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UNCLASSIFIED

HW-26022

UNITED STATES ATOMIC ENERGY COMMISSION

**REPORT OF INVENTION ON THE
PREPARATION OF PLUTONIUM
TRIFLUORIDE**

By
L. L. Burger
W. E. Roake

CLASSIFICATION CANCELLED

DATE 1/28/60

For The Atomic Energy Commission

H. F. Canale
Chief, Declassification Branch

October 22, 1952

Hanford Works
Richland, Washington

Technical Information Extension, Oak Ridge, Tennessee

AEC RESEARCH AND DEVELOPMENT REPORT

UNCLASSIFIED

846-601

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NUCLEONICS DIVISION
GENERAL ELECTRIC COMPANY
RICHLAND, WASHINGTON

ent consists of

No. 9

Series 4

A.E.C. CASE NO.

G. E. CASE NO.

REPORT OF INVENTION

TRANSMITTAL CONTROL NUMBER

TO: J. W. Underwood, 703 Building, 700 Area

HANFORD 52073

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

The preparation of plutonium trifluoride, for reduction to metal, by means of a solid phase - gas reaction between plutonium IV oxalate or plutonium III oxalate and Freon-type compounds such as dichloro-difluoro-methane (Freon-12), the dichloro-difluoro-methane also acting as a reducing agent when reacting with plutonium IV oxalate.

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

L. L. Burger, Chemist
Hanford Works
General Electric Company
Richland, Washington

W. E. Roake, Chemist
Hanford Works
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.):

Notebook HW-2765-T, Pages 96-97
Notebook HW-2774-T, Pages 91-102, 115-121

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

December 19, (1949) (HW-2765-T)
December 21, (1951) (HW-2774-T)

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

W. H. Reas
R. C. Smith

SIGNED (SUPERVISOR)

DATE

DEPARTMENT

J. W. Underwood

10/24/52

Engineering

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

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SUGGESTIONS FOR PREPARING THE INVENTION DESCRIPTION

- I. ATTACH TO THIS REPORT SHEET A DESCRIPTION OF THE INVENTION WHICH IS SUFFICIENTLY DETAILED SO THAT A PERSON CAPABLE OF UNDERSTANDING THE INVENTION CAN, FROM A READING OF THE DESCRIPTION, PLUS ANY ATTACHMENTS THERETO, LEARN WHAT THE INVENTION TEACHES AND HOW IT IS MADE AND USED. THE DESCRIPTION NEED NOT BE A DETAILED ENGINEERING SPECIFICATION GIVING PRECISE DIMENSIONAL DETAILS FOR EVERY ELEMENT, ETC., BUT IT SHOULD GIVE THE READER THE ESSENTIAL COMPONENT PARTS OF THE INVENTION AND PARTICULARLY IS THIS TRUE IF A CERTAIN FEATURE, SUCH AS A MATERIAL, TEMPERATURE, PRESSURE, ETC., IS CRITICAL. TO AVOID NEEDLESS FOLLOW - UP, THE READER OF THE DESCRIPTION SHOULD BE ABLE TO GET ENOUGH INFORMATION FROM READING THE DESCRIPTION, PLUS ANY ATTACHMENTS THERETO, THAT HE CAN COMPARE THE INVENTION WITH OTHER PRACTICES IN THE SAME FIELD. IT IS OF PARTICULAR IMPORTANCE TO MENTION WHETHER THE SUBJECT MATTER OF INVENTION IS OR IS NOT UNDER DEVELOPMENT OR IN ACTUAL USE; OR THAT SUCH USE IS CONTEMPLATED. WHEREVER POSSIBLE, DRAWINGS, SKETCHES, OR PHOTOGRAPHS, ETC., SHOULD BE USED TO LESSEN THE AMOUNT OF WRITTEN DESCRIPTION REQUIRED.
- II. THE INVENTION DESCRIPTION PLUS ANY ATTACHMENTS THERETO SHOULD BE SIGNED AND DATED BY THE INVENTOR(S) IN THE PRESENCE OF AT LEAST ONE WITNESS WHO UNDERSTANDS THE INVENTION. THE WITNESS(ES) SHOULD ALSO SIGN AND DATE THE DESCRIPTION AND ATTACHMENTS. THE FULL FIRST NAME, MIDDLE INITIAL AND FULL LAST NAME SHOULD BE USED BY BOTH THE INVENTOR(S) AND WITNESS(ES).
- III. USE OF REGULAR REPORTS AS INVENTION DESCRIPTIONS:
 1. IF A REPORT PREPARED IN THE REGULAR COURSE OF WORK, SUCH AS A HANFORD TECHNOLOGY REPORT, SATISFIES THE REQUIREMENTS STATED IN I. ABOVE, THEN IT WILL NOT BE NECESSARY TO PREPARE A SEPARATE INVENTION DESCRIPTION, INSTEAD, MERELY ATTACH A COPY OF SUCH REPORT TO THIS REPORT SHEET AND SIGN IT AS DIRECTED IN II. ABOVE.
 2. SUCH A REPORT CAN STILL BE USED AS THE BASIS OF AN INVENTION DESCRIPTION IF IT IS POSSIBLE TO SUPPLEMENT IT READILY SO THAT THE REPORT PLUS THE SUPPLEMENT MEETS THE REQUIREMENTS STATED IN I. ABOVE. IN SUCH A CASE, ATTACH THE REPORT PLUS THE SUPPLEMENTARY MATERIAL TO THIS REPORT SHEET AND SIGN THEM AS DIRECTED IN II. ABOVE.
 3. IN CASES OTHER THAN THOSE DESCRIBED ABOVE, PREPARE A SEPARATE INVENTION DESCRIPTION AND ATTACH IT TO THIS REPORT SHEET.
- IV. AN ORIGINAL AND SIX COPIES OF THIS REPORT SHEET PLUS THE INVENTION DESCRIPTION ATTACHED THERETO SHOULD BE FORWARDED AS INSTRUCTED IN ORGANIZATION AND POLICY GUIDE 12.1.

