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REACTION CHARACTERISTICS OF SPILLS OF FLUORINE AND FLUORINE-OXYGEN MIXTURES UPON VARIOUS MATERIALS

by Louis M. Russell, Harold W. Schmidt, and Robert F. Clarke

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Cleveland, Ohio

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SUMMARY

Small quantities (5 to 10 lb) of liquid fluorine, liquid oxygen, and liquid 30-percent FLOX (fluorine-oxygen mixture containing 30 percent fluorine by weight) were spilled upon various common materials which might be found or placed around a rocket test or launch facility for the purpose of investigating hypergolicity and other reaction characteristics. When fluorine or 30-percent FLOX is spilled upon many of these common materials, sufficient hypergolic reaction can occur to create a combustive "hot spill." The hot spill creates a rapid, high cloud rise, which provides efficient diffusion and dispersion of the toxic products and tends to reduce the potential downwind ground-level pollution. The reactions varied from smooth-burning reactions to strong detonations; the variation depended upon whether FLOX or fluorine was spilled and upon which materials they were spilled. Spills of liquid fluorine or FLOX upon materials with which they do not react combustively resulted in typical cryogenic "cold spills" in which the toxic vapors drifted downwind close to the ground and thus created high pollutant concentrations for a considerable distance downwind.

The desirable characteristics of the FLOX-charcoal reaction (smooth burning, high heat release, and diluent effect of the nontoxic reaction products) indicate that the placement of charcoal in areas of possible fluorine or FLOX spills could provide effective spill and pollution control.

INTRODUCTION

The use of liquid fluorine or liquid FLOX (fluorine-oxygen mixture) as an oxidizer in a rocket launch or test facility requires a knowledge of the reaction characteristics of these liquids in the event of spillage. The hypergolicity of cryogenic liquid fluorine or FLOX when spilled in an unconfined manner onto various common materials was of particular interest. In addition, the relative hypergolicity of FLOX with respect to fluorine was uncertain under this spill condition. Fluorine, FLOX, and their most common byproduct, hydrogen fluoride, are all toxic; it is desirable that these gases be inerted or made to rise upward for better dispersion and diffusion into the atmosphere for the safety of personnel and communities downwind of the spill area. An investigation was therefore made to determine the results of such spillages onto various materials. Small quantities (5 to 10 lb) of liquid (FLOX, fluorine, and oxygen) were spilled from a height of about 20 inches upon several common materials which might be found or placed around a test or launch facility (tables I to IV, Test material column). Because of recent NASA interest in 30-percent FLOX (fluorine-oxygen mixture containing 30 percent fluorine by weight), the majority of spills were made at this concentration. From these spill tests, information was obtained in the following areas:

(1) The hypergolicity of FLOX and fluorine with various materials upon which they might be spilled, including the effect of oxygen as a diluent on hypergolicity

(2) Qualitative information on reaction characteristics and combustion propagation by visual observation and high-speed photography

(3) Visual and photographic information on the path of the resultant gas clouds (shown in a motion picture[1])

(4) The feasibility of using charcoal as a reactant for fluorine spills as an effective means of pollution control

(5) Basic information for use in defining and planning a succeeding program (large-scale, 30-percent FLOX spills by General Dynamics Corp., Convair Div., under Contract NAS 3-3245, Task No. 2)

TEST APPARATUS

The spill-test apparatus (fig. 1(a)) consisted of a $3\frac{1}{2}$-gallon liquid-nitrogen-jacketed stainless-steel tank, a remotely controlled 4-inch globe valve, and a 4-inch heavy-walled spill pipe extending through an aluminum shield which protected the spill apparatus from reaction effects. The large-sized spill pipe was selected to empty the spill tank contents as a "slug" at a low velocity equivalent to that produced by a gravity fall. The valve was insulated with asbestos-covered padding. The spill pipe was similarly insulated, cooled by liquid-nitrogen coils to ensure liquid spills instead of gaseous spills, and purged with helium prior to testing to prevent contamination by the atmosphere. Stainless-steel pans, 3 by 3 by 1 foot, were used to contain the test sample material. The test apparatus without the insulation and shield is shown in figure 1(b). A charcoal sample ready for a test is shown in figure 2.

