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TITLE

REPORT OF INVENTION - THE PREPARATION OF PLUTONIUM (IV) AMMONIUM FLUORIDE AND ITS DECOMPOSITION TO PLUTONIUM TETRA-FLUORIDE FOR REDUCTION TO METAL.

AUTHOR

W. B. Tolley
R. C. Smith

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RICHLAN[redacted] WASHINGTON

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REPORT OF INVENTION

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A.E.C. CASE NO.

G. E. CASE NO. HW 12-6755

TO: W. H. Clymer

[redacted]

I: ATTACHED HERETO IS A DESCRIPTION OF WHAT MAY BE AN INVENTION IN:

The use of ammonium bifluorido as a fluorinating agent for conversion of plutonium dioxide to a plutonium ammonium tetrafluoride double salt, which can be decomposed to plutonium tetrafluoride for subsequent reduction to metal.

This document consists of 2 pages. No. 2 of 4 copies.

II: THE NAME, TITLE OR POSITION, WORKS LOCATION, AND PERMANENT ADDRESS OF THE INVENTOR(S) IS:

Willis B. Tolley, Junior Engineer, Hanford Atomic Products Operation, General Electric Company, Richland, Washington.

Robert C. Smith, Chemist II, Hanford Atomic Products Operation, General Electric Company, Richland, Washington.

III: EVIDENCE AS TO WHEN AND WHERE THE INVENTION WAS MADE CAN BE FOUND IN THE FOLLOWING LISTED WRITTEN OR PICTORIAL MATERIAL (NOTEBOOK, FILE REPORTS OR DRAWINGS, ETC.):

HW-5515-T (Secret Notebook) p 115-118 - Willis B. Tolley.
HW-4541-T (Secret Notebook) p 15 - Robert C. Smith

IV: THE APPROXIMATE DATE OF THE FIRST ENTRY IN SAID WRITTEN OR PICTORIAL MATERIAL DESCRIBING OR SHOWING SAID INVENTION IS:

November 2, 1953 - Willis B. Tolley
November 14, 1951 - Robert C. Smith

SPECIAL RE-REVIEW
FINAL DETERMINATION
DECLASSIFICATION CONFIRMED
BY A E Baulan DATE 6-28-82
BY [signature] DATE 6-28-82

V: PERSONS WHO COULD TESTIFY AS TO WHEN AND WHERE THE INVENTION WAS MADE INCLUDE THE FOLLOWING:

William H. Reas
William E. Roake

PM Eick 3-17-98
P. Sullivan 5-18-98

RESTRICTED DATA
This document [redacted] data as defin[redacted] Act of 194[redacted] transmitta[redacted] disclosure of [redacted] contents in [redacted] manner [redacted] unauthorized per[redacted] is prohibited.

RECEIVED

SIGNED (SUPERVISOR)	DATE	DEPARTMENT
JUL 9 1956 [signature]	2-26-54	Engineering

NOTE: SUGGESTIONS FOR PREPARING THE INVENTION DESCRIPTION ARE CONTAINED ON THE REVERSE SIDE OF THIS FORM.

[redacted] DECLASSIFIED [redacted]

This is a report of what may be an invention in the use of ammonium bifluoride for the preparation of a plutonium ammonium fluoride double salt which can be decomposed at low temperature to plutonium tetrafluoride for purposes of reduction to the metal. Ammonium bifluoride has been found to react with dry plutonium dioxide at 150 C to produce the plutonium (IV) ammonium fluoride double salt. This compound can be decomposed at 300 C to give a high grade plutonium tetrafluoride. The use of this reaction to produce a plutonium tetrafluoride which can be reduced to the metal may reduce considerably the corrosion now experienced during the fluorination with gaseous hydrogen fluoride in 234-5 operation.

In practice on a nominal twenty gram scale, the optimum method for the preparation of plutonium tetrafluoride by the reaction of plutonium dioxide with ammonium bifluoride has been found to be as follows: freshly precipitated plutonium (IV) oxalate is placed in a reaction vessel and dried in a stream of air at 125 to 150 C for one hour and the temperature raised to 300 C for two hours to decompose the plutonium (IV) oxalate to plutonium dioxide. Forty percent excess ammonium bifluoride is mixed with the plutonium dioxide and heated in a stream of argon for one hour at 150 C. The double salt, plutonium (IV) ammonium fluoride, which forms rapidly at 150 C is decomposed at 300 C for one and one-half hours under argon. The ammonium fluoride decomposition product is easily trapped while the plutonium remains in the reaction boat as an easily powdered tetrafluoride. The plutonium tetrafluoride is then dehydrated for two hours at 500 C. Since the temperature need never exceed 500 C while fluoride is present the corrosion to furnace equipment should be low.

Analyses have shown that the plutonium tetrafluorides prepared in this manner are approximately 95 percent converted from the dioxide. Preliminary investigations have indicated that they can be reduced with calcium using the bomb method with with high yields on a twenty gram scale.

William B Tolley 2-25-54 Robert Elmich 2-25-54

Product Metallurgy
Technical Section
ENGINEERING DEPARTMENT

WB Tolley:rd

WITNESSES

Read and understood by me this 26 day of February, 1954.

William E. Roake
Product Metallurgy
Technical Section
ENGINEERING DEPARTMENT

Read and understood by me this 26 day of February, 1954.

William H. Reis
Product Metallurgy
Technical Section
ENGINEERING DEPARTMENT