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THE BEHAVIOR OF URANIUM, THORIUM, AND OTHER SELECTED MATERIALS IN BROMINE TRIFLUORIDE, BROMINE PENTAFLUORIDE, CHLORINE TRIFLUORIDE, AND FLUORINE AT ELEVATED TEMPERATURES

by

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ABSTRACT

The reactivity of uranium, thorium, aluminum, copper, iron, magnesium, zirconium, and Fluorothene with bromine trifluoride, bromine pentafluoride, chlorine trifluoride, and fluorine was investigated at temperatures ranging from 25° to 410°C. The reactivity of platinum with bromine trifluoride was also studied.

Uranium dissolved smoothly in the liquid interhalogen compounds but ignited under certain conditions in the vapors of the compounds and in fluorine gas. The minimum temperatures at which the vapor-phase ignitions began were as follows:

Reagent (vapor or gas)	Approximate Pressure (mm)	Uranium Ignition Temperature (°C)
BrF_3	4500	190
BrF_3*	1500	150
BrF_5	5300	225
ClF_3	2800	205
F_2	4600	260

* Uranium first submerged in liquid; then exposed to vapor.

Thorium did not react appreciably with any of the reagents under the imposed conditions. Zirconium ignited at 340°C in vapor-phase reactions with chlorine trifluoride and fluorine. Fluorothene also ignited in these reagents at 275° and 330°C, respectively. Platinum dissolved rapidly in liquid bromine trifluoride at 200°C. The remainder of the materials of construction considered did not react appreciably with the test reagents during exposures of 10 to 30 minutes.

INTRODUCTION

In the bromine trifluoride process(1) for the recovery and decontamination of irradiated uranium, the following reaction is used to convert uranium metal directly to uranium hexafluoride:

$$U \text{ (solid)} + 2BrF_3 \text{ (liquid)} \longrightarrow UF_6 \text{ (liquid)} + Br_2 \text{ (liquid)}$$

The reaction may proceed stepwise, forming uranium tetrafluoride and pentafluoride as intermediates, but these are ultimately fluorinated to uranium hexafluoride. The dissolution is usually carried out at temperatures ranging from 50° to 125°C. Since the reaction is exothermic, heat removal is necessary to prevent the temperature from rising, especially if the products, which accelerate the reaction, are allowed to build up. The present experiments were undertaken principally to determine the behavior of uranium in bromine trifluoride at higher temperatures and to define an upper temperature limit for safe dissolving procedures.

The chlorine trifluoride process(2) also produces uranium hexafluoride directly from uranium metal by a liquid-phase reaction, but the conditions differ from those of the bromine trifluoride process. For comparison purposes, the reactions between uranium and chlorine trifluoride, bromine pentafluoride, and fluorine were studied, but to a lesser extent than the uranium-bromine trifluoride reaction.

Hazardous conditions were found to exist in the uranium-vapor reactions at elevated temperatures with each of these fluorinating reagents; conditions for "ignition" of the uranium were determined.

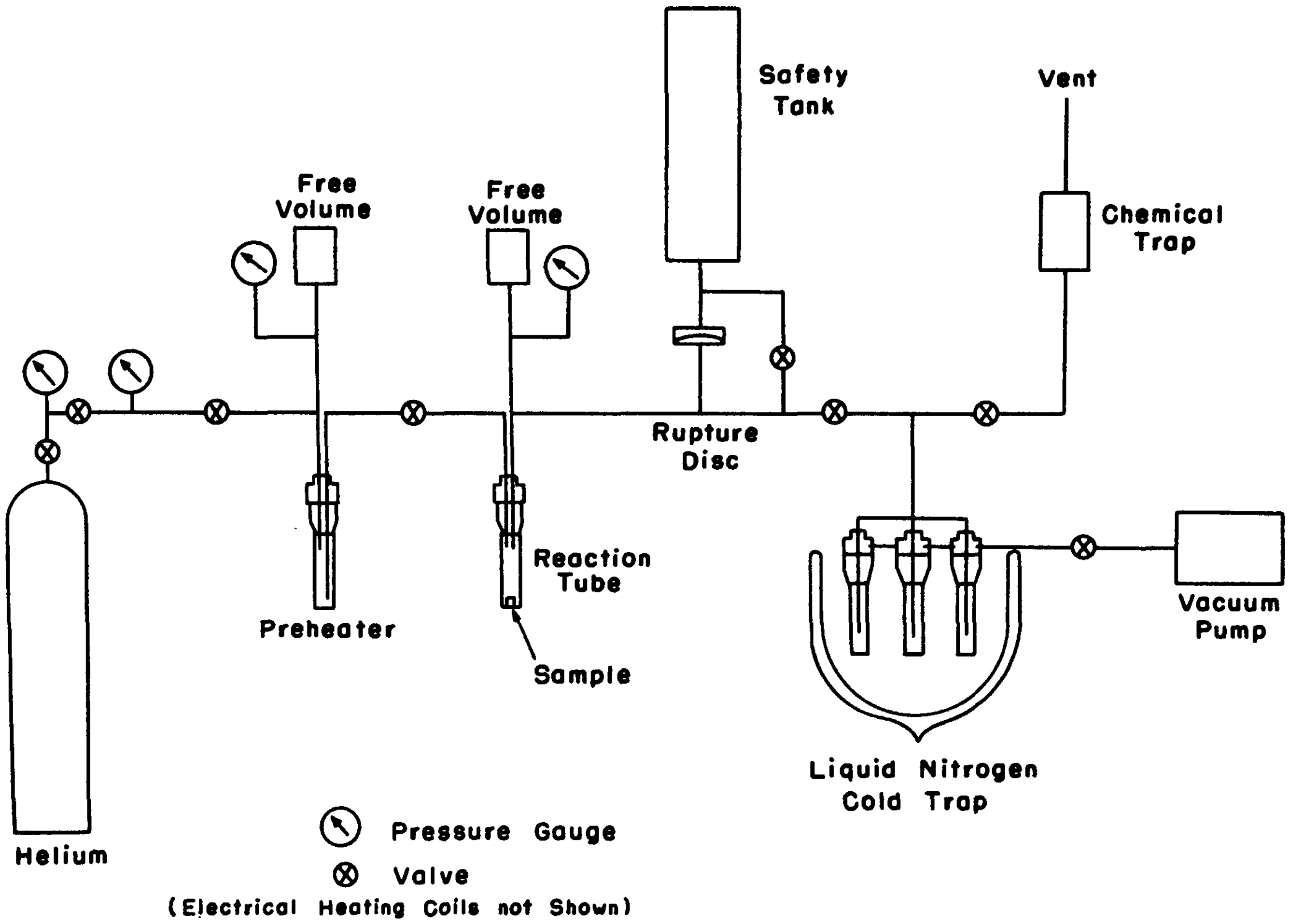
Thorium was found to be only superficially attacked by the reagents under the conditions of these experiments. The results of test exposures of some materials of construction are given in the text.

