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DP - 434
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Nuclear Technology - Chemistry and Chemical Engineering
AEC Research and Development Report

OXIDATION OF NEPTUNIUM(V) BY VANADIUM(V)

by

E. K. Dukes

Separations Chemistry Division

November 1959

[redacted]

E. I. du Pont de Nemours & Co.
Savannah River Laboratory
Aiken, South Carolina

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DP - 434

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(M-3679, 24th Ed.)

[redacted] Number 28

OXIDATION OF NEPTUNIUM(V) BY VANADIUM(V)

by

Ernest K. Dukes

November 1959

Classification Cancelled or Changed
TO C U D
By Authority of
J. W. Kahn, USAEC, 4-19-62
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ABSTRACT

Rate data are presented for the oxidation of neptunium(V) by vanadium(V) in nitric acid.

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CONTENTS

LIST OF FIGURES

OXIDATION OF NEPTUNIUM(V) BY VANADIUM(V)

INTRODUCTION

In extending the studies of the oxidation-reduction behavior of neptunium reported by Siddall and Dukes[1] it was found that vanadium(V) rapidly oxidized neptunium(V) to neptunium(VI). Preliminary experiments showed that the oxidation occurred in solutions of nitric acid if the ratio of vanadium(V) to vanadium(IV) were large and if the concentration of neptunium were low. This report describes the results of the study of rates of reaction between neptunium(V) and vanadium(V) in nitric acid solutions.

SUMMARY

Rates of oxidation of neptunium(V) were determined by a solvent extraction technique based on the difference in distribution coefficients for neptunium(V) and neptunium(VI). The rate expression was

$$\frac{d[Np(VI)]}{dt} = K_1[Np(V)][H^+]^2[VO_2^+]$$

where $K_1 = 11.7 \pm 2.5$ mols^{-3} min^{-1} at 24°C

The activation energy for the reaction was 11.7 ±0.4 kcal/mol between 24 and 50°C.

Plutonium(IV) was not oxidized to an appreciable extent under the conditions used for the oxidation of neptunium(V).

DISCUSSION

EXPERIMENTAL PROCEDURE

Rate data for the reaction between neptunium(V) and vanadium(V) were obtained by the following procedure. An aliquot of almost pure Np(V) was added to nitric acid of the desired concentration and a portion of the solution was extracted with 30% tributyl phosphate (TBP) in n-dodecane at 2°C to determine the amount of Np(VI) present initially with the Np(V). Vanadium(V) was added to the remainder of the solution, and aliquots of the reaction mixture were extracted with TBP at regular intervals to follow the progress of the reaction. After extraction with TBP, the amount of neptunium in each phase was determined by counting the alpha activity, and the concentration of Np(VI) was calculated from the equation

$$Np(VI) = C_o + \frac{C_a}{E\ o/a}$$

where C_o = concentration of Np in organic phase

C_a = concentration of Np in aqueous phase

E o/a = distribution coefficient for pure Np(VI) under identical conditions of extraction

The analysis for Np(VI) is based on the difference in extraction properties of Np(V) and Np(VI). Neptunium(VI) is extracted readily from solutions of nitric acid by 30% TBP in n-dodecane (Figure 1), while the distribution coefficient for Np(V) is essentially zero[1].