MEASUREMENTS, OBSERVATIONS, AND RECORDING

In the investigation reported herein, the following measurements were

[1]Motion-picture supplement C-243 is available on loan. A request card and a description of the film are included at the back of this report.

taken: (1) the quantity of FLOX (fluorine-oxygen mixture), LOX (liquid oxygen), or fluorine to be spilled, (2) the weather data at the time of the test, that is, the wind velocity at an elevation of 10 feet and the ambient temperature gradient up to an elevation of 30 feet, (3) laboratory analysis of samples of the test material for moisture content (where applicable) and for acid content (where applicable) before and after testing, and (4) the quantity of test material in the pan, to ensure that the test material present was far in excess of stoichiometric quantities, so as to simulate an actual spill, in which unlimited reactant is usually available. The degree or efficiency of reaction was not measured quantitatively because of unavoidable loss of test material by either blast effects, by rapid expansion of the reaction products, or from residual atmosphere-fed fires.

Observations were made of (1) the type of reaction produced (fireball, smooth propagation, explosion, sputtering, or other) and (2) cloud dispersion (angle of rise (from estimated photographs), rate of rise (visually estimated), clinging to the ground, or other). A television screen within the control room, which provided an overall view from a television camera atop an adjacent structure, was used as one method of observation. Other observations were made visually by use of (1) a periscope within the control room 35 yards from the spill apparatus, (2) 7 × 50 binoculars with the observers at a distance of about 140 yards, and (3) direct vision from a distance of about 140 yards.

The results were recorded on color motion-picture film with the following three cameras: (1) a high-speed, closeup-view camera atop an adjacent 40-foot structure for recording spill and reaction, (2) a normal-speed wide-angle camera atop the same structure for recording cloud travel, and (3) a normal-speed wide-angle camera at a right angle to the wind and at ground level for recording cloud travel.

RESULTS AND DISCUSSION

Liquid-Oxygen Checkout Tests

Initial tests were made with LOX both to check out the apparatus and to help determine appropriate spill quantities and pressures. Observation indicated that 10-pound quantities were sufficient to ensure liquid spills and that a spill tank pressure of 5 pounds per square inch gage was sufficient to obtain rapid spills and yet to avoid excessive splashing. All subsequent spill tests were performed at this pressure. As shown in table I, no reaction occurred in any of these checkout runs, including one in which LOX was spilled upon top soil which had been soaked with SAE 20 oil. It is significant that no reaction occurred in this test, since hydrocarbons and liquid oxygen have been known to react explosively under confined conditions at higher pressures. After each spill, the oxygen vapors merely drifted slowly downwind along the ground and then dissipated.

30-Percent FLOX Spill Tests

Ten-pound quantities of 30-percent FLOX liquid were spilled upon various materials (table II). When no reaction occurred, such as the spill upon concrete, the toxic vapors merely drifted downwind close to the ground as is typical of a cryogenic "cold spill." In other cases, such as with FLOX and JP-4, a "hot spill" resulted. This reaction was characterized by several rapid machinegunlike microexplosions, which occurred over a period of 1 to 2 seconds. A bright yellow fireball formed and expanded to a diameter of 25 to 30 feet (fig. 3(a)), and a turbulent black cloud emerged from the fireball. A marked "chimney" effect was produced as the cloud rose rapidly and almost vertically (fig. 3(b)). A residual atmospheric fire on the JP-4 surface was manually extinguished. The multiple microexplosions or loud sputtering also occurred when FLOX was spilled upon sand soaked with JP-4 and upon sand soaked with SAE 20 oil. Slow-motion, closeup motion pictures of the reaction with the oil-soaked sand distinctly showed three large reactions and about five smaller ones, all of which occurred within less than 1 second. In an attempt to smooth the FLOX - JP-4 reaction, 30-percent FLOX was spilled into a JP-4 stream. As shown in table II, multiple explosions still occurred. A similar test was made with a coarse spray of JP-4; multiple explosions were again noticed, but they were very rapid, less severe, and considerably smoother. In all these tests, the rate of rise and the angle of rise of the resultant toxic clouds were dependent upon the apparent amount of heat produced by the reaction. Small temperature inversions (increases in temperature with height) did not seem to affect the diffusion and dispersion of these clouds appreciably, although no measurements were taken for confirmation. In some cases, for example, with spills on asphalt, there was very little reaction with low heat release. (The relative amount of heat release was based upon visual observation of the size and the brightness of the flame.) In such cases, the reaction cloud rose at low angles.