Richland, Washington
October 22, 1952

This is a report of what may be an invention in the use of Freon type compounds such as dichloro-difluoro-methane (Freon-12) as a reducing agent and as a fluorinating agent for the preparation of plutonium trifluoride for purposes of reduction to metal. Dichloro-difluoro-methane has been found to react with dry plutonium III oxalate at 400°-450°C. to produce plutonium trifluoride. Dichloro-difluoro-methane has been found to react with dry plutonium IV oxalate at 400°-450°C. to produce plutonium trifluoride, the plutonium IV being reduced to plutonium III in the course of the reaction. The use of the latter reaction to produce a plutonium compound which can be directly reduced to metal eliminates the use of highly corrosive reagents, both for the reduction of plutonium IV to plutonium III and for the fluorination of the plutonium compound. Plutonium trifluoride prepared in this manner has a bulk density greater than that prepared by hydrofluorination, which is a desirable characteristic for bomb reduction. Plutonium trifluoride prepared by the reaction with dichloro-difluoro-methane has been found to be reduced to metal with good yield (92.6% yield on nominal ten gram scale) by reaction with calcium by the bomb method.

In practice, on a nominal one gram scale, the optimum method of preparation of plutonium trifluoride by reaction with dichloro-difluoro-methane has been found to be as follows: Freshly precipitated plutonium II oxalate slurry is dried to a cake by heating in a platinum or nickel vessel in a stream of Freon-12, argon or nitrogen for one hour at 100°C. Bound water is removed by further heating for one hour at 225°C. in the gas stream. Oxygen must be excluded. Reaction is induced by heating to and holding at 400°-425°C. for one or more hours in a stream of dichloro-difluoro-methane. Commercially available Freon-12 is used without further purification. The product, plutonium trifluoride, is cooled to below 100°C. before exposing to air or oxygen.

Substitution of the described method of preparation of plutonium trifluoride for reduction to metal in place of the presently used hydrofluorination method is contemplated.

W. C. Burger 77 Oct 52
Wm. S. Roeder 10/27/52
W. H. Kees 10/27/52
R. C. Smith 29 Oct 52
846-004