EXPERIMENTAL

The original apparatus used in these experiments is shown in Figure 1. The parts were constructed of nickel or Monel except for valves, which were of brass with triple bronze and steel diaphragms (Hoke Model 322). All parts in contact with liquid or vapor were heated electrically by asbestos-covered nichrome wire. Copper-Constantan thermocouples were externally attached to the vessels and lines at numerous points for measuring temperatures. The entire apparatus was built behind a concrete block wall for the protection of the operator; valves were opened and closed by extension handles, and pressure gauges were observed with mirrors.

Figure 1

FLUORINATION APPARATUS



The two free-volume vessels shown in the figure were present to increase the vapor space and thus prevent the pressure from rising too rapidly during the course of a reaction. The rupture disc and safety tank provided further protection in the event of a rapid, uncontrolled pressure increase.

In the first experiments, cylindrical slices of α-rolled uranium, 1/4-inch in diameter and weighing two to three grams, were used. Each sample was placed in the bottom of the 3/4-inch nickel or Monel reaction tube and heated to the desired temperature under vacuum. Bromine trifluoride was placed in the preheater tube, pressurized with helium, and heated to approximately the same temperature. When the intervening valve was opened, the hot bromine trifluoride was blown onto the hot uranium. Temperature and pressure were recorded as the reaction proceeded. After a given contact time, the reaction was stopped by distilling the bromine trifluoride and volatile products into the cold trap under reduced pressure.

The temperature of the reaction tube directly beneath the uranium was found to surge occasionally during the first five runs, so that a continuous record of temperature could not be obtained with a manually operated slidewire potentiometer. A high-speed recording potentiometer (Brown Electronik, Model No. 153X17V-V-X6) was therefore employed in all subsequent runs. Voltage-dividing resistors were used on the input side of the recording potentiometer to extend the range from 10 millivolts to 100 millivolts for some measurements.

The first ten runs were made at temperatures ranging from 20° to 223°C, with contact times varying from twelve minutes to one hour. The runs were inconclusive since it was not known whether the uranium was always covered with liquid bromine trifluoride. In run 10, an extreme temperature surge occurred, beginning at 223°, which resulted in melting the bottom of the nickel test tube; the attached thermocouple was blown off by the explosive release of pressure (365 $\mathrm{lb/in.^2}$). Figure 2 is an enlarged photograph of the bottom of this test tube.

