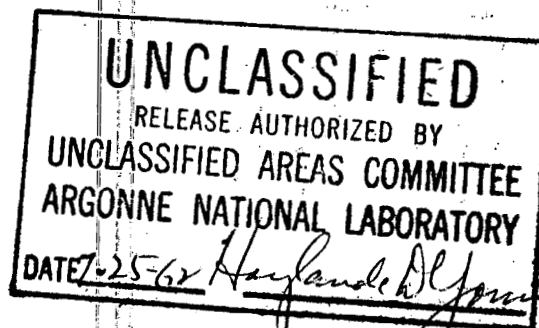
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## FIRE AND EXPLOSION TESTS OF PLUTONIUM GLOVEBOXES

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To test the fire and explosion resistance of new plutonium metallurgy gloveboxes and to obtain information pertinent to fire control, fire and explosion tests were conducted in one of the gloveboxes. It was found that over 10% oxygen is required for non-metal, and that over 5% oxygen is required for freely burning metal fires. However, metal chips will burn with as little as 1% oxygen if additional heat is furnished. Standard dry chemical, Met-L-X and carbon dioxide extinguishers were excellent for non-metal fires. An eutectic salt mixture was excellent for metal fires.

The gloveboxes designed for plutonium research in the new Fuels Technology Center at Argonne National Laboratory have features that make them more adaptable to varying research conditions and at the same time give better fire resistance.<sup>(1)</sup> They are constructed of a heavy, extruded aluminum framework and have removeable one-inch thick aluminum baseplates and one-half-inch thick aluminum side, back and top panels. Windows are three-eighth-inch laminated safety-glass.

Normally these gloveboxes will be used with an atmosphere of nitrogen containing less than 0.03 percent oxygen. In case, however, the nitrogen atmosphere were lost or a large leak should occur, a fire could develop from the spontaneous ignition of plutonium chips or pyrophoric plutonium alloys.

To test the fire resistance of the new glovebox design and obtain information pertinent to fire control, seventeen fire tests and two explosions were conducted in one of the gloveboxes.

The purposes of the tests can be outlined as follows:

- (1) To determine the effect on the glovebox of fires and explosions.
- (2) To find the lowest oxygen concentration that will support combustion.
- (3) To test the effectiveness of standard fire extinguishers and a fused salt mixture for extinguishing glovebox fires.

The glovebox used for the test had a one-inch thick aluminum baseplate, one-half-inch thick aluminum panel at one end, sloping windows in front and back and a glass panel on top and at one end, with an enclosed volume of 17.7 cubic feet.

Thermoswitch indicators were mounted outside of the top window panel and a chromel-alumel thermocouple was placed in a small hole drilled into the underside of the baseplate.

The fire tests were conducted within a metal dust and fume chamber 10' by 10' by 8' inside dimensions. They were conducted at various oxygen concentrations of 1 to 20 percent in dry nitrogen. Motion pictures were taken through a window in the chamber.

#### ALCOHOL FIRES

Because ethyl alcohol is used to a considerable extent in some of our gloveboxes, it was used to simulate a non-metal fire in the glovebox. Nine tests were conducted using 50 ml batches of alcohol under varying conditions.

The alcohol was poured into a 7" by 12" by 1-1/2" enamel tray and ignited with a Tesla spark coil. Under some conditions the fires smothered themselves in a few seconds. The remaining fires were allowed to burn for a few minutes then extinguished with a fire extinguisher mounted on the end panel. The nozzle of the extinguisher was sealed through a flexible rubber diaphragm and could be aimed over a wide area of the glovebox. Three types of extinguishers were used; dry chemical (sodium bicarbonate), Met-L-X (sodium chloride) and carbon dioxide. Contrary to what one might expect (2), the carbon dioxide extinguisher did not pressurize the glovebox more than a few inches of water as the fire was put out with just two or three short bursts of the extinguisher.

The dry chemical also put out the fire with just a few short bursts, and hardly changed the pressure in the glovebox. The Met-L-X extinguisher uses a coarser material and is normally used only on metal fires. To see if it could be used in an emergency if no other type were available, it was tried on the alcohol fire. Surprisingly, it also extinguished the fire with a few short bursts and hardly changed the pressure in the gloveboxes.

