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Berkeley, California

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Jensen Young and Will D. Phillips

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ABSTRACT

A number of tests were carried out in a gloved box with ether-nitrogen and ether-air mixtures to evaluate their explosion potential. Spark plugs were used in the box as the source of ignition while the air flow, ether volume, and hot-plate temperature were all varied. We found that

(a) nitrogen-ether concentrations were safe under our test conditions,

(b) rapid vaporization of ether in air produced local explosive concentrations that were unpredictable,

(c) explosions occur if hot-plate temperatures approach 180°C,

(d) slow evaporation of ether at low air flow was safe,

(e) the lower explosive limit of ether-air mixtures in a gloved box of 12 ft^3 was about 3% by volume,

(f) the gloved box withstood many vigorous explosions; the explosive force was vented through the gloved ports and out the gasket seal around the window.

I. INTRODUCTION

An explosion in a gloved box at another site increased concern at the Lawrence Radiation Laboratory that a similar accident could occur here.[1] The Health Chemistry Department was asked by a researcher to undertake design and fabrication of a safe enclosure to handle evaporation of ether from a plutonium solvent-extraction process. The following explosion tests were an outgrowth of this request.

The purpose of the explosion tests were threefold:

(a) To insure that an inert-atmosphere gloved box was safe for the evaporation of ether.

(b) To find the volume of ether that could be evaporated safely in a standard gloved box.

(c) To test the gloved box under explosive conditions.

II. DESCRIPTION OF TEST APPARATUS

A. Gloved box

A standard full-recess box with centrifuge well and cupola top was used. Special care was taken to make this box reasonably airtight. (See Fig. 1.)

B. Solvent-evaporating apparatus

A hot plate controlled by a Variac outside the box was used for the heat source. A modified separatory funnel contained ether; when the remote valve was operated, the ether poured down a tube to a crystalizing dish or metal pan placed on top of the hot plate. The hot-plate temperature was preset to rapidly evaporate the ether.

C. Sparking device

Four sets of spark plugs, activated by automotive ignition coils, were positioned within the box. One plug was positioned on the floor of the box beside the hot plate, another in between the front gloved ports on the front panel, another on the left side of the back panel, and the last one about 1 inch below the air exhaust outlet. The plugs fired simultaneously during each test setup.

