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AEC RESEARCH AND DEVELOPMENT REPORT

MASTER

HW-64432

**REPROCESSING URANIUM-MOLYBDENUM
ALLOY FUELS --
DISSOLUTION IN CONCENTRATED NITRIC ACID**

W. W. SCHULZ

MARCH 17, 1960

HANFORD LABORATORIES

**HANFORD ATOMIC PRODUCTS OPERATION
RICHLAND, WASHINGTON**

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HW-64432

UC-10, Chemistry-Separation
Processes for Plutonium and Uranium
(TID-4500, 15th Ed.)

REPROCESSING URANIUM-MOLYBDENUM ALLOY FUELS -
DISSOLUTION IN CONCENTRATED NITRIC ACID

By

W. W. Schulz

Chemical Development
Chemical Research and Development Operation

March 17, 1960

HANFORD ATOMIC PRODUCTS OPERATION
RICHLAND, WASHINGTON

Work performed under Contract No. AT(45-1)-1350 between the
Atomic Energy Commission and General Electric Company

Printed by/for the U. S. Atomic Energy Commission

Printed in USA. Price 75 cents. Available from the
Office of Technical Services
Department of Commerce
Washington 25, D. C.

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ABSTRACT

Dissolution of U-3 weight per cent Mo alloys in 12-14 M HNO_3 solutions is discussed. Under these conditions 80-95 per cent of the molybdenum content is precipitated as white hydrated molybdic oxide. Molybdic oxide precipitates are bulky; centrifuged volumes range from six to 17 volume per cent for dissolver solutions 1.0 to 1.5 M U. The precipitates retain some plutonium and uranium even after thorough washing with water and 1 M HNO_3 . Plutonium and uranium values in washed precipitates can be recovered by successive treatment of solid residues with caustic and nitric acid.

Removal of nitric acid from uranium-molybdenum alloy dissolver solutions by boil-down procedures and by reaction with formaldehyde is discussed. Solutions obtained after removal of nitric acid constitute satisfactory low-acid Redox process feedstocks.

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REPROCESSING URANIUM-MOLYBDENUM ALLOY FUELS - DISSOLUTION IN CONCENTRATED NITRIC ACID

I. INTRODUCTION

Several power reactors currently being designed and constructed utilize low enrichment (< 5 per cent U^{235}) uranium in uranium-molybdenum alloys as fuels.⁽¹⁾ The feasibility of preparing low-acid (0.0 - 0.5 M HNO_3), Redox process feeds from uranium-molybdenum alloy fuels by dissolution in nitric acid-ferric nitrate solutions was demonstrated in an earlier study.⁽²⁾ Preparation of Redox process feeds by dissolution of uranium-three to 10 weight per cent molybdenum (U-3 to 10 w/o Mo) alloys in about six (or less) M HNO_3 in the absence of iron (III) or other complexing agent for molybdenum is impractical, however, because of the limited solubility of uranyl molybdate.

Recent ORNL investigations have shown that satisfactory Purex process feed solutions (ca. 1 M UNH, 4 M HNO_3) can be prepared from U-8 to 10 w/o Mo alloy fuels by dissolution in solutions initially 11-14 M HNO_3 .⁽³⁾ Under such conditions a large fraction of the molybdenum is precipitated as hydrated molybdic oxide. Experimental investigation of this type of dissolution process for preparing Redox process feed solutions from U-3 w/o Mo alloy is discussed in this report.

II. SUMMARY AND CONCLUSIONS

Eighty to 95 per cent of the molybdenum content of U-3 w/o Mo alloys was precipitated as white hydrated molybdic oxide when such alloys were dissolved in solutions initially 12-14 M HNO_3 to produce dissolver solutions of the composition 1.0-1.5 M , 4-8 M HNO_3 . Approximately 90 per cent of the silicon content of a U-3 w/o Mo-0.2 w/o Si alloy was precipitated under these conditions.