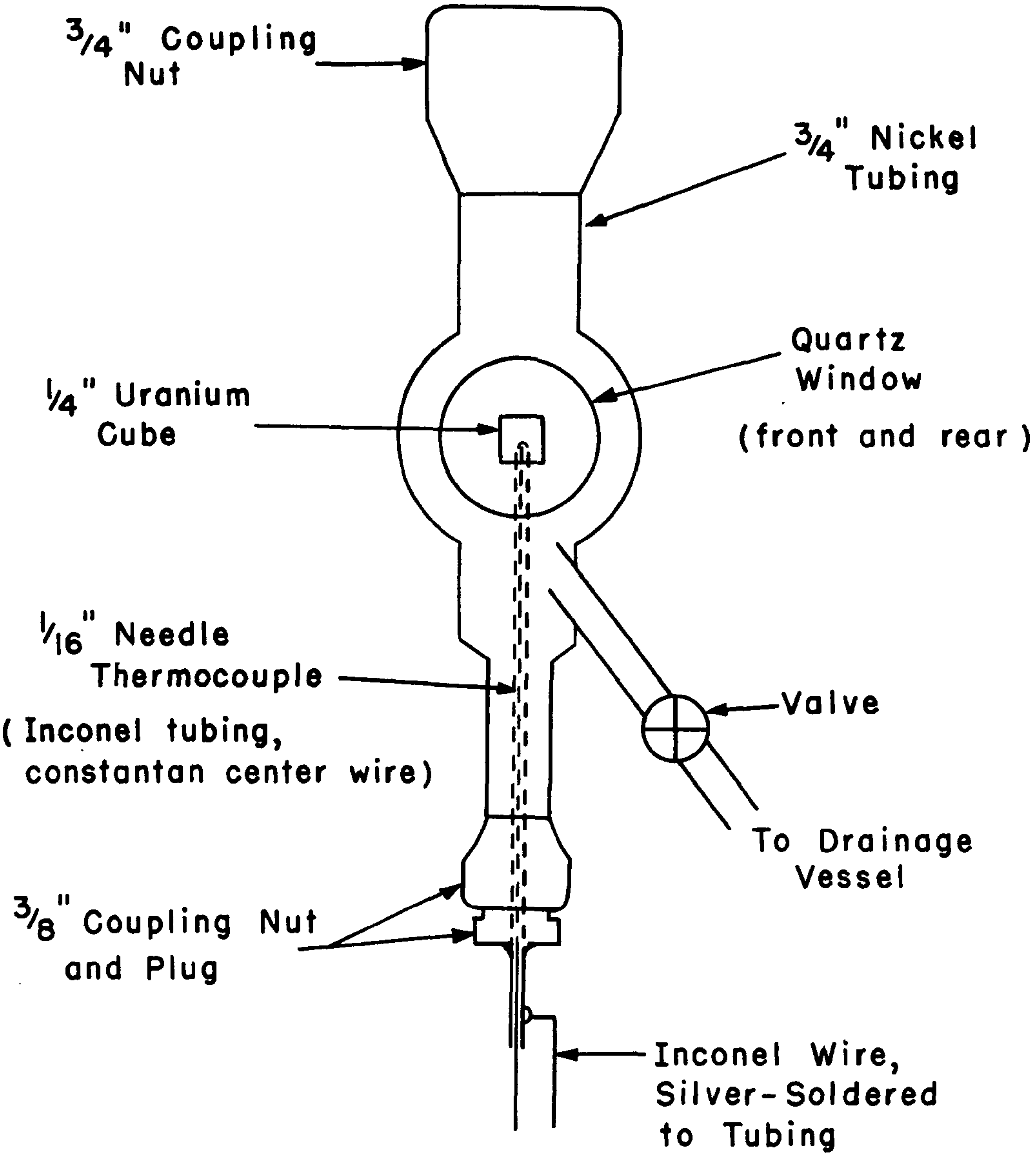
After run 10 it became obvious that the uranium was sometimes melted by the heat of the reaction; the apparatus was therefore modified so that the uranium could be watched during the fluorination. The 3/4-inch reaction tube was replaced by a cell of the type shown in Figure 3.

Quartz windows at the front and rear, 3/16-inch thick and 13/16-inch in diameter, were seated against Teflon gaskets. A light behind the rear window silhouetted the uranium sample. The operator observed an enlarged image of the cell from the other side of the concrete wall by a mirror and monocular system. The uranium used in this apparatus was in the form of a cube, slightly less than 1/4-inch on each side, with a circular hole in one face into which an Inconel-Constantan needle thermocouple(3) was inserted.

Figure 2

BOTTOM VIEW OF 3/4-INCH NICKEL TEST TUBE USED IN RUN 10.

Enlargement approximately 10X.
Droplet formation on edges of hole provides evidence of melting.



(Electrical heating lines not shown)

Figure 3

CELL FOR OBSERVING URANIUM IGNITION IN INTERHALOGEN

The sample was supported in the field of view by the thermocouple and isolated from the walls of the cell; heat loss from the sample by conduction was thereby minimized.

As before, the bromine trifluoride and uranium were usually heated separately, then the liquid bromine trifluoride was flashed into the cell to a level completely covering the uranium. The reaction could be carried out in the liquid phase, or the liquid level could be lowered to completely expose the uranium to vapor by opening the drainage valve. In the latter case, some liquid remained in the bottom of the cell and maintained its vapor pressure(4) in the system. The surface of the uranium cube probably remained wet for a short time after the liquid was drained off; the conditions were therefore not representative of a true solid-vapor reaction but of a situation which might occur in process work. Most of the experiments were done in this manner, but, for comparison, uranium was also exposed to bromine trifluoride vapor without first being wetted by liquid, and the behavior was shown to be affected by the change of conditions.

In experiments with chlorine trifluoride and bromine pentafluoride, the high vapor pressures restricted the temperatures to which the liquid phases could be heated. Uranium was heated to 170°C in liquid bromine pentafluoride, then the liquid was drained off, and the temperature was increased further with only vapor in the reaction cell. The vapor-phase fluorinations in chlorine trifluoride were carried out by condensing a small amount of liquid in the cell, then vaporizing it completely by raising the temperature. An approximate threshold temperature was determined for the ignition of uranium in each reagent.

Test specimens of thorium, aluminum, copper, iron, magnesium, zirconium, and Fluorothene were machined into 1/4-inch cubes and tested in the manner described above for uranium. Since the apparatus was constructed of nickel, Monel, Inconel, brass, quartz, and Teflon, some knowledge of the reactivity of these materials was also obtained.

DISCUSSION OF RESULTS

A. Uranium Exposed to Liquid Reagents.

In all exposures of uranium to the liquid halogen fluorides, the uranium dissolved smoothly and the temperature remained constant or rose slowly due to the heat liberated by the reaction. Data for such exposures are given in Table 1. The temperatures at which the tests were terminated are those listed in column 4. Temperatures are reported to only ± 5° since the e.m.f. characteristics of the Inconel-Constantan needle thermocouples were taken directly from ANL-5096(3) without calibration.

Table 1

EXPOSURE OF URANIUM METAL TO LIQUID FLUORINATING REAGENTS

Approximately 3 grams of alpha-rolled reactor grade uranium used in each experiment.

