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PREPARATION OF URANIUM HEXAFLUORIDE FROM URANIUM TETRAFLUORIDE USING BROMINE TRIFLUORIDE PREPARED IN SITU

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Date of Issue: May 7, 1953

Report Number: K-1012

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PREPARATION OF URANIUM HEXAFLUORIDE FROM URANIUM TETRAFLUORIDE
USING BROMINE TRIFLUORIDE PREPARED IN SITU

E. B. Olszewski

Laboratory Division
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A B S T R A C T

A low temperature system for the continuous production of uranium hexafluoride from uranium tetrafluoride and/or the oxides of uranium using bromine trifluoride produced in situ as the fluorinating agent is described. Fluorine was added continuously to regenerate the reacted bromine trifluoride. Complete conversion to uranium hexafluoride was obtained at the maximum obtainable feed rate of 3.3 kg. of uranium tetrafluoride per hour. At feed rates of 0.44 to 2.14 kg. of uranium tetrafluoride per hour, fluorine efficiencies from 98.9 to 99.7 per cent were obtained. A method for the separation of uranium hexafluoride from the reaction products using crystallization in a cold water condenser is described.

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PREPARATION OF URANIUM HEXAFLUORIDE FROM URANIUM TETRAFLUORIDE
USING BROMINE TRIFLUORIDE PREPARED IN SITU

INTRODUCTION

The work of Vogel and Vogel (6) has demonstrated that; 1) the reaction rate of bromine trifluoride on metallic uranium is appreciable in the presence of hydrogen fluoride or bromine, 2) uranium hexafluoride can be separated from bromine trifluoride by distillation, and 3) nickel, copper and Fluorothene are suitable as materials of construction for use with bromine trifluoride and its reaction products. Aluminum is also suitable as a material of construction (3).

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Further qualitative studies have indicated that the reaction of bromine trifluoride with uranium tetrafluoride or the uranium oxides to produce uranium hexafluoride proceeded rapidly and completely even at room temperature, and the reaction of gaseous bromine with gaseous fluorine to produce bromine trifluoride was rapid and controllable.

These findings indicated that a continuous, low temperature system could be designed for preparing uranium hexafluoride using bromine trifluoride as the fluorinating agent and uranium tetrafluoride or the uranium oxides as feed. The reaction of bromine trifluoride with the uranium compound to produce uranium hexafluoride and bromine, and the reaction of the bromine with fluorine to produce bromine trifluoride could be carried out simultaneously in the same reaction vessel, and would allow the use of fluorine as a raw material rather than bromine trifluoride.

The work described herein is divided into three parts: the operation of a two inch diameter batch reactor; the development of a water cooled condenser for separation of uranium hexafluoride; and the operation of a three inch diameter continuous reactor.

EXPERIMENTAL

I. The Two Inch Batch Reactor

Apparatus. The first system, constructed of welded nickel and called the Two Inch Batch Reactor, (figure 1), consisted of a reactor tube, a cold trap, and a gas circulating pump. The reactor tube, a 2-inch diameter nickel pipe, contained both the liquid reactor and the gas reactor. The liquid reactor, occupying the bottom third of the reactor tube, consisted of a percolator operated by the circulating gas for agitating the liquid, a liquid drain line leading to an evacuated tank, and an outside liquid level indicator constructed of Fluorothene. The gas reactor, occupying the upper two thirds of the reactor tube, consisted of a fluorine injector system, a gas reaction zone, and reaction zone observation ports. The fluorine injector system consisted of a valved fluorine inlet line feeding a shower head type gas injector. The reaction zone observation ports, located both above and below the gas injector, contained transparent Fluorothene windows through which the color of the gas in the reaction zone could be observed. The gases from the gas reactor passed to the carbon dioxide slush cooled cold trap for removal of the condensables.

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TWO INCH BATCH REACTOR

FIGURE 1

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The gases from the cold trap were recirculated by a dual action floating piston type pump driven by external magnets (5).

Operating Procedure. Before each run the feed powder and the bromine were placed in the liquid reactor and the gas circulating pump started.