Molybdic oxide precipitates were bulky and did not settle well. Solid-liquid separation could be achieved by centrifugation. Centrifuged volumes of unwashed precipitates ranged from six to 17 volume per cent for dissolver solutions 1.0 to 1.5 UNH. Most of the uranium and plutonium associated with the centrifuged solids was removed by careful washing with water and/or

nitric acid. Even thorough washing, however, did not remove all the uranium and plutonium. Uranium losses to thoroughly precipitates were less than 0.05 per cent. The plutonium loss to the washed solid was about 2.3 per cent in the range of plutonium content studied. Plutonium and uranium values in washed precipitates were recovered by successive treatment of solid residues with caustic and nitric acid.

In this connection the reader is referred to a "Note in Proof" on page four of Reference 2. The note stated that preliminary data indicated satisfactory feed solutions for a Redox process could be prepared through high-acid dissolution of uranium-molybdenum alloys. The further studies reported herein confirmed that statement except for the relatively large fraction of plutonium which was not readily washed from the molybdic oxide precipitate.

The acidity of solutions produced by dissolution of U-3 w/o Mo alloys in 12-14 M HNO_3 was reduced to Redox process values (0.0-0.5 M HNO_3) either by treatment with formaldehyde or by boil-down with continuous addition of water. The former method was preferable since precipitation of a small amount of molybdic oxide occurred from both twofold concentrated and unconcentrated dissolver solutions. Solids were not formed during formaldehyde treatment provided solutions were not twofold concentrated. Solutions after either boil-down or reaction with formaldehyde constituted satisfactory Redox process feedstocks. Plutonium (IV) in such solutions was readily oxidized to the hexavalent state; remaining molybdenum did not extract into hexone.

III. EXPERIMENTAL

A. Alloy Source and Description

Experimental work was conducted with U-3 w/o Mo, U-3 w/o Mo-0.2 w/o Al, and U-3 w/o Mo-0.2 w/o Si alloys prepared at Hanford by appropriate dilution of a 9 per cent alloy obtained from the Battelle Memorial Institute. One-eighth inch sheets of the 3 per cent molybdenum alloys were prepared by hot rolling at 800-850 C and water quenching. In all cases alloy specimens were unclad and nonirradiated. The composition

of the aluminum and silicon alloys corresponded approximately to that of the fuel proposed for the Piqua Reactor; this fuel is tentatively scheduled to be reprocessed at Hanford.⁽⁴⁾

B. Dissolution Studies

1. Procedures

Complete dissolution of uranium-molybdenum alloy specimens in boiling solutions initially 11-14 M HNO_3 was performed in a glass dissolver fitted with an up-draft condenser. The volume of acid in each case was such as to give a dissolver solution of the desired terminal uranium concentration. In general when dissolution was complete the resulting slurry was cooled to room temperature and centrifuged. In some cases, following dissolution, the slurries were digested one to five hours at boiling temperature before centrifugation.

Solids derived from U-3 w/o Mo-0.2 w/o Si alloy were washed by centrifugation prior to transfer with water to a weighed, medium porosity, sintered glass filter crucible. Precipitate volumes decreased on washing; accordingly, when washing by centrifugation, succeeding wash volumes were made to be equal to twice the volume of precipitate obtained in the previous centrifugation. Solids resulting from dissolution of uranium-molybdenum-aluminum alloys were transferred to the filter first and then washed. In this case a constant volume of wash liquid, equal to twice the centrifuged volume of unwashed precipitate, was used for each wash. In both cases solids were washed three or four times with water and twice with 1 M HNO_3 . Washed solids were dried four to 16 hours at 100-110 C.

In several experiments with U-3 w/o Mo alloy the dissolvent was made 0.1-0.5 g/l Pu (IV). Solids obtained in these cases were washed by centrifugation. A constant volume of wash liquid, equal to the centrifuged volume of unwashed precipitate, was used in each wash. Plutonium, uranium, and molybdenum contents of weighed portions of the washed and dried solids were determined following dissolution in sodium carbonate and acidification. Molybdenum was determined by the thiocyanate-stannous chloride procedure outlined by Sandell.⁽⁵⁾

Instantaneous dissolution rates of U-3 w/o Mo alloy in boiling nitric acid solutions were determined according to the procedure used by Moore, Schulz and Walter.⁽⁶⁾

2. Results and Discussion

Data obtained in complete dissolutions of U-3 w/o Mo alloys in 11-14.0 M HNO_3 solutions are presented in Tables I-III. Instantaneous dissolution rate data are given in Figure 1. These results and other observations made in these experiments may be summarized as follows:

a. Eighty to 95 per cent of the molybdenum content of U-3 w/o Mo alloys is precipitated as white hydrated molybdic oxide (see b. below) when such alloys are dissolved in solutions initially 12 M (or higher) in nitric acid. Approximately 90 per cent of the silicon content of a U-3 w/o Mo-0.2 w/o Si alloy is also precipitated under these conditions. Precipitation of both molybdenum and silicon appears to increase slightly when slurries are digested five hours in boiling temperature after dissolution is complete.

