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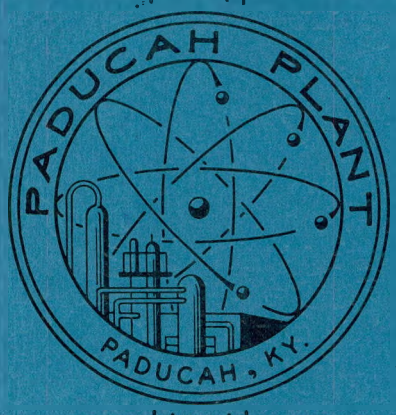


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UNION CARBIDE NUCLEAR COMPANY
A Division of Union Carbide and Carbon Corporation
PADUCAH PLANT
Paducah, Kentucky

[redacted]

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## ABSTRACT

Three materials, $UO_3$, $U_3O_8$ and $UF_4$, have been tested for their ability to absorb or react with low concentrations of $UF_6$ in the presence of large amounts of fluorine and air. It was found that at 400°F a fluidized bed of $UF_4$ will react with the $UF_6$ and that $UF_6$ in the amount of 10% of the weight of the $UF_4$ can be reacted before detectable $UF_6$ is found in the gas leaving the reactor.

[redacted]

# RECOVERY OF URANIUM HEXAFLUORIDE FROM VENT GASES

The vent gases from the $UF_6$ cold traps in the Paducah Feed Processing Plant necessarily contain a small percentage of $UF_6$ due to the vapor pressure of this material at the temperatures involved. The average composition of these vent gases is in the range of 0.1 mole % $UF_6$, 3 mole % $F_2$ and 15 mole % HF with the remainder nitrogen and oxygen. Reducing the quantity of $UF_6$ lost can be accomplished in several ways mechanically and by chemical absorption by various compounds. However, it appeared that recovery of $UF_6$ in a fluidized bed of powder such as $UO_3$, $U_3O_8$ or $UF_4$ would possess several inherent advantages. Among these are simplicity of design and production of a material suitable for fluorination reactor feed. The intent of this investigation was to determine the feasibility of recovering this $UF_6$ by absorbing or reacting it with a uranium compound in a fluidized bed.

## CONCLUSIONS

The amount of $UF_6$ that reacted with $U_3O_8$ was too small to be of significant value. $UO_3$ reacted with 6% of its weight of $UF_6$ from a mixture of air and $UF_6$ at 600°F. $UF_4$ reacted with 15% of its weight of $UF_6$ from a mixture of air and $UF_6$. This value was reduced to 10% in the presence of 3 mole % fluorine. Two different types of $UF_4$ were tested, Mallinckrodt and Paducah, and no significant difference was found.

## EXPERIMENTAL

### Apparatus

The powder under consideration was heated and fluidized in a 2 inch monel tube reactor (Figure I). A porous nickel plate supported by a drilled plate was used to distribute the fluidizing air. Metered $UF_6$ and fluorine were admitted to the system by mixing the gases with the fluidizing air stream. The off gases from the reactor were filtered through a porous nickel filter. Sample points were located on the inlet and outlet gas lines.

### Materials

The $UO_3$ used was unground Hanford continuous calcined powder designated SHS-10. The $U_3O_8$ was made from this $UO_3$ by heating the $UO_3$ at 850°C for 16 hours. Two types of $UF_4$ were used. Type I was metal grade D-38 $UF_4$ from Mallinckrodt. Type II $UF_4$ was material produced on "B" tray in the Paducah Feed Plant. This $UF_4$ was made from a shipment of sulfated Hanford pot calcined $UO_3$ received August 25, 1956. Chemical and physical data for these powders are shown in Table No. 1.

### Procedure

The system used was a batch process. The powder was charged to the bed, fluidized, and heated to the desired temperature. In all cases a static

powder bed height of approximately 14 inches was used. $UF_6$ and/or fluorine were then admitted to the fluidizing air at the proper concentrations. Inlet and outlet gas samples were analyzed at regular intervals to determine the inlet and outlet concentrations of $UF_6$ and $F_2$. The amount of $UF_6$ absorbed by the bed was determined by the weight loss of the $UF_6$ feed cylinder.

## DISCUSSION

The per cent $UF_6$ and per cent $F_2$ recovered at various temperatures by the different compounds are given in Tables No. 2 and No. 3, respectively. The capacities of the compounds for the absorption of $UF_6$ are shown in Table No. 4. The temperatures for determining the capacities of the various compounds were chosen to optimize the recovery of $UF_6$ and minimize the recovery of $F_2$.

The first powder studied was $UO_3$. It was the intent that the mixture of $UO_3$ - $UO_2F_2$ formed might be fed directly to the fluorine towers by blending this material with green salt. However, the data indicate that a relatively large amount of $UO_3$ would be needed for the recovery unit. This quantity of $UO_3$ - $UO_2F_2$ could not be handled in the existing process.