D. Ventilation

1. For the inert-atmosphere portion of the test, cylinder nitrogen was discharged into the box at a preset flow rate.

2. For the "standard condition" tests, room air entered the box through a filter at a preset flow rate.

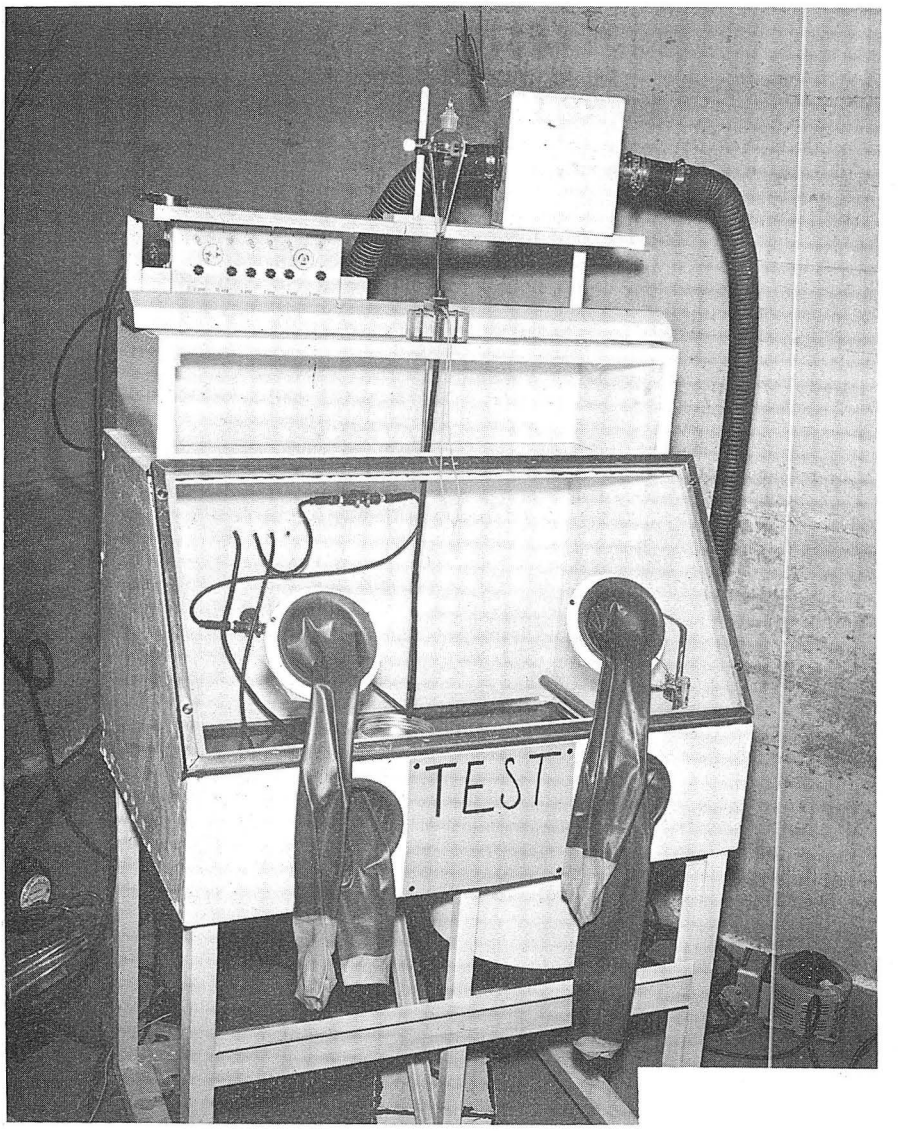

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Fig. 1. Gloved box before tests.

The atmosphere from the box passed through a high-efficiency filter and Venturi air meter, and was diluted with air and discharged through a blower.

III. CONDUCT OF TEST

We ran three series of explosion tests in which the box atmosphere was varied. These tests are described below. We wanted to get maximum use of one gloved box, so we scheduled the safest conditions first.

A. Ether-inert-gas mixtures

To prove the safety of an inert atmosphere, air in the box was replaced with nitrogen in test 1. When the oxygen content had been reduced to approximately 1% (Beckman Oxygen Analyzer, Model D-2), the nitrogen flow was reduced to 1 ft^3/min. The spark plugs were fired at 10-second intervals while 40 ml ether was evaporated. On the basis of published data, approximately 40 ml ether, if it was vaporized and homogeneously mixed throughout the volume of the box, should have formed the LEL (lower explosive limit, 1.9% by volume in air).[2] We purposely sparked the plugs during the evaporation step to catch any localized concentration that might form the LEL. Since no explosions occurred on this or a repeat test, we verified that an inert atmosphere was safe for ether evaporations.

B. Ether-air mixtures with low air flows

We undertook this series to test the problems that might arise from the buildup of local concentrations of ether vapors. The air flow, ether volume, and hot-plate temperature were varied. Test results are summarized in Table I. Ether concentration buildup inside the box was measured by an explosimeter (Johnson-Williams Alarm, Model RH, Mountain View, California).

In test 1, 100 ml of unheated ether was placed in an open container with the air flow set at 10 cfm. The plugs fired repeatedly at intervals of 10 seconds for the duration of the run. The maximum explosimeter reading of 13% of LEL was reached about 2 minutes after the start of the run; then it slowly dropped.

The explosimeter readings became unreliable for the remaining tests. The meter indicated safe conditions just prior to an explosion, or it indicated explosive conditions when no explosion could be initiated. Two possible explanations for this behavior are as follows:

(a) The lag time for the ether vapor to penetrate into the sensing element was too long.

(b) The presence of ether-concentration gradients within the box gave erroneous readings.

For tests 2 through 17, the plugs were set to fire repeatedly at intervals of 10 seconds before, during, and after the evaporation of ether,

Table I. Test for explosive air-ether mixtures under various conditions.