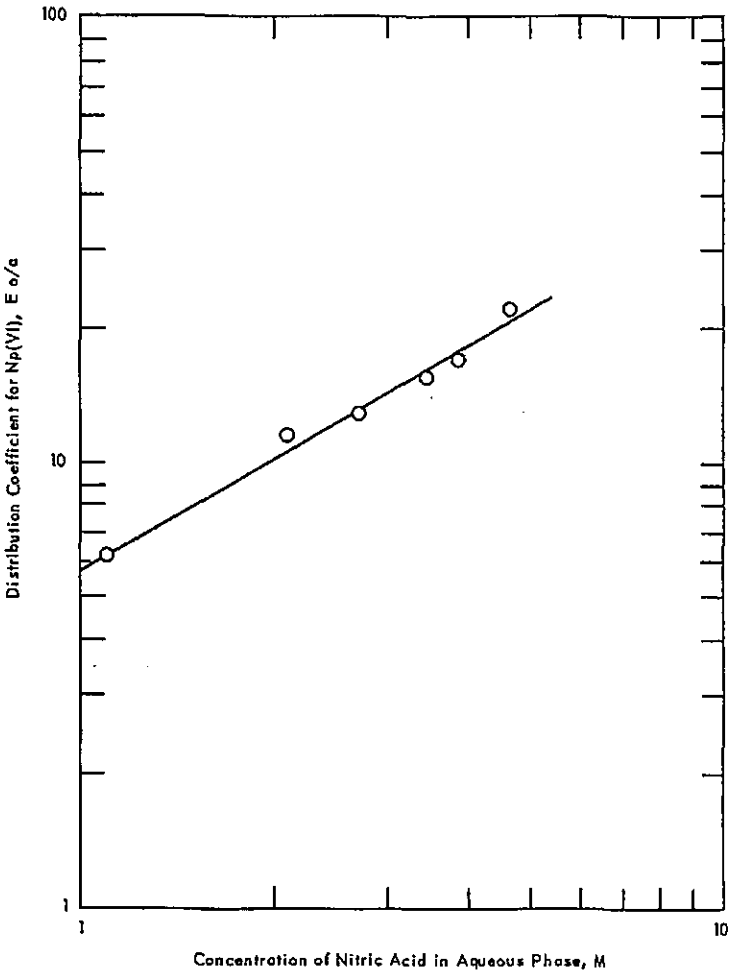

FIGURE 1 – DISTRIBUTION OF Np(VI) AT 2°C

PREPARATION OF REAGENTS

NEPTUNIUM(V) SOLUTION

A solution of Np^{237} in dilute nitric acid was adjusted to the (V) valence state by reduction of Np(VI) with nitrous acid. Sodium nitrite (0.01M) was added to a solution of 0.5M HNO_3 that was mixed with an equal volume of 30% TBP in n-dodecane that contained Np(VI). The solutions were mixed until most of the neptunium was reduced and extracted into the aqueous phase. Fresh 30% TBP in n-dodecane was then mixed with the aqueous phase to remove traces of Np(VI) and nitrous acid. A low distribution coefficient (0.01-0.05) showed that Np(V) was the predominant species.

VANADIUM(V) STOCK SOLUTION

A stock solution of V(V) was prepared from vanadium pentoxide (C.P. grade). The pentoxide was dissolved in sodium hydroxide, and nitric acid was added so that the final concentration of acid was about 3.0M. The stock solution was analyzed by the method described by Scott[2].

PLUTONIUM(IV) SOLUTION

Plutonium(IV) was absorbed from 8M HNO_3 on "Dowex" 21K resin, and was eluted with 0.5M HNO_3. An aliquot of this solution was made 3M in HNO_3 and was then extracted with 30% TBP in n-dodecane to verify that plutonium was present in the (IV) state.

OTHER MATERIALS

The methods used for the purification of Np^{237}, Np^{239}, Np(VI), tributyl phosphate, and n-dodecane were described by Siddall and Dukes[1].

TREATMENT OF DATA

The rate of the reaction between Np(V) and VO_2^+ depends on the concentration of H^+ and VO_2^+ as well as the concentration of Np(V). Both VO_2^+ and H^+ are present in excess and may be presumed to be constant. The reaction goes to equilibrium, so that simple first-order kinetics cannot be used. The reverse reaction between V(IV) and Np(VI) has to be considered; therefore, the reaction is treated as a first-order process opposed by a second-order process.

These kinetics are treated by Laidler[3] for the case where the initial concentrations of the products are zero. In this study Np(V) was not entirely free of Np(VI) so that the treatment described by Laidler was modified to take into account the initial concentration of Np(VI).