When 30-percent FLOX was spilled into lake water (untreated water from Lake Erie), no combustive reaction occurred; however, a postspill chemical analysis revealed that the acid content (hydrofluoric) of the water had increased considerably after the spill (table II). In order to observe any possible effect of greater contact-surface area on reactivity, 30-percent FLOX was also spilled into a shower-head spray of lake water. Again, there was no combustive reaction, and the acid content of the water was increased considerably. For 30-percent FLOX spills, therefore, the use of water as a reactant to control air pollution is unfeasible. Water also does not appear to be feasible as a scrubbing agent. Very large quantities would have to be used in a very short interval of time (in excess of the flashing rate of the cryogenic FLOX). In order to prevent soil and stream pollution, treatment of the water would also be required to neutralize the acidity.

When FLOX was spilled onto charcoal, a smooth, large, brilliant flame occurred over a period of about 2.5 seconds. A white cloud rose rapidly at about a 65° angle and many minute, short-lived sparks (about 1-sec duration) were swept upward (fig. 4). The flame was so brilliant that observers approximately 140 yards away viewing through 7×50 binoculars experienced a loss of normal vision for about 10 minutes. The duration of the reaction and the size of the fireball indicated that a large percentage of the spilled FLOX was involved in the reaction. The absence of any explosive effect in the reaction of FLOX with charcoal compared with some of the other reactions apparently prevented any explosive separation of fuel from oxidizer and thereby allowed a more complete reaction.

In addition to the smoothness of this reaction, a unique feature is that the reaction product of fluorine and charcoal is carbon tetrafluoride, which is an inert and nontoxic gas (refs. 1 to 3). The use of charcoal to dispose of fluorine is standard practice in fluorine facilities because of the highly efficient manner in which burning occurs and because of the inert combustion products. Hypergolicity has been demonstrated at fluorine concentrations as low as 1 percent in charcoal reactors (ref. 4). In case of a fluorine spill onto charcoal, the carbon tetrafluoride produced would serve as a diluent to both the unreacted fluorine and the hydrogen fluoride produced with atmospheric moisture. The concentration of pollutants is thereby reduced. In case of a FLOX spill onto charcoal, both carbon tetrafluoride and carbon dioxide act as diluents. The overall characteristics of the FLOX-charcoal reaction appear to make the placement of charcoal in areas of possible fluorine or FLOX spills an effective method of providing spill and pollution control. Providing weatherproofed containers or bins would be desirable to keep the charcoal clean and dry.

100-Percent Fluorine Spill Tests

Results of spills of 100-percent fluorine onto several materials are shown in table III. In previous occurrences at Lewis, where fluorine was unintentionally spilled or impinged upon concrete and metals within test cells, either no reaction or minimal surface effects resulted; therefore, a spill test of fluorine onto concrete was not performed in this program.

Fluorine reacted violently both with water-soaked sand and with a puddle of lake water. In the water-puddle test, a loud detonation occurred, an almost instantaneous small flash of white light was observed, and a strong shock wave was felt by observers 140 yards away. (Liquid fluorine in contact with water and/or ice is a highly unstable combination and after an indefinite time delay reacts explosively.) A white cloud rose at an angle of only about 35°, which indicated a relatively small heat release. The type of reaction indicated that only a small amount of the fluorine spilled reacted combustively with the water; the remainder was blown away by the blast. The heat content of the cloud represented the heat release from a small but very rapid reaction.

When liquid fluorine was spilled upon JP-4, smooth burning occurred, as opposed to the multiple explosions previously discussed with the 30-percent FLOX spills. A yellow fireball about 25 feet in diameter developed, and the resultant black cloud rose rapidly, almost vertically, then drifted downwind, and slowly dissipated. The smoothness of this reaction compared with the multiple explosion effect of the 30-percent FLOX - JP-4 reaction indicated that the presence of oxygen permitted the formation of many small zones of explosive mixtures, which resulted in a series of rapid microexplosions.