Test	Air flow (cfm)	Ether volume (ml)	Sparking rate	Hot plate temp. (°C)	Other conditions or comments	Result
1	10	100		Room temp.	Ether not heated, but evaporated from open vessel	None
2	10	10		160-170	Spacer on hot plate to reduce evaporation rate	None
2a [a]	10	10		140-150		None
2b	10	10		140-150		None
3	10	20		160-170		Fire
3a	10	20		160-170	Deactivate #1 plug	Explosion and fire
3b	10	20		140-150	Deactivate #1 plug; test of autoignition temp. of hot plate	None
3c	10	20		140-150		Explosion and fire
3d [a]	10	20	No spark 'til all ether has dropped onto vessel.	140-150	Test of autoignition temp. of hot plate	None
3e [a]	10	20		140-150		None
3f	10	20		140-150	New set of plugs for this and subsequent tests	Explosion and fire
3g	10	20		140-150	Ether release malfunction; only 9 ml ether used	Explosion and fire
4	10	30		160-170	Deactivate #1 plug	None
4a	10	30		140-150		None
5	10	40		160-170	Deactivate #1 plug	None
6	10	15		140-150		None
7	5	10		140-150		None
8	5	20		140-150		Explosion and fire
9	5	30		140-150		Explosion and fire
9a	5	30	No spark 'til all ether has dropped onto vessel.	140-150	Test of autoignition temp. of hot plate; some liquid still unvaporized	Explosion and fire on 1st or 2nd spark
10	5	60		140-150	Some liquid splashed over vessel onto hot plate	Spontaneous ignition
11	5	40		140-150	Some liquid still unvaporized	Explosion and fire on 1st or 2nd spark
12	5	10		96	Slow evaporation	None
13	5	20		96	20 ml/1.25 minutes	None
14	5	30		96	30 ml/2 minutes	None
15	5	40		96	40 ml/3.5 minutes	None
16	5	50		96	50 ml/5.2 minutes	None
17	5	100		96	100 ml/8.9 minutes	None

a. Possible #1 spark plug failure (#1 spark plug close to hot plate, floor of box).

except as noted in the table. During a given run, the surface temperature of the hot plate may have increased beyond the preset temperature. This would account for the autoignition explosions noted (tests 3, 3a, 10); several tests were designed to verify this (tests 3b, 3d, 9a).

The spark plug located near the base of the hot plate was the principal source of ignition. When this plug was bypassed (test 3b versus 3c) no explosions occurred under similar explosive conditions. This can be explained in that ether vapor has a density 1.8 times that of air; during rapid vaporization, the vapors would form a concentration gradient towards the floor of the box. When this reached the LEL near the spark plug, an explosion would occur.

Tests 3g and 4a were an anomaly. In test 3g, only 9 ml ether produced explosive conditions, whereas in 4a, 30 ml ether didn't. We offer no explanation other than that the results were unexpected.

In tests 12 through 17, the evaporation rate of ether was reduced to 10 to 16 ml per minute. No explosions occurred in these tests. Obviously, the ether vapors were diluted below the LEL as they were formed.

C. Ether-air mixtures under static conditions

We realize that a gloved box is not a good test apparatus for the determination of the LEL.[3] However, we felt it worthwhile for future reference to determine the LEL of ether under our conditions.

For this test series, the box was tightly sealed to eliminate leaks, the centrifuge well and gloved ports were sealed off to exclude their volumes, and the box was left in a static, or no-air-flow, condition. Ether was slowly and completely evaporated (hot plate temperature 96°C), and the atmosphere was mixed with a propeller for 8 minutes before a spark was started. Table II shows the results of these tests.

Table II. Test for LEL of air-ether mixtures.

Test	Ether volume (ml)	Result
#1	40	No explosion
#2	45	No explosion
#3	50	About 20-second delay before explosion
#4	50	Explosion
#5	55	Explosion
#6	60	Vigorous explosion

From the above results, we found that 50 ml ether had to be evaporated into a gloved box of 12 ft^3 before the LEL was reached. This LEL was calculated to be about 3% by volume.