The general equation describing the reaction is

$$\frac{d[Np(VI)]}{dt} = K_1[Np(V)] - K_{-1}[Np(VI)][V(IV)]$$

It is assumed that all of the V(IV) formed is due to the reduction of V(V) by Np(V). The equation then becomes

$$\frac{dx}{dt} = K_1(N - x) - K_{-1}x(x - x_o) \qquad (1)$$

where x = concentration of Np(VI) at time (t)

x_o = concentration of Np(VI) at time (o)

N = total concentration of Np

Equation (1) can be written in the form

$$\frac{dx}{dt} = K_1(N + bx + cx^2) \qquad (2)$$

and constants can be evaluated in terms of x_e (the value of x at equilibrium) and other known quantities:

$$c = \frac{x_e - N}{x_e^2 - x_e x_o}$$

$$b = -(x_o c + 1)$$

The solution to equation (2) with initial conditions $x = x_o$ at $t = o$ is:

$$\frac{-2}{\sqrt{-q}} \tanh^{-1} \frac{2cx + b}{\sqrt{-q}} = \frac{-2}{\sqrt{-q}} \tanh^{-1} \frac{2cx_o + b}{\sqrt{-q}} + Kt \qquad (3)$$

where $$q = 4Nc - b^2$$

The first term in equation (3) was plotted against time, and K, the rate constant, was determined from the slope of the straight line. A typical plot is shown in Figure 2.

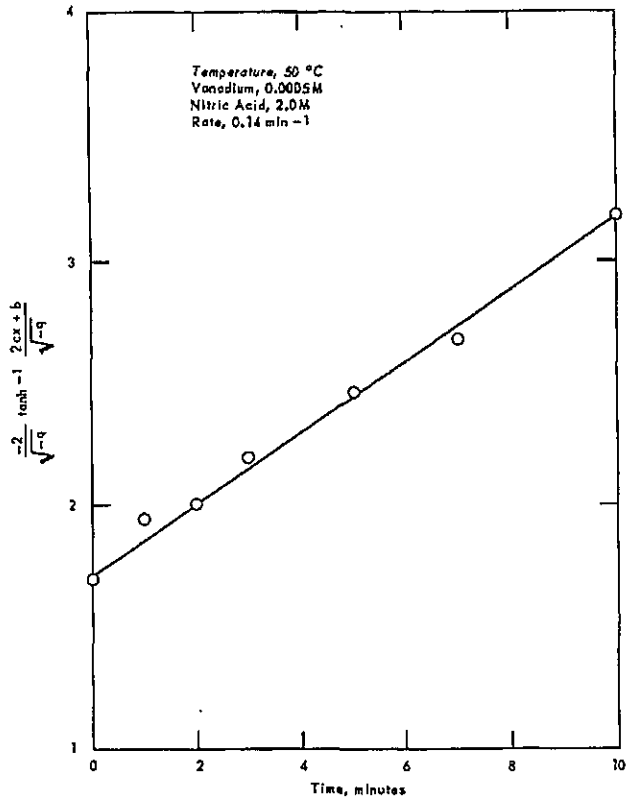

FIGURE 2 - TYPICAL RATE CURVE

RESULTS

RATE STUDIES

The reaction between tracer concentrations of Np(V) and VO_2^+ was first order with respect to neptunium. Rates of oxidation were almost the same at 10^{-5} and 10^{-12}M neptunium as shown in the following table.

Effect of the Concentration of Neptunium

2.8M HNO_3 24°C 0.0005M VO_2^+

Np Conc., M	Rate of Oxidation, min^{-1}
1×10^{-5} (Np^{237})	0.041
1×10^{-12} (Np^{239})	0.034

Rates of oxidation depended on the first power of the concentration of VO_2^+ and the second power of the concentration of HNO_3. Rates are shown as a function of the concentration of VO_2^+ in Figure 3 and as a function of the concentration of HNO_3 in Figure 4.