Black or cream colored precipitates were obtained when U-3 w/o Mo alloys were dissolved in 11 M HNO_3 . The cream colored solids are believed to be a mixture of molybdic oxide and uranyl molybdate. The black material was not identified; spectroscopic analysis showed some 15 metals to be present. It is believed to consist of impurities introduced during alloy fabrication at Hanford and not soluble in nitric acid. All white and cream colored solids obtained in this investigation were contaminated with small amounts of the black material. The black material was not soluble in the 12 per cent Na_2CO_3 solution used to dissolve molybdic oxide precipitates.

b. X-ray diffraction and chemical analyses of the white solids indicate them to be principally hydrated molybdic oxide. This identification agrees with that made at ORNL.⁽³⁾ White solids were blue after drying at 100-110 C; this color change is characteristic of MoO_3 .⁽⁷⁾

Precipitation of MoO_3 from strong acid solutions by anionic aggregation of polymolybdate species has been discussed by Killeffer and Linz.⁽⁷⁾ These authors point out that the MoO_3 polymer may not only be more or less hydrated but may also carry with it iso- and heteropoly acids and their salts

TABLE I

DISSOLUTION OF U-3 w/o Mo-0.2 w/o Al ALLOY IN 11-14 M HNO₃ SOLUTIONS

Conditions: See text, page 5.

Dissolvent HNO ₃ , <u>M</u>	Final Soln Comp			Per Cent Mo Pptd	Volume (1) Per Cent	Weight (2) Per Cent	Solids Color Washed Solids	Composition ⁽³⁾	
	<u>U</u> <u>M</u>	<u>HNO₃</u> <u>M</u>	<u>Mo</u> <u>M</u>					w/o U	w/o Mo
11.0	1.49	3.34	0.03	74.0	16.3	3.7	Cream	10.5	49.4
12.0	1.50	4.94	0.02	82.8	16.0	3.0	White	0.30	60.2
13.0	1.42	4.58	0.02	81.7	17.0	2.8	White	0.07	51.6
14.0	1.51	4.22	0.02	82.8	17.3	2.9	White	0.08	62.5
11.0	1.09	6.79	0.07	0.00	--	0.14	Black	--	--
12.0	1.04	6.40	0.01	87.5	9.9	3.2	White	0.06	57.2
13.0	0.95	7.52	0.01	86.3	9.2	2.8	White	0.06	54.7
14.0	1.00	8.29	0.01	87.0	17.4	2.9	White	0.06	57.8

(1) After centrifugation; per cent of initial volume of acid.

(2) Per cent of initial alloy weight.

(3) Composition of washed and dried solids.

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TABLE II

DISSOLUTION OF U-3 w/o Mo-0.2 w/o Si ALLOY IN 11-14 M HNO₃ SOLUTIONS

Conditions: See text, page 5.

Dissolvent HNO ₃ , M	Final Solution Comp.				% Mo Pptd	% Si Pptd	Volume ⁽¹⁾ %	Weight ⁽²⁾ %	Solids		
	U M	HNO ₃ M	Mo M	Si M					Color Washed Solids	Composition ⁽³⁾	
										w/o U	w/o Mo
11.0	1.54	3.10	0.021	0.002	82.4	92.4	6.0	3.8	Cream	15.5	48.4
13.0	1.40	3.84	0.019	---	82.4	--	11.4	3.9	White	0.30	--
14.0	1.30	3.80	0.017	0.002	83.0	91.0	11.4	3.7	White	0.46	49.8
14.0 ⁽⁴⁾	1.37	4.28	0.013	---	87.6	--	11.7	4.3	White	---	--
11.0	1.00	--	0.077	---	0.00	0.00	--	0.12	Black	---	--
12.0	0.99	6.78	0.008	0.001	89.5	94.1	--	4.0	White	0.06	50.6
13.0	0.97	7.65	0.007	0.002	90.6	87.9	6.4	4.2	White	0.19	50.8
14.0	0.88	6.17	0.006	0.002	91.2	86.7	7.3	4.0	White	0.19	--
14.0 ⁽⁵⁾	0.93	6.94	0.005	0.001	93.0	93.7	8.7	4.3	White	---	--
14.0 ⁽⁶⁾	0.81	5.75	0.006	0.001	90.4	90.6	6.4	4.4	White	0.15	46.4
14.0 ⁽⁴⁾	0.93	6.84	0.004	0.001	94.4	91.8	7.8	4.6	White	0.13	60.0