Liquid-Oxygen Spill Tests

The final spill tests were made with LOX upon JP-4 to obtain comparisons between LOX-fuel spills and FLOX-fuel spills. Two tests were made, and the results are tabulated in table IV. First, LOX was spilled upon JP-4 with no separate source of ignition; there was no reaction, and the vapors drifted downwind along the ground. Then, LOX was spilled upon JP-4 with a continuous spark-plug arc in the vicinity of the mixture. A violent detonation occurred with a brilliant white flash, and a strong shock wave was felt at a distance of about 140 yards. The resultant black cloud rose rapidly, almost vertically, then drifted downwind, and slowly dissipated. Considerable damage was done to the blast shield by the explosion.

The explosive LOX - JP-4 reaction thus produced a high degree of heat release, as compared with the explosive fluorine-water reaction. This fact is explained by the fact that the reaction delay allowed considerable mixing of the LOX - JP-4 before reaction ignition. On the other hand, in the fluorine-water reaction, the time delay was sufficient to permit an explosive combination to form but not long enough to allow a large portion of the fluorine to enter into reaction before being blown away by the blast.

Comparisons in Reactivity Among LOX, FLOX, and Fluorine

An abbreviated composite of the test results is shown in table V. This table shows the reactivity of LOX, 30-percent FLOX, and 100-percent fluorine with the various materials in generally increasing order of reactivity and in such a manner that comparisons can easily be made. A comparison between the 100-percent fluorine column and the 30-percent FLOX column illustrates the diluent effect of oxygen; some materials which did not react with FLOX did react with fluorine. There is thus some fluorine concentration between 30 and 100 percent at which FLOX becomes hypergolic with each of these materials; however, this hypergolicity would also depend upon the particle size of the materials.

This table also gives the approximate angle of cloud rise, which is indicative of the efficiency or the amount of heat produced by the reaction. Wind speeds are given, so that they may be taken into consideration when comparisons are made. The high angles of rise produced by reactions with hydrocarbons and charcoal are noted.

CONCLUSIONS

In an investigation of the reaction characteristics of liquid fluorine and FLOX (fluorine-oxygen mixture) spills upon various materials, the following conclusions were drawn:

1. When fluorine or FLOX (as low as 30-percent fluorine concentration) is spilled upon many common materials which might be found around a test or launch facility, sufficient hypergolic reaction can occur to create a combustive "hot spill." The hot spill creates a rapid cloud rise, which provides efficient diffusion and dispersion of the toxic products. This effect could be a useful tool in pollution control.

2. Spills of liquid fluorine or FLOX of any concentration upon concrete or metal surfaces results in a typical cryogenic "cold spill" with only minimal surface effects or no reaction at all. The characteristic cold spill creates a downwind ground-level pollution problem, which, if permitted, should be antic-

ipated and controlled during operations.

3. The smoothness of the 100-percent fluorine - JP-4 reaction compared with the multiple explosion effect of the 30-percent FLOX - JP-4 reaction and the violent detonation of the artificially ignited LOX - JP-4 reaction indicated that an increase in the percentage of fluorine present tends to smooth out the reaction by reducing the formation of pockets of explosive mixtures.

4. The smoothing out of the reaction which occurred when FLOX was spilled into a spray of JP-4 instead of a pool of JP-4 demonstrated that smoother reactions occur when the particle size of the reactants is reduced.

5. A combination of hypergolicity, the particle size of the reactants, and the time delay in reaction initiation determines the efficiency of a reaction and the violence or smoothness of a reaction from an unconfined spill.

6. Since water is not hypergolic with 30-percent FLOX under ordinary circumstances, water used on a 30-percent FLOX spill would be ineffective in producing the heat required for a hot rise. If used as a deluge, acidification of the water would result, which would necessitate treatment to neutralize the water.

7. The desirable characteristics of the 30-percent FLOX - charcoal reaction (smooth burning, high heat release, and diluent effect of the nontoxic reaction products) indicate that placement of charcoal in areas where fluorine or FLOX spills might occur could be an effective means for air pollution control.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, September 14, 1965.

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2. Haszeldine, R. N.; and Sharpe, A. G.: Fluorine and Its Compounds. John Wiley & Sons, Inc., 1951.

3. Simons, J. H.: Scientific and Utilitarian Value of Fluorine Chemistry. Ind. Eng. Chem., vol. 39, no. 3, Mar. 1947, pp. 238-241.

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TABLE I. - CHECKOUT RUNS WITH LIQUID OXYGEN

[No weather data taken nor material weight measured.]

