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STUDIES AND RESEARCH
CONCERNING BNFP

CONVERSION OF NEPTUNIUM NITRATE TO NEPTUNIUM OXIDE

FINAL REPORT

Hans G. Tepp

July 1979

~~APPLIED TECHNOLOGY~~

ALLIED-GENERAL NUCLEAR SERVICES
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PREPARED FOR THE
DEPARTMENT OF ENERGY
FUEL CYCLE PROJECT OFFICE
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ABSTRACT

Means of converting neptunium nitrate to neptunium oxide were studied, as part of a study of a fuel cycle concept based on increasing the isotopic concentration of Pu-238. The process chosen was one entailing precipitation of neptunium as an oxalate and calcination of the oxalate to neptunium oxide. A quantitative flowsheet was developed, representing the conversion of 6 kilograms of neptunium per day.

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1.0 INTRODUCTION

This report summarizes a study of certain processes by which neptunium nitrate solution could be converted to neptunium oxide. It is one of a series of reports generated under DOE Contract No. DE-AC09-78ET-35900, a part of which is Task 3, Proliferation Resistant Fuel Cycle Concept. This task is a study of a fuel cycle concept which may enhance proliferation resistance by increasing the isotopic concentration of Pu-238 in the plutonium product, the so-called Heat Spike Concept. One means by which this isotopic enrichment can be achieved is by recycling neptunium generated in a uranium fuel cycle, as neptunium is a precursor to Pu-238.

Task 3 is divided into various subtasks concerned with the overall fuel cycle -- concept definition, evaluation of processing alternatives, neptunium isolation, research and development studies of related process steps, handling of products and wastes from the cycle, and an economic model of the concept. This report is part of Sub-Task 3.4, Products and Waste Handling, and it covers the conversion of the neptunium nitrate, extracted from the fuel cycle, to neptunium oxide suitable for recycle.

2.0 SUMMARY AND CONCLUSIONS

A literature search was performed on the subject of converting neptunium nitrate to neptunium oxide. Four reasonable processes were identified. These are: (1) direct thermal denitration, (2) peroxide precipitation, (3) hydroxide precipitation, and (4) oxalate precipitation. Following precipitation, the products are calcined to the oxide.

The four processes were assessed as to demonstrated operating reliability and product quality. Of the four processes, oxalate precipitation was found to be the most developed and had the most data available for developing a flowsheet which could be used at a facility such as the Barnwell Nuclear Fuel Plant (BNFP). A quantitative flowsheet was developed for converting six kilograms of neptunium per day from a nitrate solution to an oxide. This flowsheet is to be used in performing preliminary engineering studies which are reported under Task 4 of the AGNS work.

3.0 CHOICE OF PROCESS

The starting material for the conversion is neptunium nitrate product solution from the Separations Facility. The process for producing this product is described by Murbach.(8) Typical of many metal nitrates, this solution is amenable to various process routes to the final oxide form. Among these are direct thermal denitration and several indirect means, such as precipitation as an oxalate, hydroxide, or peroxide, followed by calcination to the dioxide.(1,7) All processes have the stable NpO_2 as the final product, thus a choice must be made based on divers parameters, including, among others, operability, effectiveness, yield, product purity, extent of practice, operating costs, and wastes generation.

3.1 Thermal Denitration

The most direct process is thermal denitration of the neptunium nitrate.

$$Np(NO_3)_4 \underset{\text{heat}}{\rightarrow} NpO_2 + 2NO_2\uparrow + 2NO\uparrow + 2O_2\uparrow \qquad \text{(Eq. 1)}$$

This reaction would occur at a temperature of 700°C in an air atmosphere, for the dioxide is expected to be the final product of denitration of neptunium of any oxidation state. At a lower temperature range of 275 to 450°C and with neptunium at valences of (V) or (VI), Np_3O_8 could be the product.(1) In any event, heating the neptunium nitrate for several hours in equipment provided with moderate stirring would form NpO_2 powder. The product is cooled, ground, and screened as necessary, and packaged.