Fluorine was introduced into the system where it reacted with the bromine to produce bromine trifluoride which condensed in the cooler upper portion of the reactor tube and drained into the liquid reactor where it reacted with the feed powder to produce uranium hexafluoride and bromine. The uranium hexafluoride and bromine vapors were carried by both the circulating gas and diffusion to the gas reactor where the bromine reacted with fluorine to produce bromine trifluoride while the uranium hexafluoride remained unchanged. Since bromine trifluoride and uranium hexafluoride are colorless gases while bromine is red, the fluorine flow was so adjusted that the color of the gas below the injector remained red while the gas above the injector remained colorless. Since these conditions would be altered by either an excess of fluorine or an insufficient amount of fluorine, it was possible to adjust the fluorine flow to satisfy stoichiometric requirements. The gases from the gas reaction zone were carried by the circulating gas into the cold trap where uranium hexafluoride and the bromine trifluoride condensed. The uranium hexafluoride could subsequently be separated from the bromine trifluoride by fractional distillation. The effluent gases from the cold trap condenser were circulated through the percolator back into the reactor tube by the magnetic pump.

Results. At room temperature using gram mole quantities of uranium tetrafluoride, uranous oxide and uranic oxide as feed materials, the following operational results were obtained.

1. The reaction of the feed powder with bromine trifluoride was rapid, complete, and controllable.
2. The reaction of gaseous bromine with gaseous fluorine was also rapid and controllable. Since no increase in system pressure due to bromine pentafluoride was apparent, any bromine pentafluoride produced during this reaction either reacted with the feed material or with bromine (2).
3. The gas operated percolator provided sufficient agitation to prevent caking of reacting powder.
4. Since the heat of reaction of uranium oxides was greater than the combined heat of vaporization of uranium hexafluoride and bromine, while the heat of reaction of uranium tetrafluoride was less, the liquid reactor temperature increased when using uranium oxides as feed and decreased when using uranium tetrafluoride as feed.
5. Copper impregnated Teflon seats in Kerotest valves failed on exposure to bromine trifluoride, while copper seats were satisfactory.
6. Monel Hoke valves failed frequently during exposure to liquid bromine trifluoride, but only rarely during exposure to gaseous bromine

trifluoride. Failure was due to corrosion of the silver solder on the diaphragm.

7. The nickel and Fluorothene parts of the equipment showed no visible evidence of corrosion.
8. Condensation of bromine trifluoride in the magnetic pump caused erratic pumping with frequent stoppages.
9. Bromine was found as one of the contaminants in the uranium hexafluoride. Since the vapor pressures of bromine and uranium hexafluoride were almost identical at the melting point of uranium hexafluoride (3), fractional distillation as a means for separating pure uranium hexafluoride was impractical.

Although these results indicated that the continuous production of uranium hexafluoride by this method was feasible, it was also obvious that the development of a method for the purification of the uranium hexafluoride was necessary.

II. Development of the Water Cooled Condenser

Preliminary Work. Since the separation of uranium hexafluoride from bromine by fractional distillation was impractical, while its separation from bromine trifluoride by distillation was simple (3), a study of the fluorination of the bromine to bromine trifluoride either prior to or during distillation was made. Because bromine was not completely fluorinated or was produced by reaction of bromine trifluoride on the equipment, pure uranium hexafluoride was not separated and further work with fractional distillation was discontinued.

During operation of the Two Inch Batch Reactor, white uranium hexafluoride crystals had been observed on the sight glass below the fluorine inlet when the reactor tube was cold. Since this crystallization occurred in the presence of bromine and appeared to be free of bromine color, the possibility of using crystallization as a method for obtaining pure uranium hexafluoride was investigated.

Apparatus. The apparatus consisted of a feed cylinder, a condenser, and a vacuum system protected by a cold trap and a soda-lime trap. The feed cylinder was constructed of nickel and contained a mixture of uranium hexafluoride and bromine. The condenser, constructed of nickel, consisted of a vertical tube, jacketed on the outside for water cooling and contained a feed inlet tube, located near the bottom, a liquid drain line at the bottom and an evacuation line at the top (figure 2). A Fluorothene sight glass was placed in the liquid drain line to permit observation of the color of the liquid. Copper seated Kerotest valves were used on all lines.

Operational Procedure. The apparatus was operated in the following manner. The feed cylinder was heated with resistance heaters to transfer the feed material as a vapor to the condenser. With the condenser wall temperature maintained at approximately 20°C., the vapor, containing uranium hexafluoride, bromine, and bromine trifluoride, entered the condenser near the bottom. The uranium hexafluoride condensed as a solid on the

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FIGURE 2

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condenser wall while both the bromine and bromine trifluoride condensed as liquids. The liquid drained to the bottom of the condenser and was withdrawn leaving a cylinder of solid uranium hexafluoride adhering to the condenser wall. The bromine and bromine trifluoride vapors remaining in the condenser along with some of the uranium hexafluoride were removed by intermittent flashing into an evacuated cold trap.