Run No.	Reagent	Initial U Temp (°C)	Final U Temp (°C)	Contact Time (min)	Results
12	BrF_3	170	180	44	Smooth Reaction
13	BrF_3	205	205	26	Smooth Reaction
19	BrF_3	210	210	16	Smooth Reaction
31	BrF_3	25(a)	160	17	Smooth Reaction
27	BrF_3 + UF_6(b)	195	220	22	Smooth Reaction
33	BrF_5	130	170	25	Smooth Reaction
35	BrF_5	25(a)	160	28	Smooth Reaction
41	ClF_3	25(a)	70	7	Smooth Reaction

(a) U and interhalogen heated together from room temperature.

(b) Initial solution = 10.3 mole per cent UF_6.

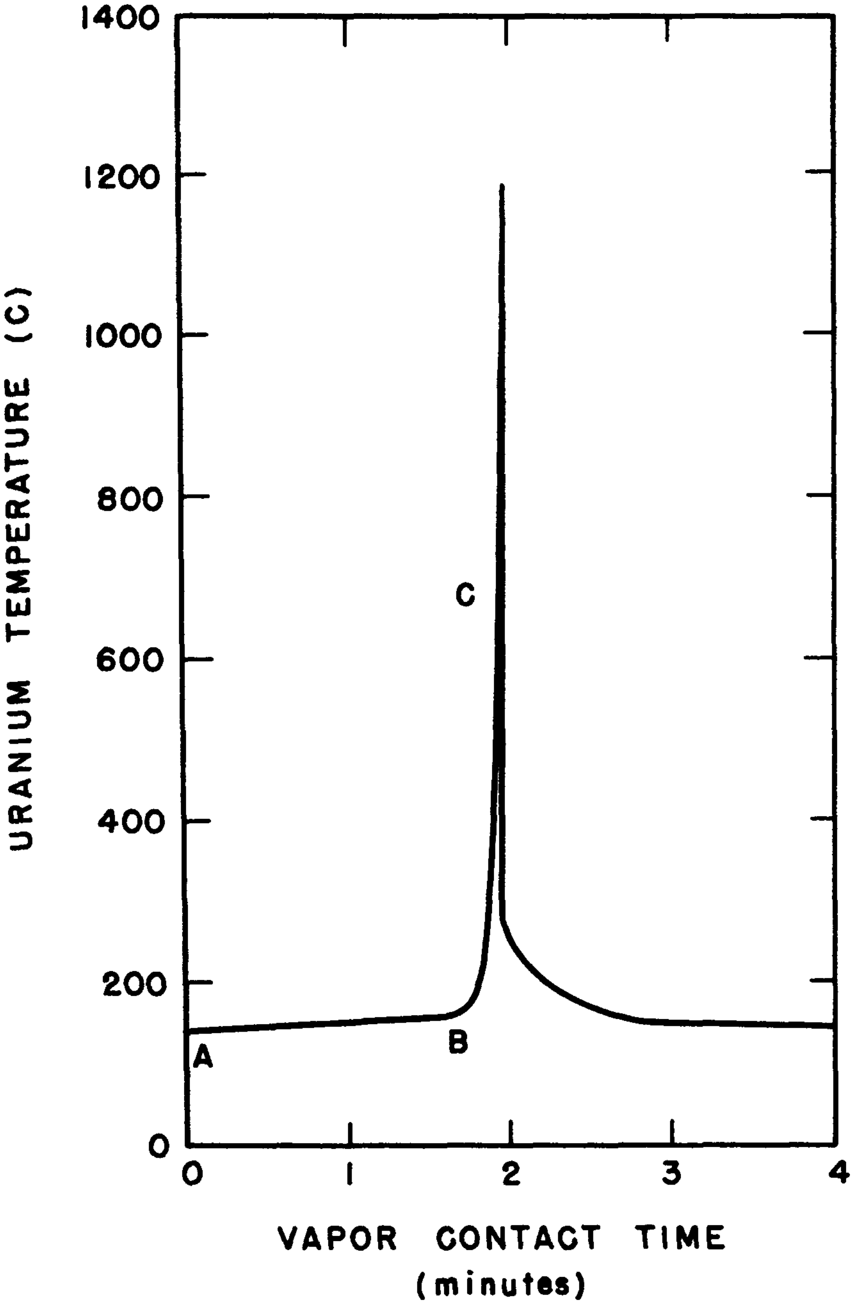
B. Uranium Exposed to Gaseous Reagents.

In all experiments in which uranium was first covered with liquid bromine trifluoride and then exposed to bromine trifluoride vapor at 150°C or higher, ignition of the test specimen occurred. The temperature rose slightly for about one or two minutes, then surged abruptly above the melting point of uranium. In a typical ignition, the color of the vapor near the uranium first changed rapidly from yellow to red; the uranium quickly became incandescent, formed a molten yellow globule, and flowed to the bottom of the cell. Usually the Inconel-Constantan thermocouple also melted, indicating that the temperature exceeded 1400°C at the peak. The pressure increased only slightly during ignition. Some uranium was recovered after the reaction, either alloyed with the thermocouple metals as a shiny, spherical button or surrounding the thermocouple stem as a black deposit coated with green fluoride film.