(1) After centrifugation; per cent of initial volume of acid.

(2) Per cent of initial alloy weight.

(3) Composition of washed and dried solids.

(4) Digested 5 hours at boiling temperature after dissolution complete.

(5) Digested 1 hour at boiling temperature after dissolution complete.

(6) Digested 3 hours at boiling temperature after dissolution complete.

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TABLE III
RETENTION OF URANIUM AND PLUTONIUM
ON WASHED MOLYBDIC OXIDE PRECIPITATES

Conditions: See text, page 5.
In all cases U-3 w/o Mo alloy specimens dissolved
in 14 M HNO₃ - Pu (IV) solution to produce 1.5 M
U - ca. 4.0 M HNO₃ dissolver solutions.

Alloy Weight grams	Dissolvent		Weight grams	Solids ⁽¹⁾			Per Cent Loss	
	Pu(IV) g/l	Vol ml		Composition			Pu	U
				w/o Mo	w/o U	w/o Pu		
7.45	0.1	20.5	0.214	64.6	0.36	0.02	2.1	0.011
9.84 ⁽²⁾	0.1	26.7	0.320	65.5	1.5	0.02	2.3	0.049
12.5	0.1	34.1	0.360	61.9	0.52	0.02	2.2	0.015
21.1	0.1	57.3	0.582	62.7	0.45	0.03	2.6	0.013
10.0	0.5	27.3	0.286	64.8	0.96	0.11	2.3	0.028
19.4	0.5	52.5	0.630	--	--	0.09	2.4	---

(1) After washing and drying.

(2) Two water washes, two 0.1 M Na₂Cr₂O₇ washes, and then two more water washes. All others washed four times with equal volume of water and then twice with equal volume of 1 M HNO₃.

FIGURE 1
Instantaneous Dissolution Rates
of U-3 w/o Mo Alloy in Nitric Acid Solutions

that exist as ions in the solution. In this latter connection all white precipitates obtained in this study contained small amounts of uranium (and also plutonium when present) which could not be removed even by extensive washing (see c. below).

c. The white molybdic oxide precipitates were bulky and did not settle well. Solid-liquid separation could be achieved by centrifugation. Centrifuged volumes of unwashed precipitates from U-3 w/o Mo alloy ranged from 6 to 17 per cent of the original volume of acid used in the dissolution step. Precipitate volumes decreased by about 50 per cent on washing. Difficulties involved in handling these large volumes of solids constitute a major disadvantage to dissolution of U-3 w/o Mo alloy fuels in concentrated nitric acid. Such difficulties are magnified if critically safe equipment must be used.

The bulk of the uranium and plutonium associated with the centrifuged solids was removed by careful washing with water and/or nitric acid. Even exhaustive washing, however, did not remove all the uranium and plutonium. Uranium losses to thoroughly washed precipitates did not exceed 0.05 per cent in any case and were generally 0.02 per cent or less. The amount of plutonium retained by the washed solid appeared to vary directly with the dissolver plutonium concentration. In consequence, plutonium loss to the washed solid was about 2.3 per cent in each experiment. It is important to note that plutonium and uranium losses observed in these experiments are minimum values; less efficient washing procedures such as might be employed in plant-scale operation would result in even higher losses.