$U_3O_8$ was investigated next, with the theory that the $U_3O_8$ could be reclaimed in the reactor by pyrohydrolyzing the $U_3O_8$ - $UO_2F_2$ mixture. The ability of $U_3O_8$ to recover $UF_6$ under the conditions studied was so small that the process was deemed not to be feasible.

The data with $UF_4$ indicate that a fluidized bed of $UF_4$ would be capable of recovering $UF_6$ in the presence of fluorine and air. No attempt was made to determine the reaction products. However, the black color of the reaction products and the relatively high dissociation pressure of $UF_5$ at 400°F lead to the theory that the reaction products of $UF_4$ and $UF_6$ under these conditions are $U_2F_9$ and/or $U_4F_{17}$. Mixtures of these compounds could be fed directly to the fluorine towers.

There was no evidence that these intermediates formed in any part of the system other than in the bed. At all times the powder in the bed remained free flowing, and no evidence of any plugging in the bed, filter or outlet lines was found.

While the preliminary data indicate that a fluidized bed of $UF_4$ would be capable of recovering the $UF_6$ in the feed plant cold trap vent gases, additional information should be obtained. Some of the factors requiring further study include the effects of flow rates, HF, variations in gas composition, particularly large concentrations of $F_2$ and $UF_6$, $UF_4$ purity and composition, and the determination of the reaction products.

Table No. 1

PHYSICAL & CHEMICAL DATA FOR $UF_4$

| | Paducah ($UF_4$II) | Mallinckrodt ($UF_4$I) |
|---|---|---|
| % $UF_4$ | 78.1 | >99 |
| % $UO_2$ | 11.5 | < 1 |
| % $UO_2F_2$ | 10.4 | < 0.2 |
| Sieve Analysis, % Passing: | | |
| Sieve No. 40 | 99.2 | 99.5 |
| 60 | 95.4 | 99.0 |
| 80 | 92.5 | 98.2 |
| 100 | 89.5 | 92.9 |
| 200 | 81.7 | 90.7 |
| 325 | 69.1 | 76.0 |
| Density, g/cc.: | | |
| Free Flow | 2.8 | 3.0 |
| Packed | 3.6 | 4.2 |

Table No. 2

RECOVERY OF $UF_6$ BY VARIOUS URANIUM COMPOUNDS

| Type Powder | % $UF_6$ Recovered At 100°F | 200°F | 400°F | 600°F | 800°F | Fluidizing Air Velocity, ft/sec. |
|---|---|---|---|---|---|---|
| $UO_3$ | - | 37 | 45 | 99 | 93 | 1.0 |
| $U_3O_8$ | - | - | 56 | 88 | 97 | 1.0 |
| $UF_4$ (I) | 91 | 90 | 98 | - | - | 0.4 - 0.5 |

Table No. 3

RECOVERY OF $F_2$ BY VARIOUS URANIUM COMPOUNDS

| Type Powder | % $F_2$ Recovered At 100°F | 200°F | 400°F | 600°F | 800°F | Fluidizing Air Velocity, ft/sec. |
|---|---|---|---|---|---|---|
| $UO_3$ | - | 6 | 17 | 50 | 36 | 1.0 |
| $U_3O_8$ | - | - | 4 | 56 | 96 | 1.0 |
| $UF_4$ (I) | 30 | 33 | 50 | - | - | 0.4 - 0.5 |

Table No. 4

CAPACITY FOR THE RECOVERY OF $UF_6$
BY VARIOUS URANIUM COMPOUNDS

| Type Powder | Lbs. $UF_6$ Per Lbs. Powder* | Temp. Determined | Fluidizing Air Velocity, ft/sec. | Fluidizing Gas Constituents |
|---|---|---|---|---|
| $UO_3$ | 0.06 | 600°F | 1.0 | Mixture of air and ~0.1 mole % $UF_6$. |
| $U_3O_8$ | <0.030 | 800°F | 1.0 | Mixture of air and ~0.1 mole % $UF_6$. |
| $UF_4$ (I) | 0.15 | 400°F | 0.2 | Mixture of air and 0.1 - 0.7 mole % $UF_6$. |
| $UF_4$ (I) | 0.10 | 400°F | 0.2 | Mixture of air, 0.15 mole % $UF_6$ and 3 mole % $F_2$. |
| $UF_4$ (II) | 0.10 | 400°F | 0.2 | Mixture of air, 0.36 mole % $UF_6$ and 3 mole % $F_2$. |

*Before the recovery efficiency dropped below 95%.

Figure I

# Fluidized Bed Apparatus