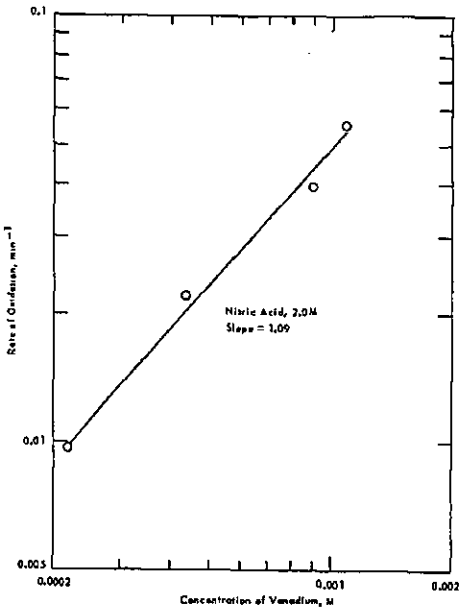

FIGURE 3 – EFFECT OF CONCENTRATION OF VANADIUM ON OXIDATION OF Np(V)

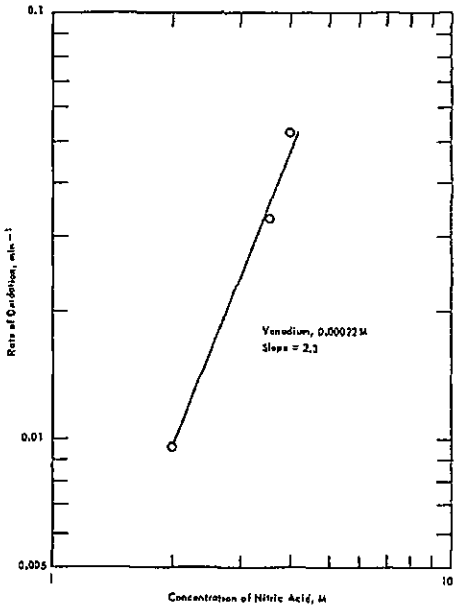

FIGURE 4 – EFFECT OF CONCENTRATION OF NITRIC ACID ON OXIDATION OF Np(V)

The power dependence of 2 on the concentration of HNO_3 was due entirely to the hydrogen ion. An experiment was performed to compare rates of oxidation in HNO_3 with rates in a mixture of HNO_3 and $NaNO_3$. For this experiment Np^{239} was used because the alpha activity of Np^{237} could not be counted accurately in the presence of sodium. The results are shown in the following table.

Effect of Nitrate Ion on the Oxidation of Np(V)

HNO_3, M	$NaNO_3$, M	NO_3^-, M	Rate, min^{-1}
2.66	2.0	4.66	0.036
5.20	-	5.29	0.127
2.78	-	2.78	0.033

The preceding results were consistent with the rate expression

$$\frac{d[Np(VI)]}{dt} = K_1[Np(V)][H^+]^2[VO_2^+]$$

The rate constant, K_1, was calculated from several experiments at 24°C where the concentration of H^+ ranged from 2.04 to 3.58M and the concentration of VO_2^+ ranged from 2×10^{-4} to 1.1×10^{-3}M. The value for K_1 was 11.7 ±2.5 $mols^{-3}$ min^{-1}.

EFFECT OF TEMPERATURE

The activation energy for the oxidation in 2M HNO_3 was 11.7 ±0.4 kcal/mol between 24 and 50°C, with two different concentrations of VO_2^+. The effect of temperature on the rate of oxidation is shown in the following table, and the data for one experiment are presented graphically in Figure 5.