However, while it is the simplest process overall, it has a major drawback in having a poor decontamination factor. The level of fission product contamination expected in neptunium extracted from 160-day cooled fuel is up to two millicuries per gram of neptunium.(8) At this level the NpO_2 product from direct calcination could be too contaminated for elementary fuel fabrication methods. The nonvolatility of fission product oxides would assure their retention in the product derived from thermal denitration.

3.2 Hydroxide Precipitation

Of the three processes entailing a preliminary precipitation before final conversion to an oxide, the hydroxide precipitation process has the least amount of data available. In this process, neptunium nitrate is reacted with ammonium hydroxide to precipitate neptunium hydroxide. The precipitate is collected in a filter and washed with dilute ammonium hydroxide, followed by water. Following air drying, the precipitate is transferred to a refractory boat and fired at 700° to 800°C in air to decompose the hydroxide and form the oxide. The off-gas contains traces of ammonia from occlusion in the precipitate, but at a level too low to warrant recovery. The hydroxide formed varies in color and structure, depending on the valence of the starting neptunium,(1) but dioxide is the final product when ignited to the 700° to 800°C temperature range regardless of the valency of the neptunium. As discussed in Section 3.1, lower ignition temperatures with neptunium valences above (IV) would form Np_3O_8.(1)

This process is not favored for use in facilities such as the BNFP. Coprecipitation of fission products and other metallic species, which may be present to some degree as corrosion products, contribute to product contamination. The gelatinous properties of these precipitates as well as those of neptunium hydroxide are also considered to be disadvantageous during filtration.

3.3 Peroxide Precipitation

Another of the conversion processes which entails a preliminary precipitation of neptunium before final calcination to an oxide is the peroxide precipitation process. It has been used on a large scale for preparing NpO_2, especially where the oxide is further converted to NpF_4.(9) The process is applicable to Np(IV), (V), or (VI) nitrate solutions in a concentration range of 5 to 50 grams Np per liter without the need for valence adjustment.(5)

The conversion is optimized when the neptunium feed solution is 3 to 4 molar HNO_3. This initial concentration not only depresses the solubility of the precipitated neptunium peroxide but also assures effective reduction of all neptunium having a valence above (IV) by hydrogen peroxide.(3, 5) The precipitation occurs at room temperature by the addition of an equal volume of 30% hydrogen peroxide solution. The slurry is cooled to 8°C to limit neptunium solubility and then digested to assure complete reaction and effective filtration. The neptunium peroxide, precipitated as $NpO_4 \cdot 2H_2O$,(1) is then filtered and the cake washed with a solution of 1.5M HNO_3 - 15% hydrogen peroxide. The precipitate is then dried and calcined to the NpO_2 final product.

3.4 Oxalate Precipitation

This method of converting neptunium nitrate solution to neptunium oxide has been practiced widely, both at the Mound Laboratory(12) and the Savannah River Plant.(5, 9) It is applicable to neptunium solutions of 5 to 50 grams Np per liter, but requires valence adjustment to the (IV) state before precipitation.

In brief, (more extensive details of the process are contained in Section 4.0) the neptunium feed solution is adjusted to a nitric acid molarity of 1 to 4M for optimum reduction of any Np above the (IV) valence and to limit solubility of neptunium oxalate. The solution is then heated to 50°C and 1M ascorbic acid is added to reduce all Np to the (IV) valence state. Following the reduction step, the neptunium is precipitated as an oxalate by the addition of a 1M oxalic acid solution. The precipitate, in the form of $Np(C_2O_4)_2 \cdot 6H_2O$, is digested at 50°C to assure completion of reaction and permit crystal growth. The slurry is then cooled to room temperature and filtered. The filter cake is washed with a 1M HNO_3-0.1M oxalic acid solution. The precipitate is then heated to 285°C to strip the waters of hydration and calcined at 500°C to convert the neptunium oxalate to the dioxide.(2, 5)

3.5 Comparison of Peroxide and Oxalate Precipitations

Both of these processes have demonstrated through successful practice their capability to convert neptunium nitrate to neptunium dioxide. The choice of one over the other for the purpose of this report, a process suitable for application at BNFP, therefore relies on specific characteristics of each as they relate to process reliability, neptunium yield, and product quality. The following discussion details some of the process features and their relative benefits.