Results. Product containing 98.9 per cent uranium hexafluoride was obtained from feed material containing 93 per cent uranium hexafluoride. Although this product when solidified was colored red, the color could be removed by further flashing, indicating that the purity of the uranium hexafluoride was dependent upon the operating technique used. In subsequent studies of improved operating techniques, product containing more than 99.99 per cent uranium hexafluoride was obtained (4). Since this method for separating uranium hexafluoride from bromine and bromine trifluoride was successful, it was incorporated in the three inch reactor.

III. The Three Inch Continuous Reactor

Apparatus. Constructed of 3-inch nickel pipe and called the Three Inch Continuous Reactor, (figure 3), this system consisted of the following units: a bromine trifluoride reservoir, an ash receiver, a liquid reactor, a gas reactor, a powder feed system, a water cooled condenser, a vent system, and a gas circulating pump. The bromine trifluoride reservoir, a welded nickel tank, contained a liquid level indicator, a valved connection to either vacuum or pressurized nitrogen, and a valved line extending from the bottom of the tank into the liquid reactor-ash receiver connecting tube. The ash receiver, a Fluorothene tube closed at the bottom and threaded at the top, was attached to the liquid reactor. The liquid reactor contained a gas operated percolator, a Fluorothene liquid level indicator, a bromine trifluoride return line, and was flange-connected to the gas reactor. The gas reactor contained a powder inlet tube, a fluorine injector system, illuminated reaction zone observation ports extending through the tube, a Bourdon gauge, and a gas exit tube leading to the water cooled condenser. The powder feed system, flange-connected to the powder inlet tube, consisted of a powder feed hopper and a motor-driven screw feed. The water cooled condenser was described in the previous section. The gas exit line from the condenser led to both a bellows sealed piston type positive displacement B-4 pump (1) and a vent system. The exit line from the B-4 pump was connected to the percolator in the liquid reactor. The vent system, used to vent inert gases to atmosphere, consisted of a carbon dioxide-slush cooled cold trap followed by a soda-lime trap.

In order to regulate the temperature of the apparatus, the following facilities were used. Hot or cold water, circulated through coils of copper tubing wrapped around both the liquid reactor and the gas reactor, was used to control the temperature of these units. The gas exit line connecting the gas reactor to the condenser, the bottom of the condenser, and the bromine trifluoride return line were heated with Nichrome resistance heating wire. Thermocouples, inserted in appropriately placed thermocouple wells, were used to measure the temperature of the reactor tube. Weston thermometers were used to measure the circulating water temperature.

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THREE INCH CONTINUOUS REACTOR

FIGURE 3

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Operating Procedure. Preliminary to actual operation, the following preparation was necessary. The condenser cooling water was turned on. The reactor tube temperature regulating water was turned on, adjusted to and maintained at 70°C. The wire wrapped lines were heated to about 80°C. The feed powder was placed in the feed hopper, and bromine trifluoride was transferred from the bromine trifluoride reservoir to the liquid reactor using pressurized nitrogen on the reservoir. The observation port lights were turned on.

The operation involved the following steps. The gas inlet valve and the gas outlet valve were opened. The B-4 pump was started, causing gas to circulate through the percolator into the liquid reactor, through the reactor tube into the condenser, through the condenser, and back into the B-4 pump. This circulating gas flow was regulated, by a valve on the inlet end of the B-4 pump, to produce visible evidence of flow indicated by a rise and fall in the bromine trifluoride level observed in the liquid reactor level glass. The screw feed was started and adjusted to give the predetermined powder flow. The powder was carried into the bottom of the gas reactor, dropped into the liquid bromine trifluoride where it reacted to produce uranium hexafluoride and bromine. As the vapors from this reaction were carried into the gas reaction zone, the fluorine was introduced into the system, and its flow regulated as described in the Two Inch Batch Reactor procedure. The gases from the gas reactor, containing uranium hexafluoride and bromine trifluoride, passed into the water cooled condenser where the hexafluoride condensed as a solid and adhered to the wall while the bromine trifluoride condensed as a liquid and drained into the bottom of the condenser. The bromine trifluoride liquid was drained back to the liquid reactor through the bromine trifluoride drain line. The B-4 pump carried the gases from the top of the condenser back to the reactor percolator. When the system pressure reached or exceeded atmospheric due to the accumulation of oxygen, nitrogen or hydrogen fluoride, the vent system to atmosphere was opened and allowed to remain open during the duration of a run. Since the vent gases contained small amounts of uranium hexafluoride and bromine trifluoride as well as inert gases, a cold trap was used to remove and store the uranium hexafluoride and bromine trifluoride for subsequent recovery. The soda-lime trap was used to react with any fluorine vented.