Figure 4 is a sample curve of "temperature" vs. "time" obtained from the chart of the recording potentiometer. At point A the liquid level

Figure 4

REPRESENTATIVE TEMPERATURE CURVE FOR URANIUM IGNITION IN BROMINE TRIFLUORIDE VAPOR



was lowered, exposing the cube to vapor. At B the temperature began to rise suddenly; the approximate starting point of this sharp ascent was taken as the ignition temperature. Incandescence of the uranium began at about point C. The maximum temperature was above 1000°C but was not accurately known. Although the needle thermocouple was often destroyed, the metal junctions in the solidified melt continued to generate an e.m.f.

The data for ignition of uranium in bromine trifluoride vapor after immersion in liquid are given in Part A of Table 2. The "Initial Uranium Temperature" is the temperature at which the liquid level was lowered, exposing the uranium to vapor, and the "Vapor Contact Time" is the elapsed time in the vapor before ignition. It can be seen that at 150°C or higher, ignition usually occurred within several minutes. No ignitions were observed below 150°. This lower temperature limit is not sharply defined, however, and is probably dependent upon such conditions as the surface-to-volume ratio of the uranium, the surface pretreatment, and especially the heat transfer to surroundings.

The addition of uranium hexafluoride or bromine to the bromine trifluoride raised the threshold temperature for ignition slightly, as shown in runs 28, 32, and 49 through 52. Both of these substances, therefore, appear to be inert diluents in the vapor-phase reaction. Since uranium hexafluoride and bromine are both more volatile than bromine trifluoride, their concentrations in the vapor phase in these experiments were higher than the solution concentrations given in Table 2.

The results of two experiments in which uranium was heated in bromine trifluoride vapor without prior immersion in liquid are given in Part B of Table 2. Here the "Vapor Contact Time" is the entire period of heating from room temperature to the ignition temperature. The uranium ignited at 190°C in both experiments. This indicates that the ignitions at lower temperatures were largely due to liquid bromine trifluoride adhering to the surface of the uranium. The surface liquid would be expected to provide a high initial reaction rate and high output of heat.

A sequence of photographs taken from a color motion picture of uranium igniting in bromine trifluoride vapor is shown in Figure 5. Figure 5a shows the uranium cube on the needle thermocouple immediately before ignition. In Figure 5b, one corner of the cube is incandescent; in Figure 5c the entire cube is incandescent and beginning to melt. In Figures 5d and 5e the molten globule is decreasing in size as metal runs down the thermocouple. Only the thermocouple tip remains in view in Figure 5f after a total elapsed time of 3.9 seconds.

Table 2

IGNITION OF URANIUM METAL IN VAPOR-PHASE FLUORINATION

Approximately 3 grams of alpha-rolled, reactor-grade uranium used in each experiment.

Pressure = Vapor pressure of liquid reagent at indicated temperature.

Run No.	Reagent (vapor)	Initial U Temp. (°C)	Ignition Temp. (°C)	Vapor Contact Time (min)
PART A - Uranium exposed to vapor after immersion in liquid:				
13	BrF_3	205	205	2
14	BrF_3	205	205	2
15	BrF_3	155	185	8
16	BrF_3	135	150	20
17	BrF_3	145	205(a)	48
18	BrF_3	150	150	2
19	BrF_3	210	210	1
20	BrF_3	185	185	1
21	BrF_3	190	190	2
22	BrF_3	185	185	2
23	BrF_3	210	210	2
24	BrF_3	175	175	2
25	BrF_3	205	205	1
26	BrF_3	260	260	1
30	BrF_3	170	170	1
31	BrF_3	160	160	2
28	$BrF_3 + UF_6$(b)	160	170	17
32	$BrF_3 + UF_6$(b)	145	175	18
49	$BrF_3 + Br_2$ (c)	135	170	11
50	$BrF_3 + Br_2$ (d)	150	160	13
51	$BrF_3 + Br_2$ (e)	135	160	14
52	$BrF_3 + Br_2$ (f)	145	160	7
PART B- Uranium exposed to vapor without prior liquid immersion:				
105	BrF_3	25	190	68
106	BrF_3	25	190	70

(a) BrF_3 inadvertently distilled into cold trap; ignition produced by addition of more BrF_3 at 205°.
(b) Initial solution = 10.3 mole per cent UF_6.
(c) Initial solution = 9.9 mole per cent Br_2.
(d) Initial solution = 4.5 mole per cent Br_2.
(e) Initial solution = 17 mole per cent Br_2.
(f) Initial solution = 16 mole per cent Br_2.

PHOTOGRAPHS OF URANIUM IGNITING IN BROMINE TRIFLUORIDE VAPOR

Enlargement approximately 3X.