Investigations at ORNL have shown that uranium values can be recovered from incompletely washed molybdic oxide precipitates by successive treatment with hot 5 M NaOH and hot 11 M HNO₃ solutions.⁽³⁾ Caustic metathesizes the solids to soluble sodium molybdate and insoluble sodium diuranate; the diuranate dissolves in the 11 M HNO₃ solution. One experiment performed in the present investigation showed that plutonium values may also be recovered by this procedure. In this case white solids resulting from dissolution of U-3 w/o Mo alloy in 14 M HNO₃-0.1 g/l Pu(IV) solution were washed four times with equal volumes of water. Solids were then

treated successively with an equal volume of hot (90 C) 5 M NaOH and an equal volume of hot 11 M HNO₃. About 2.7 per cent of the initial plutonium charge was present in the final acid solution; this value is in excellent agreement with that to be expected from the data in Table III. One per cent of the uranium was also recovered in the acid solution. Plutonium and uranium losses to the caustic solution were both less than 0.05 per cent.

d. Boiling 5-14 M HNO₃ solutions dissolve U-3 w/o Mo alloys rapidly; dissolution rates in these solutions range from about 1 to 10 g/sq cm-hr (see Figure 1). The amounts of molybdic oxide formed in short-time dissolvings did not hinder attack of the metal. Such hindrance might occur in large-scale dissolutions, however, and this point requires further investigation.

e. After removal of molybdic oxide precipitates, dissolver solutions resulting from dissolution of U-3 w/o Mo alloys in 12-14 M HNO₃ are stable with respect to precipitation of solids for at least 60 days at 25 C. Trace amounts of white solids precipitate when such solutions are stored at 50 C.

C. Redox Processing of U-Mo Alloy Dissolver Solutions

1. Feed Preparation

a. Acidity Adjustment

The dissolution procedure used in this study yielded dissolver solutions 4-8 molar in nitric acid. Such solutions can be adjusted to Redox process feed acidities (0.0-0.5 M HNO₃) either by destruction of nitric acid with formaldehyde or by boil-off of nitric acid with continuous addition of water. These methods of removing nitric acid have been investigated extensively at HAPO and other sites. (8, 9, 10, 11)

Formaldehyde treatment of uranium-molybdenum alloy dissolver solutions of the compositions listed in Tables I-III was conducted on a batch basis. In each case the calculated volume of 37 per cent HCHO (assuming 0.5 to 0.7 mole HCHO required per mole of HNO₃ to be destroyed) was added slowly to 30 ml of warm (50-60 C) dissolver solution contained in an open Erlenmeyer flask. When the last portion of formaldehyde was added the solution was boiled two to three minutes in the open container and then boiled under reflux for 30 minutes. The final solution was diluted with

water to the original 30 ml volume. Reaction proceeded smoothly in most cases, and final solutions, 0.05 to 0.8 M HNO_3 , free from solids were obtained. These final solutions were also stable with respect to solids formation for at least seven days when stored at either 25 or 50 C. Some solids, not positively identified but thought to be uranyl molybdate, formed when solutions were concentrated about twofold during reaction with formaldehyde.

Except in one case, boil-off of nitric acid from uranium-molybdenum alloy dissolver solutions was performed according to the following procedure:

1. One hundred milliliters of dissolver solution (1.3 M U-3.8 M HNO_3) was concentrated to 50 ml.
2. Water was added to the pot for 2.5 hours at a rate equal to the boil-off rate (1-2 ml/min) to maintain the volume approximately constant at 50 ml.
3. The final solution was diluted to the original 100 ml volume.

Terminal solutions obtained by this procedure were 0.4-0.6 M HNO_3 . In all cases, white solids formed when the dissolver solution was concentrated to half its volume. In one experiment the dissolver solution was not concentrated prior to boil-off of nitric acid. The solution remained clear for about three hours but white solids precipitated thereafter. The terminal acidity even after seven hours of boil-off operation was 0.95 M HNO_3 showing the desirability of concentrating prior to boil-off.

Solids precipitated during boil-off of nitric acid appeared to be hydrated molybdic oxide but were not positively identified. The amount of solids precipitated was small in all cases, about 0.5 volume per cent of the centrifuged terminal boil-down solution and 0.1 to 0.3 weight per cent of the alloy charge used to prepare the dissolver solution. Precipitation of even small amounts of solids during boil-down is undesirable, however, because of the necessity for a second liquid-solid separation operation. Destruction of nitric acid with formaldehyde thus appears preferable to removal of acid by boil-off procedures.