In order to achieve continuous operation, two interchangeable condensers were necessary. When one condenser was filled with uranium hexafluoride, it was isolated from the system and emptied while the other condenser was filling. A condenser was emptied by using either vapor transfer or liquid transfer techniques. In either case, the condenser was converted from a water cooled condenser to a water or steam heated cylinder by substituting hot water or steam for the cold water. The uranium hexafluoride was transferred to and stored in a suitable storage cylinder.

Results and Discussion. Using a 3/8-inch diameter screw feed, a series of test runs were made with a variety of feed materials. Operational data obtained during these runs are tabulated in table I. Using uranium tetrafluoride or the oxides of uranium as feed and feed rates ranging from 100 g. to a maximum of 600 g. per hour, continuous operation with complete conversion to uranium hexafluoride was obtained.

To test the capacity of this reactor, a 3/4-inch diameter screw feed was

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TABLE I
OPERATIONAL DATA USING 3/8-INCH SCREW FEED

Run	Material Fed	Rate of Feed, g./hr.	Duration Of Run, hrs.	Remarks*
1	UF ₄	375	4	
2	UO ₃	100	1.5	
3	UO ₂	333	3	
4	U ₃ O ₈	360	2.5	Intermittent operation due to screw plugging
5	UF ₄	600	7	
6	U ₃ O ₈ + UF ₄	500	6	
7	U ₃ O ₈	500	4	
8	20% U ₃ O ₈ , 80% UF ₄	350	14	Two 7 hour runs
9	UO ₃	300	6	

*Complete conversion to UF₆ was obtained in all runs.

substituted for the 3/8-inch screw and additional runs were made using uranium tetrafluoride as feed material. Operational data are tabulated in table II. The maximum powder feed rate obtained with the 3/4-inch screw was 3.3 kg./hr. of uranium tetrafluoride (run 10). Even at this maximum feed rate, fluctuations in powder feed rate caused immediate proportional fluctuations in color intensity of the gas as seen in the observation ports indicating that feed powder was reacting completely and almost instantaneously.

During runs 17 through 20, data dealing with amounts and analysis of vented gas were obtained from which fluorine efficiencies and condenser efficiencies were calculated. These data and calculated efficiencies are tabulated in table III. The calculated fluorine efficiency ranged from 98.9 to 99.7 per cent while the overall condenser efficiency ranged from 98.0 to 99.6 per cent. Sample calculations are included in the table.

A flow sheet with material balance data is presented in figure 4. This material balance was calculated by using data obtained in run 17 and the following estimations: 1) the gas circulation rate was estimated by assuming that the rate and amplitude of oscillations of the liquid bromine trifluoride as observed in the liquid reactor level glass was proportional to and caused by displacement of liquid in the reactor by circulating gas; 2) the hydrogen fluoride balance was estimated by assuming that the fluorine used contained approximately 2 per cent hydrogen fluoride and that all the hydrogen fluoride introduced into the system was vented to the chemical trap; 3) the mixture of bromine, bromine trifluoride, and bromine pentafluoride was calculated as bromine trifluoride. With the exception of the hydrogen fluoride balance, these estimations and assumptions have no real effect on the materials added and materials

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TABLE IIOPERATIONAL DATA USING 3/4-INCH
SCREW FEED AND URANIUM TETRAFLUORIDE FEED

Run No.	Feed Rate, kg. UF ₄ / hour	Total Mols UF ₄	Duration Of Run	Average Temperatures*, °C.			Remarks
				UF ₄ Liquid Reactor	Br ₂ -F ₂ Gas Reactor	Exit End Of Gas Reactor	
10	3.30	17.3	1 hr. 40 min.	70	125	95	Insufficient F ₂
11	1.24	18.4	4 hrs. 45 min.	75	125	75	Insufficient F ₂
12	2.60	30.5	3 hrs. 45 min.	65	140	80	Insufficient F ₂
13	2.60	4.6	35 min.	70	---	--	F ₂ leak
14	2.60	2.0	15 min.	70	---	--	Plug at exit of reactor
15	2.63	33.0	4 hrs.	70	150	95	BrF ₃ return plugged
16	2.62	31.4	3 hrs. 53 min.	70	150	100	None
17	2.14	10.8	1 hr. 35 min.	75	160	95	Leak at sight glass
18	0.44	4.1	2 hrs. 57 min.	70	110	40	None
19	1.64	13.2	2 hrs. 33 min.	75	140	80	None
20	1.45	11.6	2 hrs. 35 min.	45	140	80	None

*Condenser temperature for all runs was 28°C.