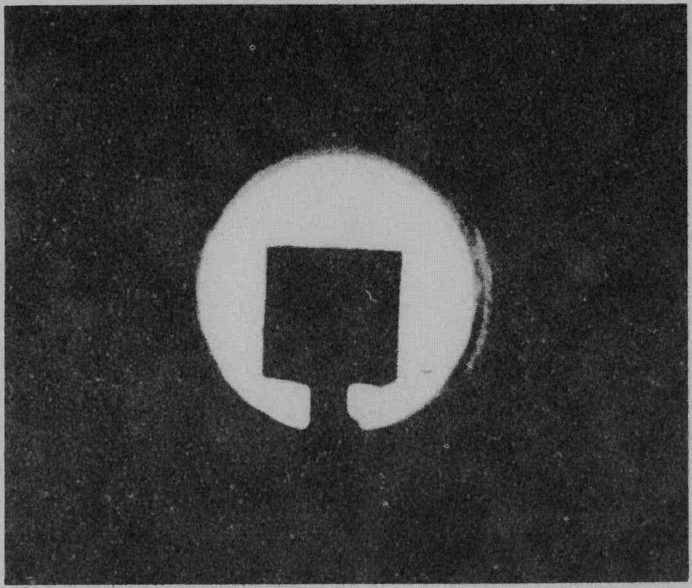


Figure 5a, zero time

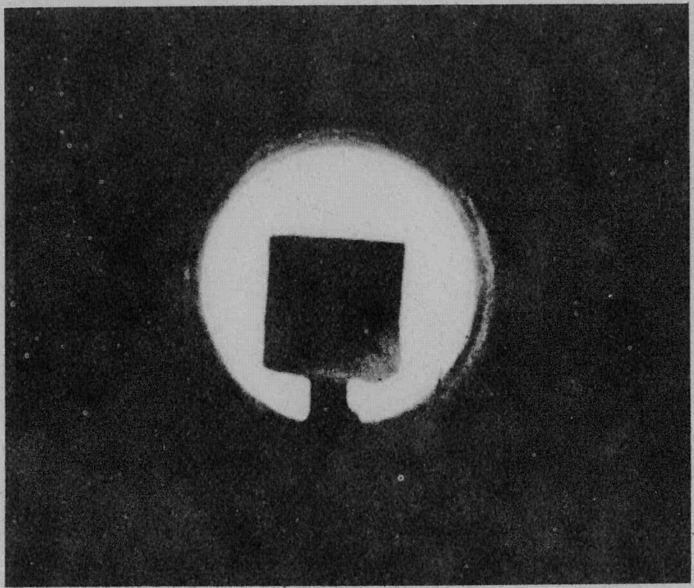


Figure 5b, 0.8 second

PHOTOGRAPHS OF URANIUM IGNITING IN BROMINE TRIFLUORIDE VAPOR

Enlargement approximately 3X.

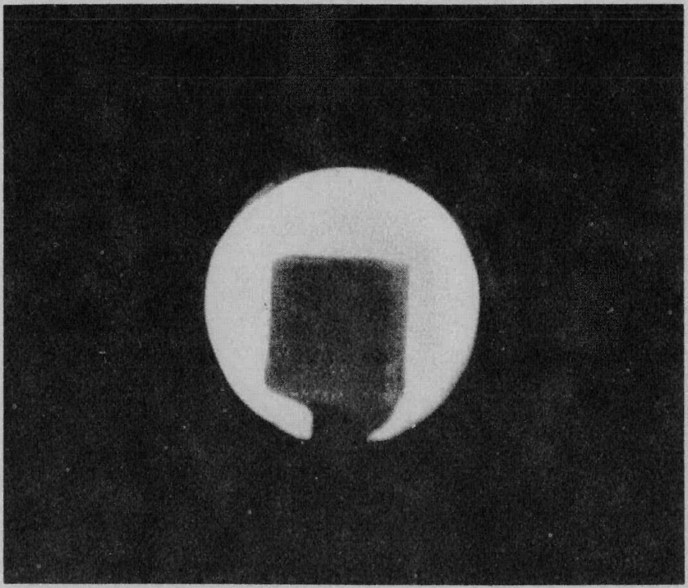


Figure 5c, 2.0 seconds

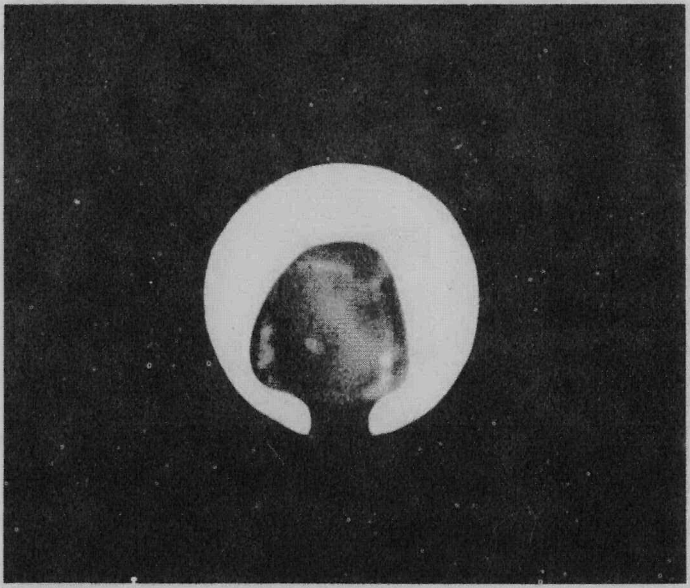


Figure 5d, 2.7 seconds

PHOTOGRAPHS OF URANIUM IGNITING IN BROMINE TRIFLUORIDE VAPOR

Enlargement approximately 3X.