To find the lowest oxygen concentration that would support combustion in case a leak should develop in a glovebox, the glovebox was purged with cylinder nitrogen until the desired oxygen level was reached, then held in a static condition. Oxygen concentrations were measured with a MSA Oxygen Analyzer. Table I summarizes the results of these tests.

Table I does not list all the tests conducted but gives representative results for the different conditions tested. Several things can be noted from the table.

- (1) Over 10 percent oxygen is needed for the combustion of alcohol in the glovebox.
- (2) When there is no pressure relief, as in the case when the exhaust filter is closed, the pressure build-up will very quickly burst the gloves.
- (3) When there is pressure relief, but essentially a static atmosphere in the glovebox, the fire quickly smothers itself without bursting the gloves.

In the tests none of the windows cracked even though flames burned against the top window for short periods. By contrast, a plastic window was ignited by a small amount of burning plutonium in a glovebox fire at another installation.(3). The fire spread to other plastic windows and did considerable damage.

Table I. Alcohol Fires

Atmosphere	Glovebox Condition	Results
Nitrogen- 10% Oxygen	Closed glovebox; static atmosphere.	Unable to ignite alcohol.
Nitrogen- 12.5% Oxygen	Closed glovebox; static atmosphere.	Ignited with difficulty; burned 4 or 5 seconds and went out.
Nitrogen 15% Oxygen	Closed glovebox; exhaust filter open.	Burned readily.
Air	Closed glovebox; exhaust filter closed.	Burned with flames against top window. Gloves swelled to 33" diameter and burst in 30 seconds.
Air	Open glovebox; (gloves removed).	Burned vigorously. Flames confined to interior of glovebox.
Air	Closed glovebox; exhaust filter open.	Fire self-extinguished in 45 seconds, leaving 10 ml of alcohol unburned.
Air	Closed glovebox; exhaust filter 20% closed.	Fire self-extinguished in 57 seconds; pressure built up to 14" water column; 18 ml of alcohol unburned.

## METAL FIRES

To simulate a plutonium metal fire 50 gram batches of magnesium chips were used. Magnesium has a heat of combustion of 5980 cal/g compared to only 1058 cal/g for plutonium (4); thus in heat produced, 50 grams of magnesium is roughly equivalent to the amount of plutonium normally permitted in one of our gloveboxes at any one time.

A spirally wound nichrome heating element covered by a small ceramic tube was installed in a 4" diameter aluminum pan. The pan was set on a 1/8" aluminum plate placed on the glovebox floor. The chips were poured on top of the heater and heated to the ignition point. Table II summarizes the results of these tests.

The significant things to note in Table II are:

- (1) The chips will burn in as little as 1 percent oxygen if additional heat is furnished, but they will not burn freely until over 5 percent oxygen is supplied.
- (2) The difference in burning characteristics between the last two tests clearly indicates that the heavy aluminum baseplate conducts away enough heat to greatly slow down the burning rate.

In the latter three of the above tests, the fires were allowed to burn for nearly ten minutes, then extinguished by pouring over the fire a mixture of finely ground fused salts (35<sup>w/o</sup> NaCl, 40<sup>w/o</sup> KCl, 25<sup>w/o</sup> BaCl<sub>2</sub>). (5) The fire was extinguished immediately, and later examination of the caked material showed that the salt mixture had melted and formed a solid crust around the burning material.

Table II. Metal Fires

Atmosphere	Glovebox Condition	Results
Nitrogen- 1% Oxygen	Closed glovebox; static atmosphere.	Burned very slowly; went out when heater turned off.
Nitrogen- 3% Oxygen	Closed glovebox; static atmosphere.	Burned slowly with slight amount of smoke; went out when heater turned off.
Nitrogen- 5% Oxygen	Closed glovebox; static atmosphere.	Burned slowly with slight flame; went out when heater turned off.
Nitrogen- 10% Oxygen	Closed glovebox; static atmosphere.	Burned freely with slight flame; box filled with smoke; continued to burn when heater turned off.
Air	Gloves removed; 100 gms of chips directly on baseplate.	Burned freely but not vigorously.
Air	Gloves removed; 100 gm of chips in enamel tray insulated from glovebox floor with 1/2" of sand.	Burned violently with brilliant flame.

Other salt mixtures used in similar tests (2) allowed the burning metal to smoulder for 20 to 30 minutes.