IV. POST MORTEM ON GLOVED BOX

As a whole, this gloved box withstood the explosive tests well. Most of the explosive force vented itself through the gloved ports (gloves were ripped off the ports or torn to shreds) and through the gasket seal around the front window. During the early tests, the window was secured to the box with wood screws which penetrated the front of the window and the sides of the box. The window was not fastened tightly in place in order to save the box; consequently, each explosion lifted off the window. In the later tests, the window was secured by angle brackets to the sides of the box. This anchoring method retained the window on the box throughout the rest of the explosions. In the final static test, the front window bowed outwards and the glove cover and right door panel were blown off. The rest of the box loosened up a bit, but retained its integrity (see Fig. 2). During a static test for a movie sequence, another gloved box was completely disintegrated.

V. CONCLUSIONS

If ether must be used in a gloved box, we recommend that the following steps be taken to insure safe conditions:

(a) Don't 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 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 is 180°C).[4]

(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 face of the researcher.

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.

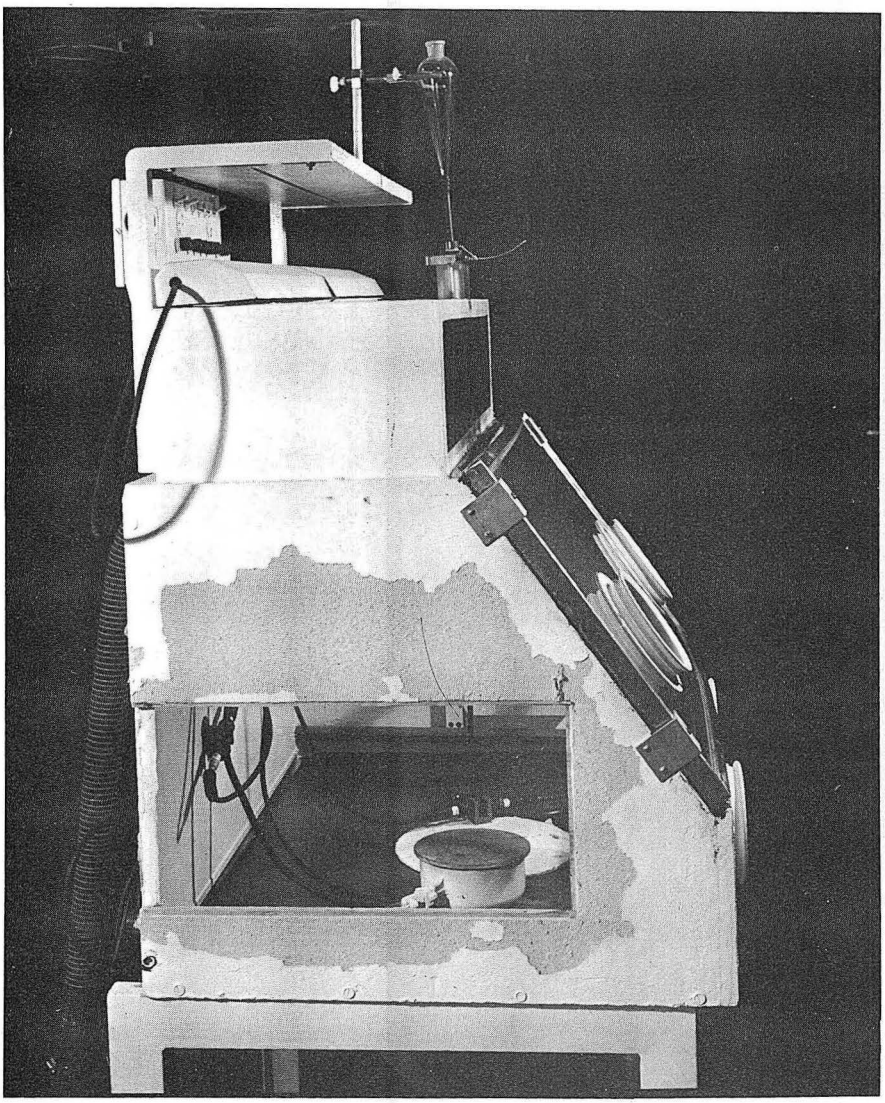

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Fig. 2. Gloved box after tests.

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