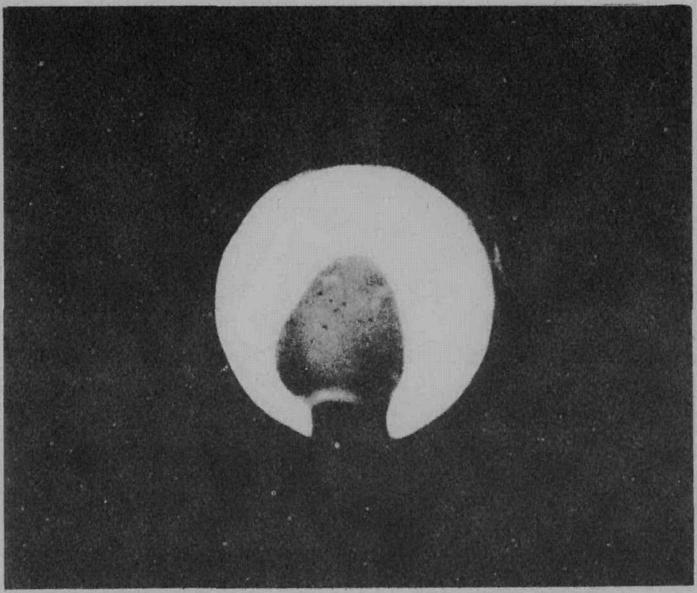


Figure 5e, 3.0 seconds

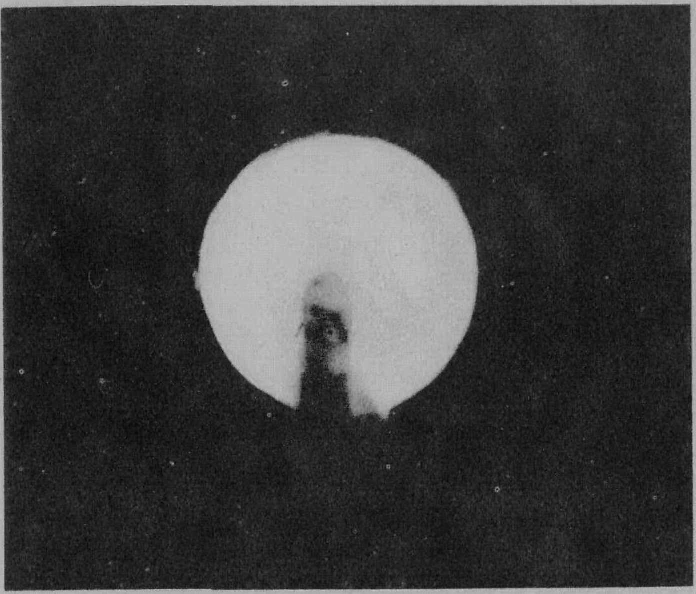


Figure 5f, 3.9 seconds

Uranium was found to ignite in vapor-phase reactions with bromine pentafluoride, chlorine trifluoride, and fluorine, as shown in Table 3. The minimum temperatures at which ignitions were produced in these reagents were 225°, 205°, and 260°C, respectively. During an ignition the pressure usually rose slightly, then dropped to a final value only two or three per cent below the initial value.

The pressures listed in column 3 were measured just prior to the ignitions. In the experiments with bromine pentafluoride, the vapor exposures were begun by draining off the liquid at 160° or 170°C. In run 41 with chlorine trifluoride, the liquid was drained off at 70°C. In the other experiments with chlorine trifluoride, the uranium was exposed to vapor throughout the heating period as the liquid beneath the uranium was vaporized. In runs 44, 45, and 46 the uranium was heated from room temperature in fluorine, but in run 47, the fluorine was introduced at 180°C.

The lowest ignition temperature of uranium in chlorine trifluoride vapor (205°C) differed but little from that in bromine trifluoride vapor (190°C) in the experiments in which the uranium was not first wetted by liquid. The pressures were of the same magnitude in these experiments; the vapor pressure of bromine trifluoride at the ignition point in runs 105 and 106 was approximately 4370 mm, and the pressure of chlorine trifluoride in run 40 was approximately 2800 mm. The ignition temperatures did not vary systematically with pressure in any single reagent, as indicated by the large scatter of the data for bromine pentafluoride, chlorine trifluoride, and fluorine. Since the experiments were done in a static system, reaction products could accumulate in the vicinity of the uranium and hinder further fluorination. It seems probable that in a flow system lower ignition temperatures would be found.

Melting of uranium slugs in reactions with fluorine was reported in earlier work at Oak Ridge.(5) The present experiments indicate that this can occur very suddenly and at temperatures as low as 260°C in the presence of excess fluorine. The heat of formation of uranium hexafluoride is -505 kcal/mole,(6) so it is not surprising that the temperature rise is abrupt once the reaction proceeds at a rapid rate. Approximately 155 calories are required to raise 3 grams of uranium from room temperature to the melting point and to supply the heat of fusion, and the consumption of only 9×10^{-4} gram-mole of fluorine in the formation of uranium hexafluoride will provide the required heat. Approximately one hundred times this amount of fluorine was present in runs 45, 46, and 47.

Poor heat removal is probably the biggest factor causing uranium to ignite in all of the vapor-phase reactions. In a dissolution of uranium in a liquid halogen fluoride considerable heat may be evolved, but the liquid serves as a heat transfer medium and prevents the temperature from rising abruptly. If the liquid is at the boiling point, heat can be removed by

Table 3

IGNITION OF URANIUM METAL IN VAPOR-PHASE FLUORINATION

Approximately 3 grams of alpha-rolled, reactor-grade uranium in each experiment.

Free volume of apparatus = 600 ml

Run No.	Reagent	Approximate Pressure at Ignition (mm)	Initial U Temp (°C)	Ignition Temp (°C)	Vapor Contact Time (min)
33	BrF_5	5500	170	240	20
34	BrF_5	5300	170	225	7
35	BrF_5	>6000	160	225	21
36	ClF_3	1250	25	no ignition(a)	50
37	ClF_3	2550	25	270	45
38	ClF_3	750	25	no ignition(b,c)	33
39	ClF_3	2000	25	255	29
40	ClF_3	2800	25	205	24
41	ClF_3	5750	70	235	31
42	ClF_3	3600	25	270	28
43	ClF_3	750	25	275(b)	35
44	F_2	900	25	no ignition(d)	52
45	F_2	4600	25	260	24
46	F_2	4800	25	355	33
47	F_2	4900	180	375	36

(a) Temperature surged from 330° to 410° upon reducing pressure at end of run.