TABLE III
FLUORINE AND CONDENSER EFFICIENCIES

Run Number	17	18	19	20
% Fluorine Efficiency	98.9	99.7	99.7	99.3
% Condenser Efficiency	98.0	99.0	98.1	99.6
UF ₄ Feed Rate: Kg. per hour	2.14	0.44	1.64	1.45
Gram mols per hour	6.82	1.40	5.23	4.62
Standard liters UF ₆ produced per hour	153	31	117	103
Calculated standard liters F ₂ required per hour	158	32	121	106
Analysis of Gas Vented: % Br ₂ + BrF ₃ + BrF ₅	18.3	15.2	44.6	24.5
% F ₂	8.1	2.9	1.9	5.9
% UF ₆	13.1	7.7	9.6	2.6
% Inerts by difference	60.5	74.2	43.9	67.0
Liters of Gas Vented Per Hour: Inerts measured	13.7	2.8	10.5	9.2
UF ₆ calculated	3.0	0.3	2.2	0.4
F ₂ calculated	1.8	0.1	0.4	0.8
BrF ₅ + Br ₂ + BrF ₃ calculated	4.1	0.6	10.3	3.4

Analysis of UF₆ used

% UF ₄	97.63	Calculated M.W. = 313.8
% UO ₂	0.7	
% UO ₂ F ₂	1.67	
% U	75.87	

Sample Calculations

$$\text{Liters UF}_6 \text{ vented} = \frac{\text{liters inerts vented} \times \% \text{ UF}_6 \text{ vented}}{\% \text{ inerts vented}} = \frac{13.7 \times 13.1}{60.5} = 3.0$$

$$\% \text{ F}_2 \text{ efficiency} = \frac{\text{liters F}_2 \text{ required}}{\text{liters F}_2 \text{ required} + \text{liters F}_2 \text{ vented}} \times 100 = \frac{158}{158 + 1.8} \times 100 = 98.9$$

$$\% \text{ Condenser efficiency} = \frac{\text{liters UF}_6 \text{ produced} - \text{liters UF}_6 \text{ vented}^*}{\text{liters UF}_6 \text{ produced}} \times 100 = \frac{153 - 3.0}{153} \times 100 = 98.0$$

*UF₆ is recovered and returned to the system.

LEGEND:

LR --- Liquid Reactor
 GR --- Gas Reactor
 C --- Condenser
 T --- UF_6 Storage Tank
 P --- B-4 Pump
 CT --- Cold Trap
 ChT --- Chem Trap
 FH --- Feed Hopper
 A --- Ash Receiver

REACTIONS:

FLOW SHEET AND MATERIAL BALANCE OF CONTINUOUS REACTOR

FIGURE 4

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withdrawn. In the case of the hydrogen fluoride, an accumulation of hydrogen fluoride would have been apparent as an increase in volume of liquid bromine trifluoride; this was not observed. The absence of alpha activity in the ash substantiated the completeness of conversion to uranium hexafluoride.

CONCLUSIONS AND RECOMMENDATIONS

Using nickel, Monel, copper and Fluorothene as materials of construction, a system was developed to produce uranium hexafluoride on a continuous basis using uranium tetrafluoride or the oxides of uranium as feed material and bromine trifluoride produced in situ as the fluorinating agent. At feed rates up to 3.3 kg. per hour, conversion of the feed material to uranium hexafluoride was complete. Fluorine efficiencies higher than 98 per cent were obtained. Extended continuous operation of this system would require equilibrating the fluorine flow to the powder feed flow, and periodically removing the uranium hexafluoride produced. A method for separating the uranium hexafluoride from the reaction products using a cold water condenser was developed.

The efficient and complete fluorination of the uranium compounds tested to uranium hexafluoride obtained in this study indicates that this method could be adapted for large scale production of uranium hexafluoride. The absence of uranium in the ash suggests the use of this method with facilities for ash removal for fluorination of impure uranium tetrafluoride or oxides. Further studies with larger equipment should be made for the purpose of obtaining operational costs with a variety of feed materials.

ACKNOWLEDGEMENTS

Acknowledgement is given to the following. Mr. H. H. Lunn who assisted in the design and construction of the equipment; Messrs. G. J. Vogel and S. D. Schiffman who did the fractional distillation; Mr. H. H. Lett who assisted in operating the equipment during the final phases; and Mr. J. Dykstra who provided space and assisted in the procurement of materials.

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