Addition of 50 per cent NaOH to neutralize uranium-molybdenum alloy dissolver solutions directly to 0.0-0.5 M HNO_3 is not practical because of

the large volumes required. After removal of acid by boil-off or by treatment with formaldehyde, however, either caustic or DIBAN (dibasic aluminum nitrate) may be used if required to adjust uranium-molybdenum alloy dissolver solutions to final feed acidities in the range -0.2 to 0.5 M HNO_3 . No solids were obtained with either reagent.

b. Plutonium Oxidation

Sodium dichromate readily oxidizes plutonium (IV) in uranium-molybdenum alloy solutions obtained by high-acid dissolution techniques to the hexavalent state. In a typical experiment a dissolver solution of the composition 1.26 M U - 3.91 M HNO_3 - 0.5 g/l Pu(IV) was adjusted to 0.46 M HNO_3 by treatment with formaldehyde. Portions of the resulting solution were adjusted to 0.0 and -0.2 M HNO_3 by addition of 50 per cent NaOH . These solutions were made 0.2 M $\text{Na}_2\text{Cr}_2\text{O}_7$ and heated two hours at 90 - 95 $^\circ\text{C}$. In both cases 99.7-99.8 per cent of the plutonium was oxidized to plutonium (VI) in one-half hour.

Solutions in this and similar experiments were stable toward solid formation during the two-hour period at 90 - 95 $^\circ\text{C}$. The oxidized solutions were stable toward precipitation of solids for at least seven days when stored at 25 $^\circ\text{C}$.

2. Solvent Extraction Behavior

Molybdenum remaining in solution after dissolution of U-3 w/o Mo alloys in 12 - 14 M HNO_3 solutions is not extracted under Redox process conditions. Dissolver solutions of the composition 1.0 - 1.5 M U , 4 - 8 M HNO_3 were treated with formaldehyde to adjust acidities to 0.0 - 0.6 M HNO_3 . The resulting solutions were adjusted to 0.0 M HNO_3 by addition of caustic and made 0.2 M $\text{Na}_2\text{Cr}_2\text{O}_7$. Solutions were heated two hours at 90 - 95 $^\circ\text{C}$ to simulate the plutonium oxidation step. After cooling to 25 $^\circ\text{C}$, the solutions were adjusted to 0.4 M HNO_3 to prepare Redox process acid feeds. Five-milliliter portions of these solutions were mixed with one-fifth volume portions of scrub solution (1.3 M ANN - 0.0 M HNO_3 - 0.01 M $\text{Na}_2\text{Cr}_2\text{O}_7$); the resulting solutions were contacted with an equal volume of neutral hexone. Molybdenum was not detected in any of the organic phases.

D. Chemical Flowsheet

A study flowsheet for preparation of Redox process feedstock from U-3 w/o Mo alloy is shown in Figure 2.

IV. ACKNOWLEDGEMENT

The assistance of L. C. Neil in obtaining experimental data is gratefully acknowledged.

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FIGURE 2

Study Flowsheet for Preparation of Redox Process Feed from U-3 w/o Mo Alloy Fuel

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1	Nuclear Development Corporation of America
1	Nuclear Metals, Inc.
1	Oak Ridge Institute of Nuclear Studies
10	Office of Naval Research
1	Office of Ordnance Research
1	Patent Branch, Washington
6	Phillips Petroleum Company (NRTS)
1	Power Reactor Development Company
2	Pratt and Whitney Aircraft Division
2	Public Health Service
1	Sylvania Electric Products, Inc.
1	Tennessee Valley Authority
3	Union Carbide Nuclear Company (ORGDP)
4	Union Carbide Nuclear Company (ORNL)
1	Union Carbide Nuclear Company (Paducah Plant)
1	U. S. Geological Survey, Denver
1	U. S. Geological Survey, Washington
1	U. S. Naval Ordnance Laboratory
1	U. S. Naval Postgraduate School
1	U. S. Patent Office
2	University of California, Berkeley
2	University of California, Livermore
1	University of Puerto Rico
1	University of Utah
1	Westinghouse Electric Corporation (Schafer)
1	Yankee Atomic Electric Company
325	Technical Information Service Extension
75	Office of Technical Services, Washington