Test material	Spill tank pressure, psig	Liquid-oxygen spill quantity, lb	Reaction	Cloud or vapor behavior
Empty stainless-steel pan	20	15	None	Vapors drifted close to ground
Top soil	10	30	None	Vapors drifted close to ground
Top soil	5	20	None	Vapors drifted close to ground
Top soil soaked with SAE 20 oil	5	10	None	Vapors drifted close to ground

TABLE II. - 30-PERCENT FLOX SPILL TESTS

Test material		Weather conditions		FLOX spill quantity, lb	Reaction	Cloud or vapor behavior	Moisture content, percent		Acid content, pH	
Substance	Weight, lb	Wind speed, mph	Temperature gradient up to height of 30 ft, °F/ft				Before test	After test	Before test	After test
Concrete slab	480.0	8	-0.02910	10	None	Vapors drifted close to ground	----	----	----	----
Limestone slab	332.0	5	-0.03637	10	None	Vapors drifted close to ground	----	----	----	----
Crushed limestone	452.0	3	-0.07270	10	None	Vapors drifted close to ground	----	----	----	----
Sand	475.0	8	-0.00727	10	None	Vapors drifted close to ground	1.00	1.34	----	----
Water-soaked sand	552.0	1	+0.03637	10	None	Vapors drifted close to ground	19.28	14.97	8.40	7.60
Lake-water puddle (5 gal)	41.7	1	-0.03637	10	None	Vapors drifted close to ground	----	----	8.30	2.30
Lake-water spray	Not measured	5	-0.06910	10	None	Vapors drifted close to ground	----	----	6.70	1.30
Coke (low grade)	265.0	3	-0.07270	10	Small reaction; small flame	Gray cloud rose at about 15° angle	2.56	5.77	----	----
Asphalt (from building)	177.0	8	0	10	Sputtered; small flames	Gray cloud drifted close to ground at about 10° angle	----	----	----	----
Asphalt (from road)	258.0	2	+0.03637	10	Loud sputtering; small flashes	Gray cloud rose at about 25° angle	0.08	0.37	----	----
Top soil	311.0	8	0	10	Bright flame	White cloud rose high at about 45° angle	8.51	12.17	----	----
Oil-soaked sand	478.0	8	0	10	Several loud, rapid explosions	Gray cloud rose high at about 55° angle	1.03	1.31	----	----
JP-4 soaked sand	478.4	2	+0.01813	10	Loud sputtering; large flames	Dark cloud rose high at about 60° angle	14.[illegible]5	1.[illegible]5	----	----
JP-4 puddle (3 gal)	20.1	2	0	10	Several loud, rapid explosions; large fireball	Black cloud rose high, almost vertically, at about 60° angle	----	----	----	----
JP-4 stream (bad timing)	6.7	5	-0.06910	10	Several loud, rapid explosions; large fireball	Black cloud rose high at about 60° angle	----	----	----	----
JP-4 stream (good timing)	6.7	5	-0.08000	10	Several loud, rapid explosions; large fireball	Black cloud rose high at about 55° angle	----	----	----	----
JP-4 (coarse spray)	6.7	5	-0.07270	10	Very rapid, smooth explosions; large fireball	Black cloud rose high at about 65° angle; smoke ring formed	----	----	----	----
Charcoal	180.0	8	0	10	Large, smooth, brilliant fireball	White cloud rose high at about 65° angle and disappeared	5.94	17.95	----	----

TABLE III. - 100-PERCENT FLUORINE SPILL TESTS

Test material		Weather conditions		Liquid-fluorine spill quantity, lb	Reaction	Cloud or vapor behavior	Moisture content, percent		Acid content, pH		Remarks
Substance	Weight, lb	Wind speed, mph	Temperature gradient up to height of 30 ft, °F/ft				Before test	After test	Before test	After test	
Crushed limestone	411.0	15	0	10	Sputtered; small yellow flames	Grayish cloud rose at about 30° angle	----	----	----	----	------------
Sand	320.0	5	-0.02546	10	Bright flame	White cloud rose at about 50° angle	2.70	4.20	----	----	------------
Water-soaked sand	321.0	5	0	10	Strong explosion	Light gray cloud rose at about 30° angle	16.30	12.68	8.70	3.55	------------
Asphalt (from road)	275.0	5	0	5	Sputtered; small flashes	Gray cloud drifted close to ground at 5° angle	0.03	0.05	----	----	------------
Lake-water puddle (5 gal)	41.7	12	-0.04000	5	Loud, sharp detonation	White cloud rose at about 35° angle	----	----	8.15	1.35	Blast shield damaged
JP-4 puddle (2 gal)	13.4	5 to 10	0	5	Smooth, fast burning; large fireball	Black cloud rose at about 60° angle	----	----	----	----	------------