The maximum temperature reached by the baseplate in these tests was 152°F.

#### ALCOHOL AND METAL FIRES

Up to this point none of the windows had cracked, although blisters had begun to appear in the plastic laminate of the safety glass. Combination alcohol and metal fires were then tried, using larger amounts of material. Table III summarizes these tests.

Table III. Alcohol and Metal Fires

Atmosphere	Glovebox Condition	Results
Air	Gloves removed; 100 gm Mg and 500 ml alcohol directly on baseplate.	Alcohol burned freely and ignited Mg chips; burned for 10 minutes; all windows cracked.
Air	Gloves removed; 300 gm Mg chips and 1000 ml alcohol in enamel tray insulated from floor.	Burned very vigorously; windows further cracked but still intact. Mg continued burning after all alcohol was burned. Fire left to burn itself out.

The significant thing about these two tests is that the windows remained intact, even though badly cracked and blistered.

Three more alcohol fires with the glovebox closed and exhaust filter open were conducted after these two tests, and the windows remained intact for two of them. On the third, the exhaust filter was 80 percent closed, and the pressure buildup blew out one of the cracked windows. Figures 1 and 2 show the appearance of the glovebox after the fire tests. The blistering of the plastic laminate can be seen in the glass end panel of Figure 2. The black streaks are melted binder from the felt used in the window clamps.

After the fire tests the glovebox was disassembled and examined. Aside from the broken windows and a slight set taken by the gaskets, there was no damage to the glovebox.

Although the fire tests were conducted under static conditions, we feel that this closely simulates conditions in our plutonium gloveboxes as the normal flowrate in our nitrogen atmosphere boxes is only 0.2 CFM per 100 cubic foot glovebox. To overcome the possibility of pressurizing the gloveboxes in case of a fire, or for any other reason, we have added a pressure relief vent to all our glovebox lines.

### EXPLOSION TESTS

In our previous glovebox work we have had several minor explosions in the gloveboxes, even though they contained high purity atmospheres of helium or nitrogen. Most of these have been due to mixtures of nitric acid and organic materials such as alcohol or acetone. In none of these instances were the gloveboxes ruptured, although the interiors were shambles. To see what we might expect in case of a more violent explosion, two explosion tests were conducted.

The glovebox was reassembled with new windows and moved outdoors to an open field where the tests were photographed with both standard speed and high speed motion picture cameras using color film.

A Tesla coil mounted near the front window so that it would arc to the glovebox frame was used to ignite a 10% hydrogen and air atmosphere. The explosion was triggered by a timing circuit used to start the cameras.

In the first test the gloves were not covered. One glove was blown off intact; the other three were completely shattered. Pieces were found 35 feet away. Figure 3 shows three frames taken from the high speed film and reveals that the gloves swelled to about 35 inches in diameter before bursting. None of the windows were cracked.

In the second test the gloves were covered with steel glove port covers. The explosion shattered the front window but did not even crack the others. From Figure 4 it can be seen that the window first bulged out, then lifted away from the frame in a mass of splinters. A two to three-inch border of glass remained in the window frame.

Had the glovebox contained plutonium, contamination would have been widely scattered in both explosions. It was quite obvious, however, that the explosion without the glove ports covered did much less damage to the glovebox and would have been less injurious to anyone near it. The gloves were in effect acting as explosion vents.

Except for the broken window and shattered gloves, no damage was done to the glovebox.



## CONCLUSIONS

The tests showed that:

- (1) Our new gloveboxes are quite rugged. For small metal and alcohol fires, the fires could be safely left to burn out if necessary, providing the gloves are not involved. In case of an explosion, less damage is done if glove port covers are not used.
- (2) Over 10 percent oxygen is required for an alcohol fire in the glovebox. The metal chips will burn in 1 percent oxygen if some additional heat is supplied.
- (3) The dry chemical, Met-L-X and CO<sub>2</sub> extinguishers are all excellent for alcohol fires in the glovebox. The fused salt mixture is excellent for metal fires.

The safety glass has good fire resistance for the type of fires that we might expect in our gloveboxes. Although we have not demonstrated it, the safety glass would undoubtedly fall out after being badly cracked in an extremely hot fire, or prolonged fire, because the plastic laminate would soften and melt. However, it would add very little fuel to the fire.