Feed Adjustment

The oxalate precipitation process requires some feed adjustment to optimize the subsequent reduction of neptunium species having valences of (V) and (VI). This adjustment is made by adding hydrazine to a very slight excess. A valence adjustment is not necessary for the peroxide precipitation process.

Reduction

The major advantage associated with the peroxide precipitation process is that the hydrogen peroxide reactant is both a reductant as well as a precipitant. The oxalate process requires ascorbic acid for reduction and oxalic acid for precipitation. The added reduction step and the need for two reagents thus places the oxalate precipitation at a disadvantage.

Precipitation and Digestion

Experience with both processes is that the precipitation of neptunium oxalate is superior to precipitation of neptunium peroxide.(5) Neptunium oxalate has been precipitated with losses to the filtrate being at levels of 10 milligrams neptunium or less per liter with only a nominal excess of oxalic acid precipitant.(2) Neptunium peroxide generally has a higher solubility than neptunium oxalate and can exhibit the chemical instability of metal peroxides. The higher solubility and lower stability for the peroxide results in greater neptunium losses in the filtrate.(3, 4, 5) To precipitate neptunium peroxide, several molar excess of hydrogen peroxide precipitant is required to achieve a low solubility level.(3, 4)

Digestion of the neptunium peroxide precipitate at a temperature of approximately 8°C was found necessary to assure a low solubility. Although precipitation and digestion of neptunium oxalate is performed best at approximately 50°C, maintaining the slurry at an elevated temperature is considered less difficult and cumbersome than sustaining the slurry at a reduced temperature level, as recommended for the peroxide precipitate.

Filtration

Filterability of the neptunium oxalate is substantially superior to that of neptunium peroxide. Under optimum conditions, filtration times for the peroxide were found to be about two to three times longer than for the oxalate and generally less reliable.(2, 3)

Decontamination

Decontamination from fission products for the peroxide precipitation process was less than that for the oxalate precipitation.(2, 3, 5) The peroxide precipitation process retained 30% of Zr^{95}-Nb^{95} originally present while the oxalate process retained only 2 to 3%. The oxalate process would retain only about 1% of the ruthenium while the peroxide process would most likely retain a higher percentage. Neither the peroxide nor the oxalate was found to be effective in reducing plutonium and protactinium contamination.

3.6 Process Choice

Based on the foregoing review of the various means of converting neptunium nitrate to neptunium oxide, it is apparent that prudency dictates the rejection of thermal denitration and hydroxide precipitation as viable choices for converting neptunium at this time. Both processes have had little development or practice on a meaningful scale. It is, therefore, unlikely that either of these two processes would provide NpO_2 of acceptable quality in a reliable fashion.

The strongest process candidates for adaptation are the oxalate and peroxide precipitation processes. Both have been extensively developed and have provided NpO_2 of quality suitable for the nuclear fuel cycle. All in all, the oxalate precipitation process is considered preferable by virtue of its greater operating reliability, more extensive practice, and higher decontamination factors. It is, therefore, chosen as the process to be applied to the conversion of BNFP neptunium product.

4.0 PROCESS DESCRIPTION

In this section, the flowsheet and material balance for conversion of neptunium nitrate to neptunium oxide are developed. The flowsheet is based on that used by the Savannah River Plant for the conversion of neptunium nitrate to neptunium oxide. In the SRP flowsheet the neptunium is in the form of a neptunium nitrate solution isolated and purified by solvent extraction and anion exchange techniques.(2, 5, 9) The neptunium for the process model of this report is neptunium nitrate solution derived by solvent extraction only, as described by Murbach.(8) Although the ion exchange process is not part of the BNFP flowsheet, the neptunium nitrate solution which will be the feed for this process should contain essentially the same impurities. The feed for the process of this report is a 30 gram neptunium per liter nitrate solution, 3 to 4 M in HNO_3, while the Savannah River Plant Facility feed is a 5 to 50 grams neptunium per liter nitrate solution, 1 to 4 M in HNO_3. One difference is that the Savannah River Plant solution has the neptunium in the (IV) valence state as a result of the preceding anion exchange purification, and stabilized by 0.05 M hydrazine, while the feed solution used for the flowsheet for this report has the neptunium (V) valence present. As a result, the latter feed solution will require more extensive feed adjustment before precipitation.