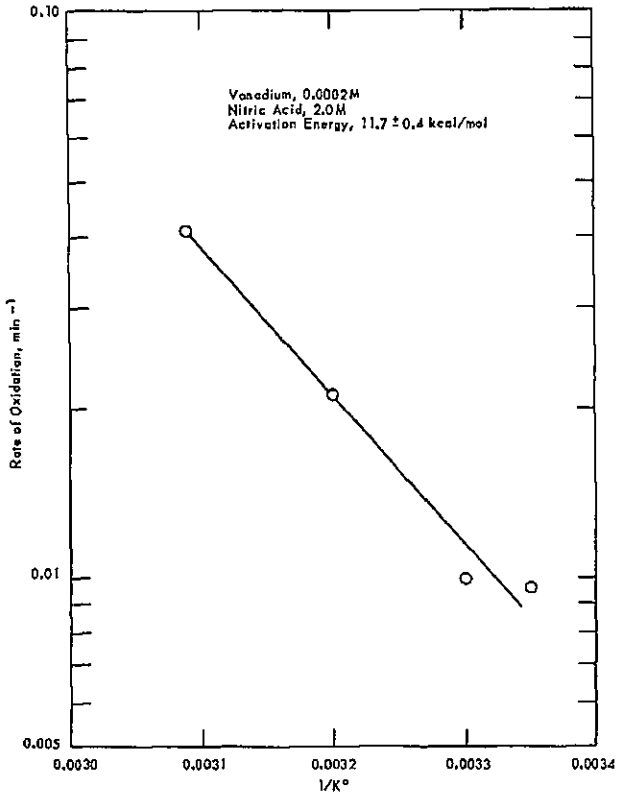

FIGURE 5 – EFFECT OF TEMPERATURE ON RATE OF OXIDATION OF Np(V)

Effect of Temperature on Rates of Oxidation

	Rate, min^{-1}	
Temp., °C	VO_2^+, 2 x 10^{-4}M	VO_2^+, 5 x 10^{-4}M
24	9.6 x 10^{-3}	2.2 x 10^{-2}
30	1.0 x 10^{-2}	5.4 x 10^{-2}
40	2.1 x 10^{-2}	7.2 x 10^{-2}
50	4.1 x 10^{-2}	1.4 x 10^{-1}

OXIDATION OF Pu(IV) BY VO_2^+

Oxidation-reduction potentials for the species of Np, Pu, and V that were involved in this study are

Np(V)	- Np(VI)	-1.15
Pu(IV)	- Pu(VI)	-1.10
V(V)	- V(IV)	1.00

The oxidation of Pu(IV) and Np(V) by VO_2^+ was determined under identical conditions. As shown below, VO_2^+ oxidized most of the Np(V) but only a small per cent of the Pu(IV).

Oxidation of Np(V) and Pu(IV) by VO_2^+

Sample	E o/a	% Oxidized
Np(V) (original solution)	0.1	-
Np(V) + VO_2^+ (30 min)	9.8	96
Np(VI) (pure)	15.5	-
Pu(IV) (original solution)	15.6	-
Pu(IV) + VO_2^+ (30 min)	14.3	3
Pu(VI) (pure)	3.0	-

Distribution coefficients, E o/a, between 3M HNO_3 and 30% TBP were used in the following equation[4] to calculate the per cent of Pu(IV) and Np(V) that was oxidized.

$$F_1 = \frac{(E_2 - E_S)(E_1 + 1)}{(E_S + 1)(E_2 - E_1)}$$

where E_1 = E o/a for first pure component

E_2 = E o/a for second pure component

E_S = E o/a for sample

E. K. Dukes

E. K. Dukes
Separations Chemistry Division

BIBLIOGRAPHY

1. Siddall, T. H., III, and Dukes, E. K.,"Kinetics of HNO_2 Catalyzed Oxidation of Neptunium(V) by Aqueous Solutions of Nitric Acid." J. Am. Chem. Soc. 81, 790-94 (1959).

2. Scott, W. W., Standard Methods of Chemical Analysis. Edited by N. H. Furman, Vol. I, 5th Edition. New York: Van Nostrand (1939).

3. Laidler, K. J., Chemical Kinetics. New York: McGraw-Hill (1954) p. 21.

4. Dukes, E. K.,"Kinetics and Mechanisms for the Oxidation of Trivalent Plutonium by Nitrous Acid." J. Am. Chem. Soc. 82, 9-13 (1960).