TABLE IV. - LIQUID-OXYGEN SPILL TESTS

Test material		Weather conditions		Liquid-oxygen spill quantity, lb	Reaction	Cloud or vapor behavior	Remarks
Substance	Weight, lb	Wind speed, mph	Temperature gradient up to height of 30 ft, °F/ft				
JP-4 puddle (1 gal)	6.7	8	-0.07270	5	None	Vapors drifted close to ground	--------
JP-4 (2 gal) with spark-plug igniter	13.4	8	-0.07270	10	Loud, sharp detonation	Black cloud rose fast, at about 80° angle	Blast shield damaged

TABLE V. - COMPOSITE RESULTS

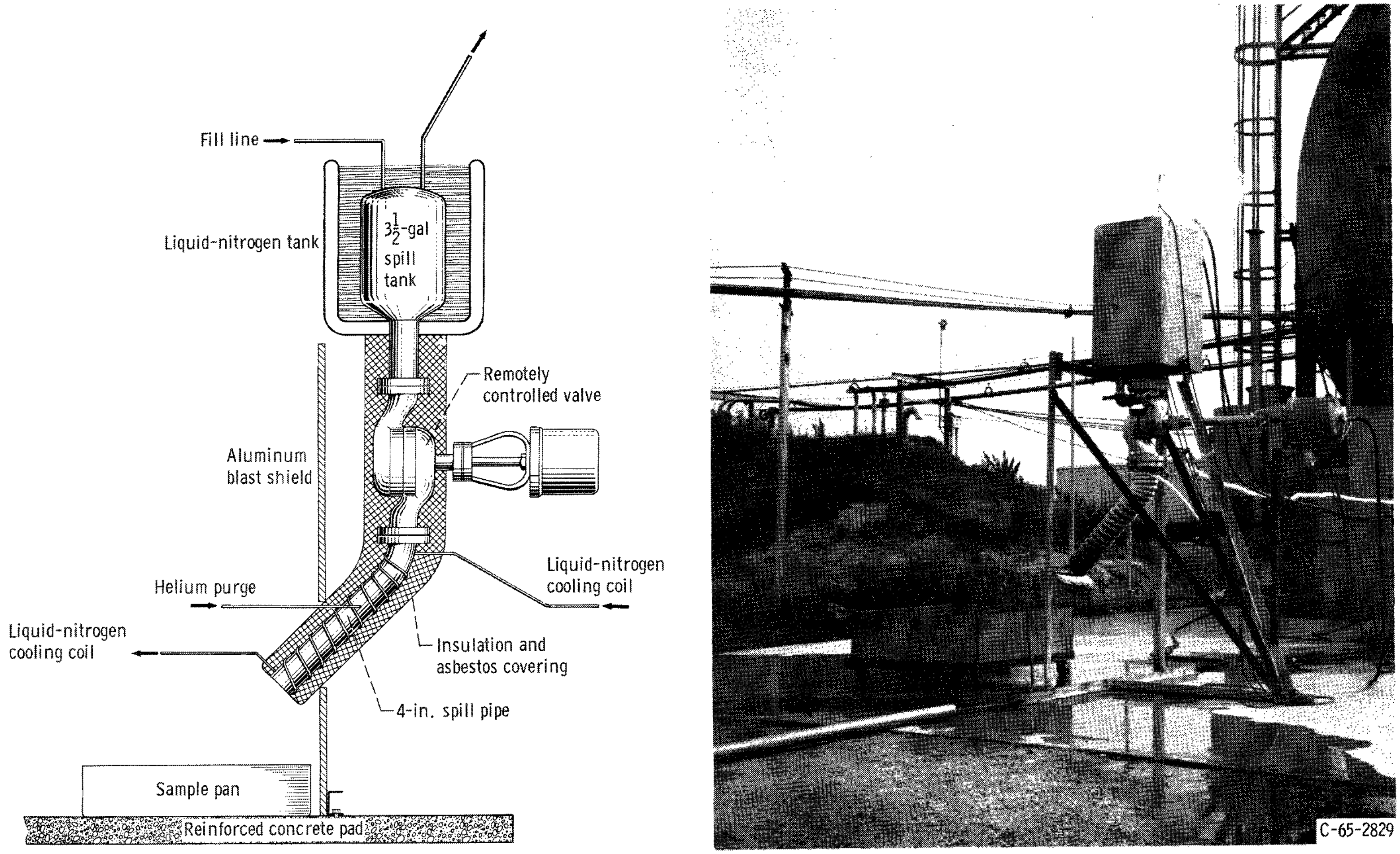
[Unless otherwise noted, 10-lb quantities were spilled.]