A simplified block flow diagram of the process is depicted by Figure 1. A quantitative flowsheet for conversion of six kilograms of neptunium per day, from a nitrate solution to neptunium oxide is found in Figure 2. The quantities in Figure 2 are on a daily basis. No attempt was made to reduce the flows to a batch basis for operating and plant design practicability. The actual operating mode of the model facility is covered in the report(10) covering Sub-Task 4.3, Engineering Evaluation of Products Handling and Storage Facilities, of the subject Department of Energy contract.

4.1 Feed Adjustment

The neptunium nitrate feed solution originating in the Separations Facility contains 30 grams neptunium per liter and is approximately 3.4 M HNO_3. The valence state of neptunium is primarily (V). A higher nitric acid content would be needed to oxidize the neptunium to neptunium(VI).(1) The boiling-down step in the Separations Facility for concentration should not cause significant further oxidation.(9) Next is added sufficient water (or nitric acid) to adjust the nitric acid concentration to 3 M HNO_3. This lower nitric acid concentration is necessary to minimize the solubility of the precipitated neptunium oxalate.

Finally, hydrazine is added to assure reduction of any neptunium(VI) which may be present to neptunium(IV) and to stabilize the ascorbic acid. The hydrazine is added to 0.05 M excess in the form of a 35 wt% solution. An aqueous 35 wt% hydrazine solution is used to minimize the flammability hazard of hydrazine.

4.2 Reduction

The reduction of neptunium(V) to (IV) occurs by the action of ascorbic acid.

$$NpO_2^{+} + C_6H_8O_6 \rightarrow Np^{++++} + C_6H_6O_6 \qquad \text{(Eq. 2)}$$

Extensive testing by the Savannah River Laboratory(2) resulted in the choice of ascorbic acid over other reducing agents. Ascorbic acid is a rapid and effective reductant at normal temperature without addition of species which would contaminate the final oxide product. The reduction is optimized in the presence of nitric acid.

The ascorbic acid reductant is added as a 1.0 M solution. An ascorbic acid solution can be readily prepared by dissolving ascorbic acid crystals in water.

The reduction rate and completion is optimized when the adjusted neptunium solution, with HNO_3 and hydrazine, is first heated to 50°C, and the ascorbic acid solution is added to an 0.03 M excess.

4.3 Precipitation

The precipitant is oxalic acid, prepared at 1.0 molar concentration by dissolving oxalic acid crystals in water. The oxalic acid form is usually a dihydrate crystal, and exhibits limited solubility in water at room temperature. The 1.0 molar concentration used for precipitation is slightly less than the solubility of oxalic acid in water at room temperature. As a result, dissolution of the oxalic acid crystal is normally carried out in warm water for greater effectiveness.

The addition of oxalic acid to the prepared neptunium feed solution at 50°C is performed over a 30- to 45-minute period, until a 0.1 M excess oxalic acid is achieved. The equation for neptunium oxalate precipitation is as follows:

$$Np(NO_3)_4 + 2H_2C_2O_4 \rightarrow Np(C_2O_4)_2\downarrow + 4HNO_3 \qquad \text{(Eq. 3)}$$

The precipitation should proceed immediately after the addition of ascorbic acid. There is a small but definite loss of reductant due to degradation of the ascorbic acid when exposed too long at 50°C to the excess HNO_3. A resultant incomplete reduction of neptunium would increase neptunium loss in the filtrate due to the greater solubility of neptunium(V) oxalate compared to neptunium(IV) oxalate. Therefore, precipitation always proceeds immediately after reduction.

Once the oxalic acid is added and the excess achieved, the slurry is maintained at 50° ± 5°C under slow agitation for 30 minutes to permit digestion. This allows the precipitated neptunium oxalate crystals to grow for easier filtration, permits the precipitation to achieve completion, and results in a higher decontamination factor for various impurities.