EX-1184

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WILMINGTON, DELAWARE

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H. K. Clark - Legal Dept.
Hood Worthington-C. W. J. Wende-L. C. Evans
S. A. McNeight-F. M. Burns
W. P. Overbeck-J. W. Morris
~~S. W. O'Rear~~
J. E. Beach
W File (2)

DP 434

See note below. 2) Ltr Erdley to Johnson

October 22, 1963

Mr. Randall G. Erdley, Chief (2)
Patent Branch
U. S. Atomic Energy Commission
Savannah River Operations Office
Post Office Box A
Aiken, South Carolina

Dear Mr. Erdley:

AEC CASE NO. S-28,523

The subject case pertains to the use of vanadium(V) for oxidizing neptunium(V), as described in DP-434. Your October 15 letter stated that the Commission will not file an application in this case and asked whether Du Pont is interested in filing. Du Pont is not.

Very truly yours,

ATOMIC ENERGY DIVISION

Hood Worthington, Director
Technical Division

Original signed by
By L. C. Evans
L. C. Evans

LCE:EV

bcc: S. W. O'Rear:

DP-434 was designated Official Use Only because of the Commission's interest in patentability. I assume that you will initiate a request for release. J. E. Beach has a copy of Erdley's October 15 letter.

L.C.E.

11/13

UNITED STATES
ATOMIC ENERGY COMMISSION
SAVANNAH RIVER OPERATIONS OFFICE
P. O. BOX A
AIKEN, SOUTH CAROLINA

TELEPHONE
AUGUSTA, GA.
PARK 4-6311

TELEGRAM ADDRESS
AUGUSTA, GA.

REFER TO: STC:TAB:mep

October 25, 1963

Mr. W. P. Overbeck, Director
Savannah River Laboratory
E. I. du Pont de Nemours & Company
Aiken, South Carolina

Attention: Dr. J. W. Morris, Director
Separations & Services Section

Dear Mr. Overbeck:

Our letter of September 5, 1962 notified you of declassification of the following report:

DP-434, "Oxidation of Neptunium(V) by Vanadium(V)"

Patent clearance for this report has now been received; accordingly, the Official Use Only marking should be removed from all copies.

Very truly yours,

Paul J. Hagelston, Director
Safety & Technical Services Division

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ATOMIC ENERGY COMMISSION
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AIKEN, SOUTH CAROLINA

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AUGUSTA, GA.

OR REPLY REFER TO:

October 15, 1963

Mr. Hood Worthington, Director
Technical Division, AED
E. I. du Pont de Nemours and Company
Wilmington 98, Delaware

Dear Mr. Worthington:

Subject: AEC CASE NO. S-28,523

A patentability search has been conducted in the subject case and it has been determined that, in view of the close prior art encountered and the limited project use for this development, no patent application will be filed on this development.

In view of this determination, there is no longer any objection from the Commission's standpoint to the release for publication of Report No. DP-434 to effect a statutory bar in the subject case.

Please advise whether or not Du Pont is interested in filing a patent application on this development.

Sincerely yours,

Randall G. Erdley, Chief
Patent Branch

cc: J. E. Beach, SRL

UNITED STATES
ATOMIC ENERGY COMMISSION
SAVANNAH RIVER OPERATIONS OFFICE
P. O. BOX A
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September 5, 1962

Mr. W. P. Overbeck, Director
Savannah River Laboratory
E. I. du Pont de Nemours & Company
Aiken, South Carolina

Attention: Dr. J. W. Morris, Director
Separations & Services Section

Dear Mr. Overbeck:

This will confirm conversations between Messrs. O'Rear and Niland of our respective staffs concerning report DP-434, "Oxidation of Neptunium(V) by Vanadium(V)", by E. K. Dukes.

You will recall that although this report was considered declassifiable at the time of its original review in 1959, it was released as Secret Restricted Data to enable its prompt distribution since patent clearance was not expected to be immediately forthcoming.