Test material	Test fluid								
	Liquid oxygen			30-Percent FLOX			100-Percent Fluorine		
	Combustive reaction	Wind speed, mph	Approximate cloud angle, deg	Combustive reaction	Wind speed, mph	Approximate cloud angle, deg	Combustive reaction	Wind speed, mph	Approximate cloud angle, deg
Empty stainless-steel pan	No	Not measured	0	--	--	--	--	--	--
Concrete slab	--	--------	--	No	8	0	No[a]	Not measured	Not measured
Limestone slab	--	--------	--	No	5	0	----	--------	--------
Crushed limestone	--	--------	--	No	3	0	Yes	15	30
Sand	--	--------	--	No	8	0	yes	5	50
Water-soaked sand	--	--------	--	No	1	0	Yes	5	30
Lake-water puddle	--	--------	--	No	1	0	Yes[b]	12	35
Lake-water spray	--	--------	--	No	5	0	----	--	--
Coke (low grade)	--	--------	--	Yes	3	15	----	--	--
Asphalt slab	--	--------	--	Yes	2	25	Yes[b]	5	5
Top soil	No	Not measured	0	Yes	8	45	----	--	--
Oil-soaked sand	--	--------	--	Yes	8	55	----	--	--
Oil-soaked top soil	No	Not measured	0	---	--	--	----	--	--
JP-4 soaked sand	--	--------	--	Yes	2	60	----	--	--
JP-4 puddle	No[b]	8	0	Yes	2	80	Yes[b]	5 to 10	60
JP-4 stream	--	--------	--	Yes	5	55	----	--	--
JP-4 coarse spray	--	--------	--	Yes	5	65	----	--	--
JP-4 with spark-plug ignition	Yes	8	80	---	--	--	----	--	--
Charcoal	---	--------	--	Yes	8	65	Yes[c]	Not measured	Not measured

[a]Previous Lewis experience.

[b]5-lb spill.

[c]Ref. 4.



(a) Diagrammatic sketch (not to scale).

(b) Photograph of apparatus without insulation and shield.

Figure 1. - FLOX spill test apparatus.