As seen by the precipitation equation, nitric acid is a by-product. Based on this analysis, the final slurry attains a total free nitric concentration of approximately 2 molar. This molarity, coupled with the approximately 0.1 molar excess of oxalic acid, should provide a satisfactory filtrate composition conducive to a neptunium oxalate solubility of less than 10 milligrams neptunium per liter at approximately 20°C.(3)

4.4 Filtration

The slurry, after digestion at 50°C for 30 minutes, is cooled to room temperature before filtration. The composition of the precipitate is actually a hexahydrate, $Np(C_2O_4)_2 \cdot 6H_2O$. It filters readily and retains free water in a range of 3 to 30 wt% with 20 wt% a typical level. The precipitate is washed three times, each with an equal cake volume of wash solution. To retain low solubility of the neptunium precipitate, the wash solution composition is 0.1 M oxalic acid and 1.5 M HNO_3.

After the final wash, air drying the filter cake for 30 minutes should reduce the free water level to the 15 to 20 wt% range. At this point, the precipitate has a bulk density of approximately 1.0 gram per milliliter.

4.5 Drying and Calcination

The filter cake of $Np(C_2O_4)_2 \cdot 6H_2O$ is then transferred to a furnace for sequential drying and calcination. The rate of heating is not critical, with a rate of 5° to 6°C per minute typical, for crepitation of the precipitate does not occur. The drying step proceeds smoothly, with the 6 moles of water of hydration lost between 90° and 285°C, at which point the anhydrous oxalate is achieved.

$$Np(C_2O_4)_2 \cdot 6H_2O \rightarrow Np(C_2O_4)_2 + 6H_2O\uparrow \qquad \text{(Eq. 4)}$$

With continued heating, the oxalate powder then decomposes to its oxide, achieving completion at 330° ± 5°C.(5)

$$Np(C_2O_4)_2 \rightarrow NpO_2 + 2CO\uparrow + 2CO_2\uparrow \qquad \text{(Eq. 5)}$$

Although 330°C suffices to decompose the oxalate completely, the practice is to heat the calcining furnace to 150°C over a 1-hour period, hold it there for 1 hour to complete the removal of free surface water, and then proceed to 500° to 550°C over a 1-hour period. After reaching the temperature, the furnace is held at 500° to 550°C for 2 hours to complete the calcination.

During the drying and calcination steps, room air is passed through the furnace to sweep out the moisture, CO, and CO_2. An inert atmosphere is not necessary, for NpO_2 is stable to further oxidation at the temperature of practice.(7)

After calcination, the NpO_2 is cooled and discharged for packaging.

4.6 Liquid Wastes

The liquid wastes from the process consist of the filtrates of both the primary filtration cycle and the precipitate wash cycle. As a result, they are a water solution of the composition shown in Figure 2 (stream 19) and are primarily 0.1 M in oxalic acid, 0.02 M in ascorbic acid, and approximately 2 M in HNO_3. Besides small quantities of neptunium being present, (less than 10 milligrams neptunium per liter), the bulk of the trace fission products originally present in the neptunium feed solution will also be found.

Disposition of the wastes from the neptunium conversion process are covered in other reports.(10, 11)

4.7 Product Quality

The neptunium oxide made by calcination of neptunium oxalate is a free-flowing powder, dark green to olive in color. The particle size of the material made by the process described has a median of 9 to 13 μm, and has a bulk density of 2.5 grams per milliliter. Work has been carried out(6) to study means of preparing oxide of different particle size ranges to suit particular needs of end-uses. These tests have resulted in preparing oxide of appreciable particle size variations, made possible through changes in precipitation conditions, such as temperature, reactant concentration, and neptunium concentration in the feed solution.

Quality of the product as to radioactive contamination is a direct result of the purity of the neptunium feed. Reference 2 quotes decontamination factors of 40 for Zr-95-Nb-95, greater than 100 for Ru-103, and no significant separation from plutonium and protactinium. Applying these DF's to the product solution from 2-year cooled fuel processed in the neptunium separation and purification facility,(8) the neptunium oxide product could have the following levels of radioactive contamination:

• Zr-95-Nb-95	0.05 μCi per gram neptunium
• Ru-106	0.08 μCi per gram neptunium
• Uranium	3200 ppm, neptunium basis
• Plutonium	20 ppm, neptunium basis
• Pa-233	0.7 mCi per gram neptunium*

---

*The protactinium content is based on approximately 90 days storage time following separation.