Subsequently the report was declassified without deletions by authority of Jack H. Kahn, Assistant Chief, Declassification Branch, Division of Classification, U.S. Atomic Energy Commission, effective April 19, 1962. Since patent clearance had not been secured we were instructed to handle the report as Official Use Only. This clearance has not yet been secured; however, it is suggested that notices of declassification be issued, citing the foregoing authority, with the Official Use Only proviso.

Very truly yours,

Paul J. Hagelston, Director
Safety & Technical Services Division

cc:

UNITED STATES
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October 8, 1962

Mr. W. P. Overbeck, Director
Savannah River Laboratory
E. I. du Pont de Nemours and Company
Aiken, South Carolina

Dear Mr. Overbeck:

Subject: REVIEW OF CERTAIN DOCUMENTS

Pursuant to our telephone conversation today with Mr. J. E. Beach, it will be appreciated if you would have the following Savannah River reports, which have been proposed for declassification, reviewed:

DP-198 - A Simple Leak Detector for Tritium

DP-434 - Oxidation of Neptunium(V) by Vanadium(V)

Toward facilitating the declassification action, your review of the above-mentioned reports for items of impressive technological novelty will be appreciated.

Very truly yours,

R. M. Poteat, Patent Engineer
Savannah River Patent Group

cc: Mr. Hood Worthington, Du Pont, Wilmington
Mr. J. E. Beach, Savannah River Laboratory

DP - 434-8

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TI:LAB:mcp

April 11, 1960

Mr. Hood Worthington, Director
Technical Division, AED
Explosives Department
E. I. du Pont de Nemours and Company
Wilmington 98, Delaware

Dear Mr. Worthington:

This letter will serve to confirm status of review for release of the following formal research and development reports:

DP-356, "An Automatic Gas Chromatograph for Monitoring of Reactor Fuel Failures - Part I - Design", by W. R. Kritz. Transmitted April 13, 1959, for classification and patent review for release to standard TID-4500 distribution.

There is no objection from the classification standpoint to release of this report; however, since patent approval for unclassified release has not yet been secured, external distribution of the document cannot be approved at this time.

DP-434, "Oxidation of Neptunium(V) by Vanadium(V)", by E. K. Dukes. Transmitted December 18, 1959, for classification and patent review for release to standard TID-4500 distribution.

This document is declassifiable, only, and should be handled as Secret, Restricted Data, until formal declassification is accomplished. Review by the Declassification Branch, Oak Ridge, is being held pending patent clearance. If you desire to make distribution of DP-434 at this time, it can be done so only on a classified basis.

The foregoing confirms conversations between Messrs. O'Rear and Niland of our respective staffs.

Very truly yours,

Paul J. Hagelston, Director
Technical & Production Division

cc: M. H. Wahl, du Pont Operations, SRP (2)
S. A. McNeight, du Pont, Wilmington

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AIKEN, SOUTH CAROLINA
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EXPLOSIVES DEPARTMENT
SAVANNAH RIVER LABORATORY

CC: F. A. Robertson, SROO
S. A. McNeight
J. E. Cole
L. C. Evans - H. Worthington
M. H. Wahl - L. W. Morris -
S. W. O'Rear - TIS File

December 18, 1959

Classified Letter in Doc. Sect
DP.W-60-106 1/11/60
S.A. McNeight & P.J. Hagelston

Mr. P. J. Hagelston, Director (2)
Industrial and Technical Services Division
Savannah River Operations Office
U. S. Atomic Energy Commission
Post Office Box A
Aiken, South Carolina

Dear Mr. Hagelston:

PROPOSED PUBLICATION - DP-434

Attached for review as to classification and patent matter are five copies of the following report:

Oxidation of Neptunium(V) by Vanadium(V)
by E. K. Dukes

We propose to release the report for standard external distribution.

To facilitate the release of this report, it would be appreciated if you would telephone your comments to M. H. Wahl's office and send a confirming letter to me with a copy to M. H. Wahl. The report will be released when approval is received, but not until after 14 days from the date shown above.