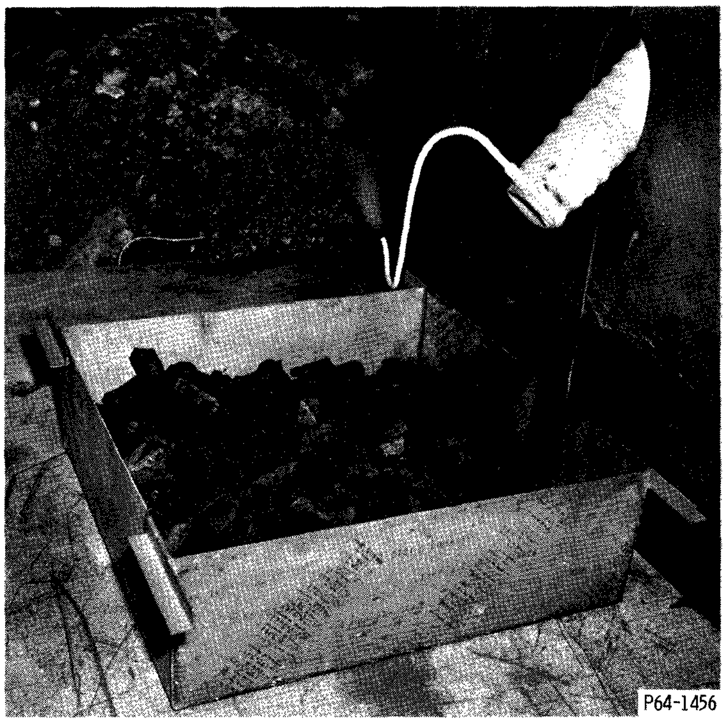


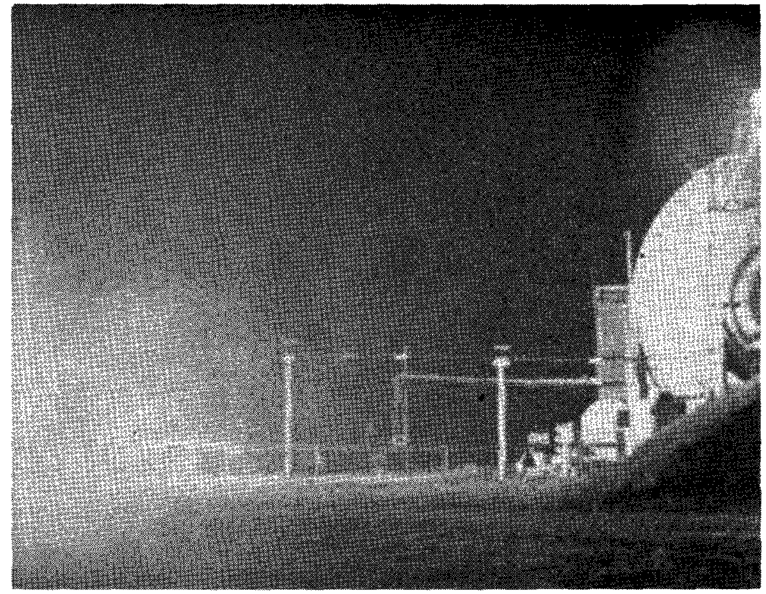
Figure 2. - Charcoal test sample.

(a) Fireball.

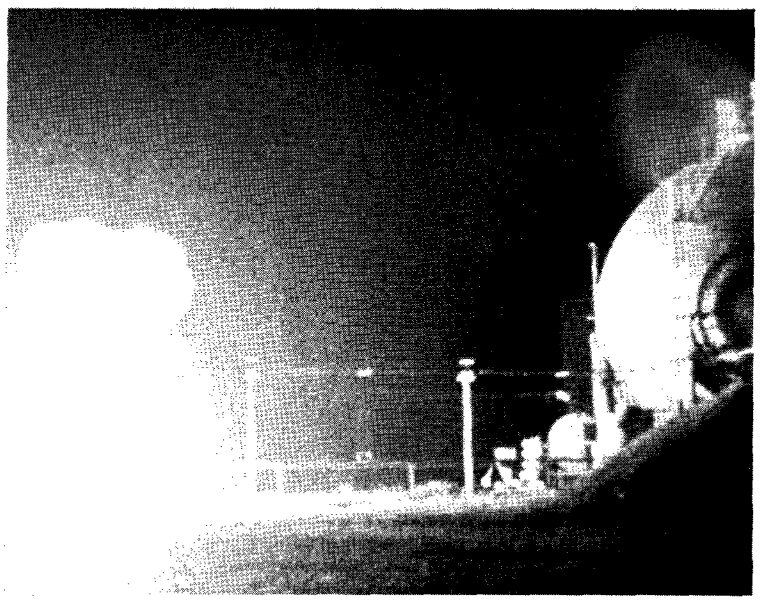
(b) Reaction cloud (2.5 sec later).

Figure 3. - 30-Percent FLOX spill onto JP-4. Distance, approximately 120 yards.

(a) Initial reaction.



(b) Fireball.



(c) Reaction cloud.

(d) Sparks.

Figure 4. - 30-Percent FLOX spill onto charcoal. Distance, approximately 50 yards.

A motion-picture film supplement C-243 is available on loan. Requests will be filled in the order received. You will be notified of the approximate date scheduled.

The film (16 mm, 16 min, color, sound) shows different views of reaction characteristics when liquid fluorine, liquid oxygen, and liquid FLOX were spilled upon various materials.

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