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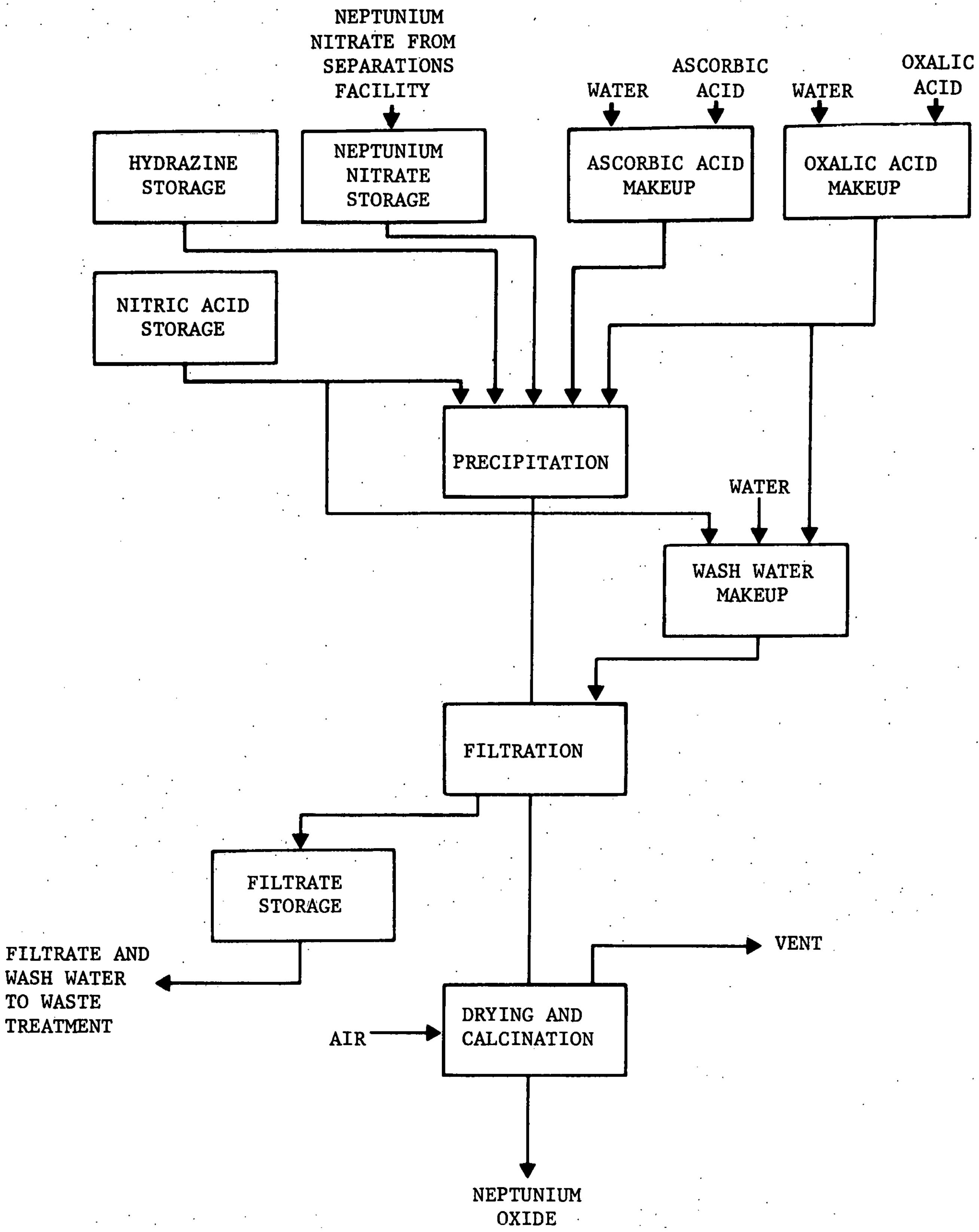
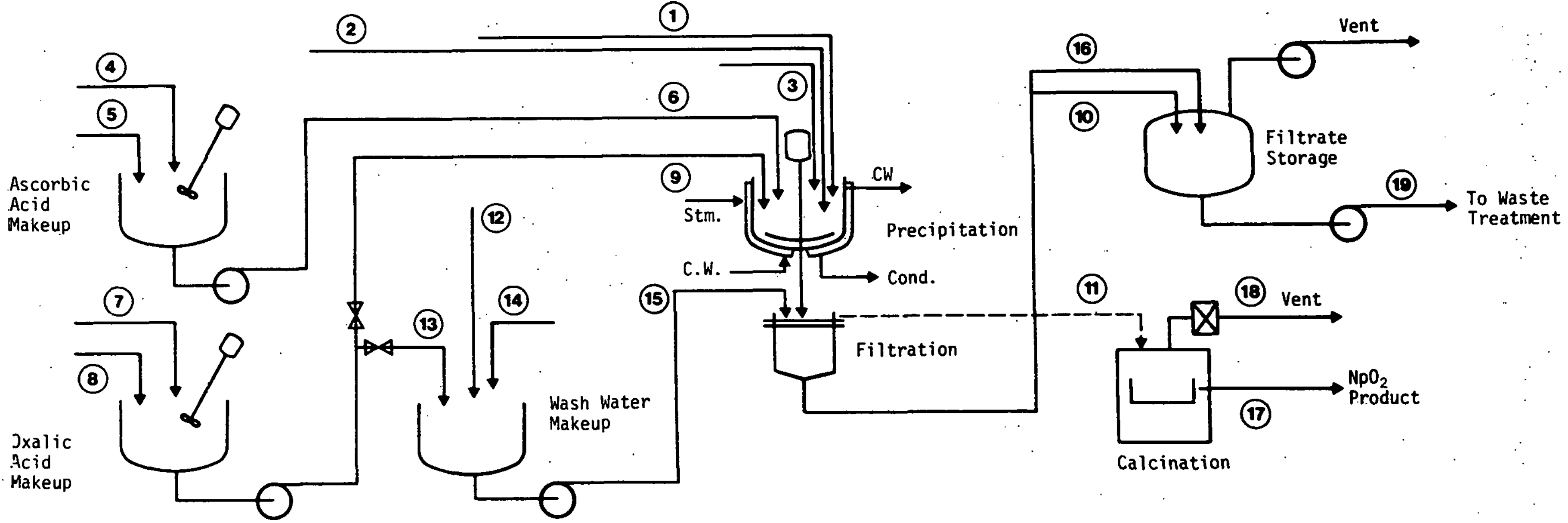