(b) ClF_3 from cylinder allowed to flow over heated U at atmospheric pressure.

(c) Run terminated at 205°.

(d) Run terminated at 340°.

vaporization as well as by conduction to the walls. In a vapor-phase reaction, however, the rate of dissipation of heat from the uranium is much lower, and the rising temperature accelerates the reaction until the ignition point is reached.

C. Thorium and Materials of Construction, Liquid and Gaseous Reagents.

The test exposures of thorium and materials of construction are listed in Table 4. All of the test specimens were 1/4-inch cubes except the platinum, which consisted of 1-gram pieces of 10-mil foil wrapped around the tip of the needle thermocouple. In the vapor-phase experiments, the liquid beneath the specimen was usually vaporized completely before the final temperature was reached; the maximum pressure is therefore given in column 4.

Thorium, aluminum, copper, iron, and magnesium were very little attacked by any of the reagents. The 1/4-inch cubes usually lost or gained a few milligrams as the result of corrosion or surface fluorination.

Platinum readily dissolved in hot bromine trifluoride and was not tested further.

Zirconium was only slightly attacked by liquid bromine trifluoride and bromine pentafluoride but proved to be unstable in the vapor-phase reactions. The test specimens ignited in vapor-phase reactions with chlorine trifluoride and fluorine. In one experiment with bromine trifluoride vapor, no noticeable changes occurred, but in all subsequent vapor exposures the zirconium became heavily coated with a thick white scale, and rapid temperature surges were recorded.

Fluorothene ignited in vapor-phase reactions with chlorine trifluoride and fluorine. Although it did not ignite in the vapors of bromine trifluoride and bromine pentafluoride, the number of experiments was too small to establish this with certainty. It probably cannot be considered safe for use with the latter two reagents above 125°C.

The Teflon gaskets used to seal the windows of the cell did not react noticeably below 200°C, but at higher temperatures they occasionally caught fire in the fluorinating agents. Fluorothene and Teflon, as well as other fluorocarbons, are known to be thermodynamically less stable than carbon tetrafluoride,(7) which might be one of the ultimate products of vigorous fluorination.

The nickel, Monel, and Inconel parts of the apparatus were stable in all of the experiments. The brass parts were slightly corroded but remained serviceable.

Table 4

EXPOSURE OF THORIUM AND MATERIALS OF CONSTRUCTION TO FLUORINATING REAGENTS

Duration of Exposures = 10-30 minutes.

Test Specimen	Reagent	Maximum Temperature (°C)	Maximum Pressure (mm)	Results
Th	BrF_3 liquid	165	-	A
Th	BrF_3 vapor	365	2000	A
Th	BrF_5 vapor	340	2100	A
Th	ClF_3 vapor	350	4300	A
Th	F_2 gas	340	3000	A
Al	BrF_3 liquid	225	-	A
Al	BrF_3 vapor	395	>2000	A
Al	BrF_5 liquid	120	-	A
Al	BrF_5 vapor	340	2100	A
Al	ClF_3 vapor	340	5700	A
Al	F_2 gas	340	4700	A
Cu	BrF_3 liquid	225	-	A
Cu	BrF_3 vapor	410	2300	A
Cu	BrF_5 liquid	100	-	A
Cu	BrF_5 vapor	365	2700	A
Cu	ClF_3 vapor	310	5700	A
Cu	F_2 gas	380	7000	A
Fe	BrF_3 liquid	170	-	A
Fe	BrF_3 vapor	410	4600	A
Fe	BrF_5 liquid	130	-	A
Fe	BrF_5 vapor	350	3000	A
Fe	ClF_3 vapor	340	4900	A
Fe	F_2 gas	360	6000	A
Mg	BrF_3 liquid	225	-	A
Mg	BrF_3 vapor	375	>2000	A
Mg	BrF_5 liquid	130	-	A
Mg	BrF_5 vapor	350	2700	A
Mg	ClF_3 vapor	340	3400	A
Mg	F_2 gas	340	4000	A
Zr	BrF_3 liquid	145	-	A
Zr	BrF_3 vapor	380	>2000	A
Zr	BrF_3 vapor	370	1500	B
Zr	BrF_5 liquid	120	-	A
Zr	BrF_5 vapor	370	2000	B
Zr	ClF_3 vapor	340	5200	C
Zr	F_2 gas	340	2600	C
Pt	BrF_3 liquid	150	-	D
Pt	BrF_3 liquid	205	-	E
Pt	BrF_3 vapor	400	4000	A
Fluorothene	BrF_3 liquid	150	-	A
Fluorothene	BrF_3 vapor	310	2300	A
Fluorothene	BrF_5 vapor	310	5200	A
Fluorothene	ClF_3 vapor	275	4200	C
Fluorothene	F_2 gas	330	3500	C

(A) Little or no reaction; test specimen unchanged except for small loss or gain in weight.

(B) Erratic reaction; occasional temperature surges; formation of thick fluoride coating.

(C) Ignition of test specimen at stated maximum temperature.

(D) Test specimen partly dissolved.

(E) Test specimen completely dissolved.

The quartz windows were etched by the halogen fluorides. However, they remained transparent while wetted with liquid and could be used for a number of experiments before replacement became necessary.

ACKNOWLEDGEMENT

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