As for explosion resistance, the safety glass is probably worse than a plastic window because of the many glass splinters formed. A solution to this problem might be to install a high impact-resistant plastic window over the outside of the safety glass window and have it held loosely at the edges so that it would come off in one piece and act as a shield.

## REFERENCES

1. L. R. Kelman, et al, "Gloveboxes for Plutonium Metallurgy Research at Argonne," Proceedings of the Ninth Conference on Hot Laboratories and Equipment, November 7-9, 1961, p. 64.
2. R. R. King, "The Prevention and Control of Fires in Gloveboxes Containing Plutonium," Proceedings of the Ninth Conference on Hot Laboratories and Equipment, November 7-9, 1961, p. 71.
3. Serious Accidents Bulletin, Number 130, November 27, 1957, USAEC.
4. ASM Metals Handbook, 8th Edition, 1961, pp. 1213 and 1218
5. L. H. Cope, The Extinguishing of Uranium and Plutonium Fires, UKAEA, DEGR-29D, November, 1959.

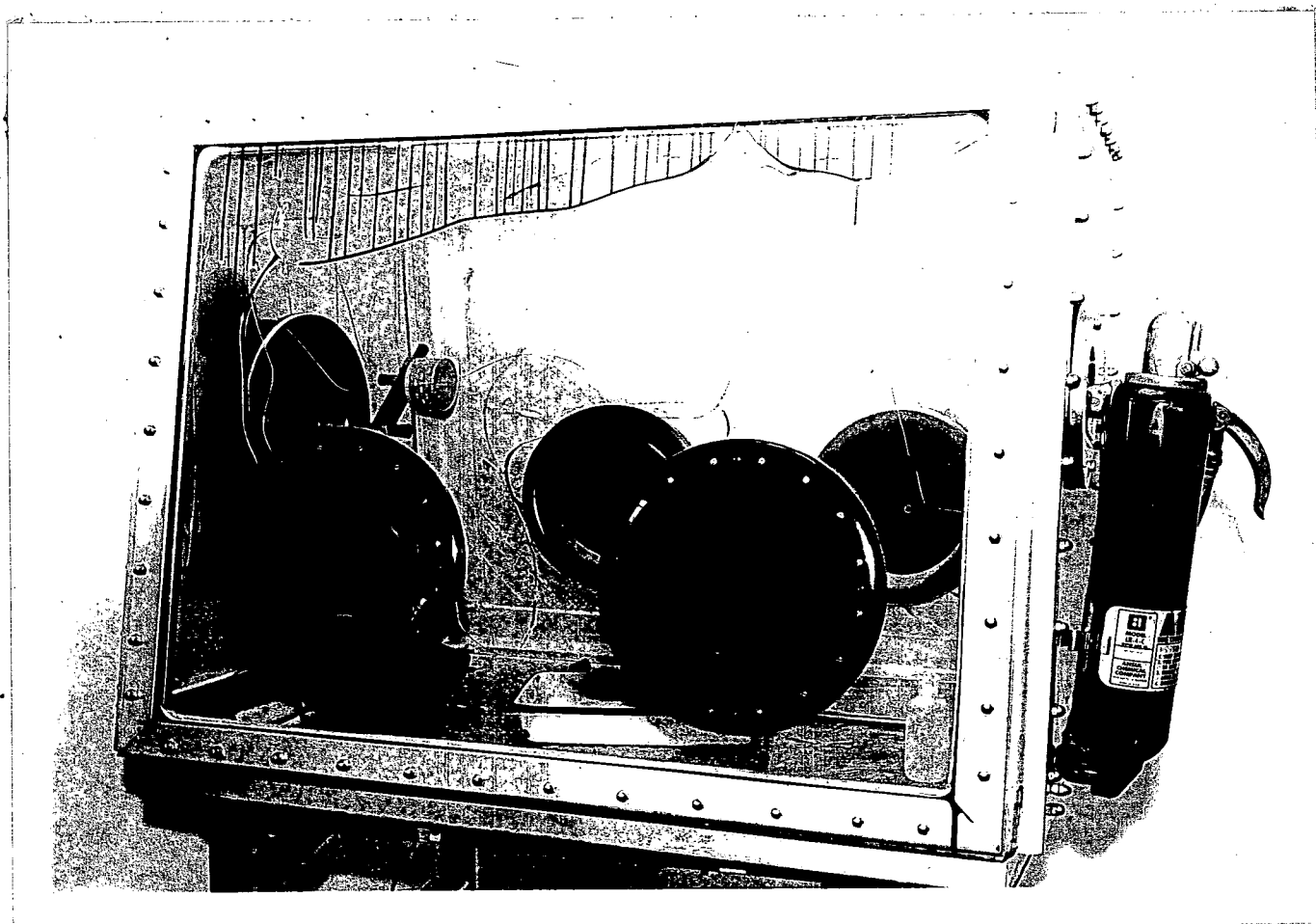


Figure 1. Front View of Glovebox After 17 Consecutive Fire Tests.



Figure 2. Rear View of Glovebox after 17 Consecutive Fire Tests.

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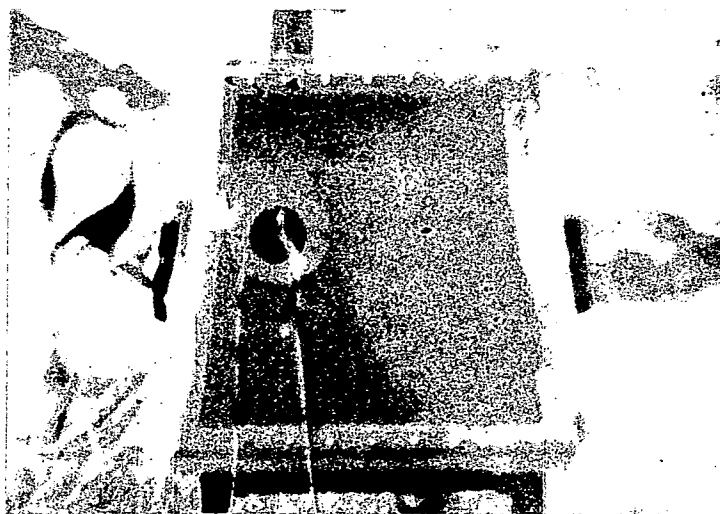
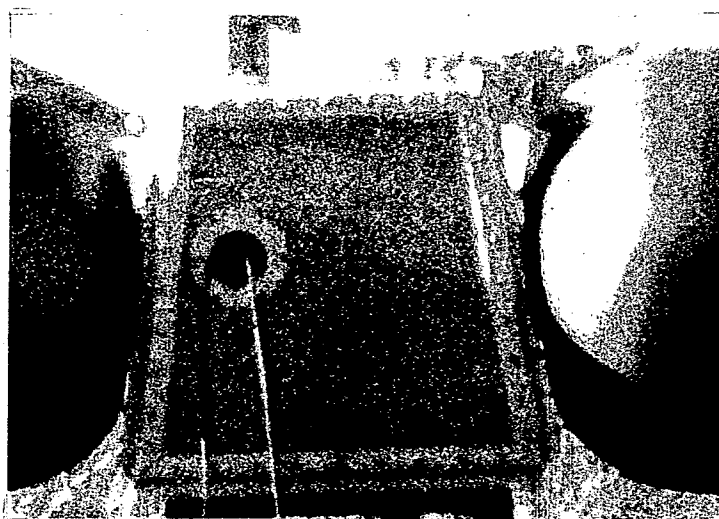
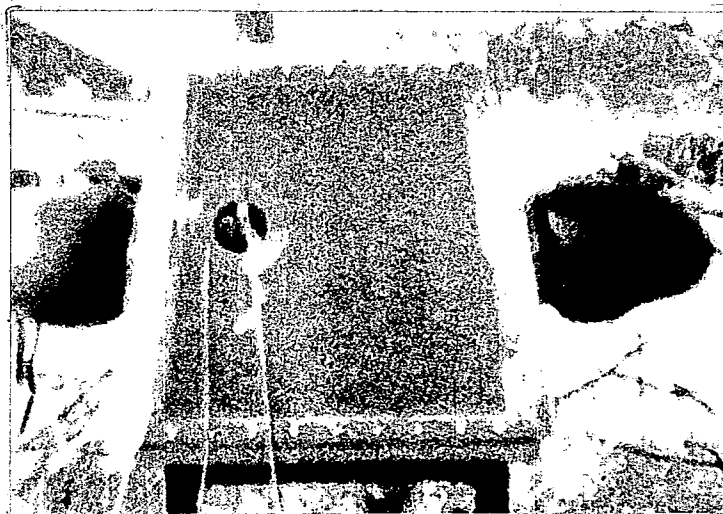


Figure 3. Frames from High Speed Movie Film  
Showing Glovebox Explosion When  
Glove Port Covers are Not Used.