If any clarification or technical information is needed to aid in your patent review, we suggest you get in touch with

C. H. Ice, Research Manager
Savannah River Laboratory

If you decide to pursue a patent on any development covered in the accompanying material, I shall be happy to supply the additional information required such as appropriate references and the name of the person responsible for the development.

Very truly yours,

SWOR

Hood Worthington, Director
Technical Division

HW/bch
Enc.
OSR 24-A148

[redacted]

BETTER THINGS FOR BETTER LIVING . . . THROUGH CHEMISTRY

REPORT DATA SHEET

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5/10/60 Phone from T.Niland to SWO upgrade to Secret & issue all copies - recall Unclassified copies

Report Number DP-434 S C (U) Approved by SWO Rel

Author(s) E. K. Dukes 2104 5/10/60 To Edna by Elin Ls recall

Title Oxidation of Neptunium(V) by Vanadium(V)

Division

Approval (Introduction, Summary, Cover Letter) Section Director 9/28 Laboratory Director 10/5

	Author	Supv.	Div. Hd.
Manuscript Approval (MS)	E.K.D 9/21/59		C.H. Lee
Reproduction Copy Approval (RC)	E.K.D	.....	
Recommended for Publication		.....	

Publication in

Presentation at

TO BE COMPLETED BY TIS

Category Chemistry - General C-446 SWO 5/10/60

Classification of Abstract Unclassified Dates - same Title, abstract - secret Classified names for Org. changes

Classification of Title Unclassified

Transmittal Letter DP TL S C U TIR is unclassified SWO to Elin 5/19 per phone T.Niland

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11/23					X Vari-Typer		X						
11/24				X			X	X					
11/25				X				X					
11/27				X				X					
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12/1					X Vari-Typer								
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12/4					X Layup					X			
12/7					X "			X	X				
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12/10					X								
12/11					X								
12/14		X Katz								X [illegible]			
12/15											X		
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1/11/60	+14 Days												
3/2/60	Letter [illegible] returned [illegible] W-60-106 (Classified) 1/11/60												
4/26/60	Conf. Letter (in File with DP-356)												
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10/17	9	X											
10/18		X Vivian											
10/19		X "											
10/12		X											
10/13		X [illegible]											
10/14		X [illegible]											
10/15			X (1)										
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TELEPHONE
AUGUSTA, GA.
PARK 4-6311

TELEGRAM ADDRESS
AUGUSTA, GA.

IN REPLY REFER TO:
TI:LAB:mep

April 11, 1960

Mr. Hood Worthington, Director
Technical Division, AED
Explosives Department
E. I. du Pont de Nemours and Company
Wilmington 98, Delaware

Dear Mr. Worthington:

This letter will serve to confirm status of review for release of the following formal research and development reports:

DP-356, "An Automatic Gas Chromatograph for Monitoring of Reactor Fuel Failures - Part I - Design", by W. R. Kritz. Transmitted April 13, 1959, for classification and patent review for release to standard TID-4500 distribution.

There is no objection from the classification standpoint to release of this report; however, since patent approval for unclassified release has not yet been secured, external distribution of the document cannot be approved at this time.

DP-434, "Oxidation of Neptunium(V) by Vanadium(V)", by E. K. Dukes. Transmitted December 18, 1959, for classification and patent review for release to standard TID-4500 distribution.

This document is declassifiable, only, and should be handled as Secret, Restricted Data, until formal declassification is accomplished. Review by the Declassification Branch, Oak Ridge, is being held pending patent clearance. If you desire to make distribution of DP-434 at this time, it can be done so only on a classified basis.

The foregoing confirms conversations between Messrs. O'Rear and Niland of our respective staffs.

Very truly yours,

Paul J. Hagelston, Director
Technical & Production Division

cc: M. H. Wahl, du Pont Operations, SRP(2)
S. A. McNeight, du Pont, Wilmington

[redacted]