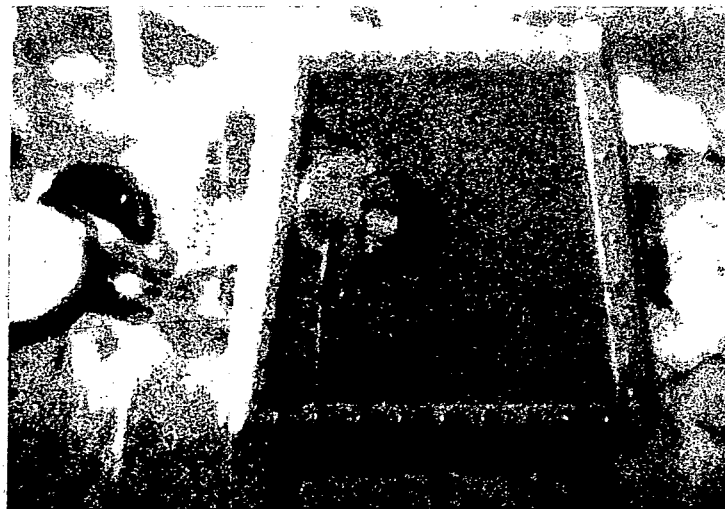
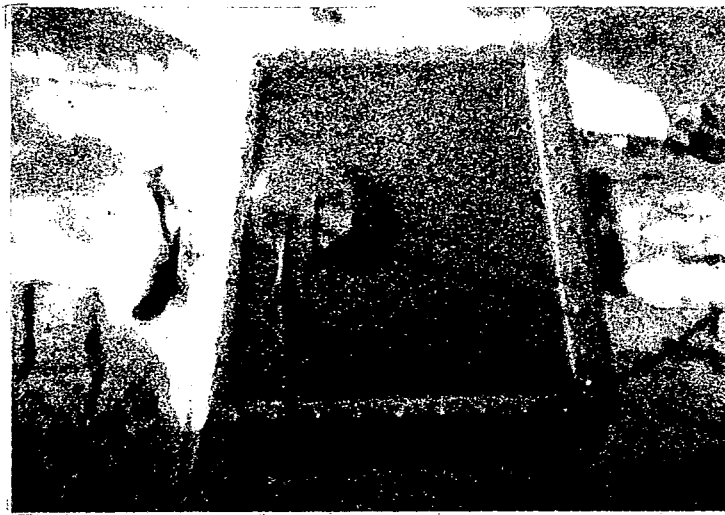
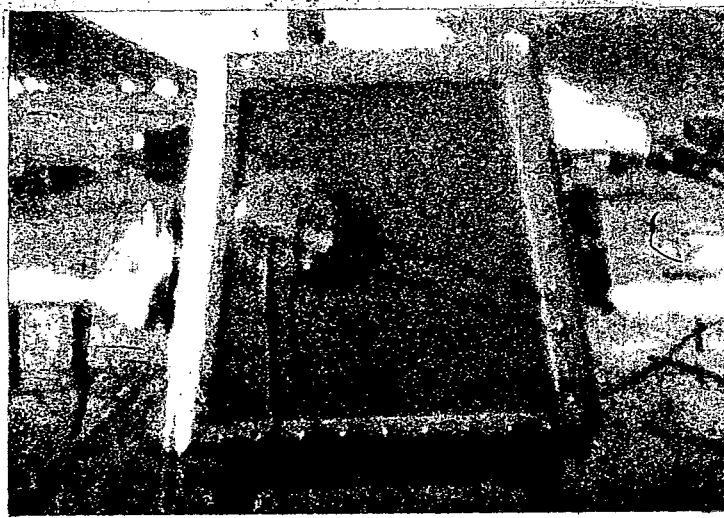


Figure 4. Frames from High Speed Movie Film  
Showing Glovebox Explosion when  
Glove Port Covers are Used.