FIGURE 1

BLOCK FLOW DIAGRAM
CONVERSION OF NEPTUNIUM NITRATE TO NEPTUNIUM OXIDE



Basis: 6.0 kg Neptunium
per day

STREAM NO.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19
STREAM NAME	Neptunium Feed	Hydrazine Solution	Water Diluent	Ascorbic Acid	Water	Ascorbic Acid Solution	Oxalic Acid	Water	Oxalic Acid Solution	Primary Filtrate	Filter Cake	Nitric Acid	Oxalic Acid Solution	Water	Wash Water	Wash Water Filtrate	NpO_2 Product	Calciner Vent	Liquid Waste
TYPE FLOW	L	L	L	S	L	L	S	L	L	L	S	L	L	L	L	L	S	V	L
TEMP. °C	20	20	20		20	20	20	20	20	20	20	20	20	20	20	20	20	400	20
DENSITY AT T.P. gm/cc	1.13 (Est.)	1.0	1.0		1.0	1.08	1.65	1.0	1.04	1.05	1.0	1.41	1.04	1.0	1.05	1.05	2.5		1.05
LITERS/BATCH	200	1.12	44		20.64	20.64		87.47	85.79	335.15	16.49	4.73	4.95	39.79	49.47	49.47	2.72	24.10×10^3	384.62
KILOGRAMS/BATCH	226.0 (Est.)	1.12	44	3.64	20.64	22.29	11.44	87.47	89.22	351.91	16.49	6.70	5.15	39.79	51.94	51.94	6.81	(exclusive of air)	403.85
COMPONENTS & CONCENTRATION																			
NpO_2NO_3 gm Np/L	30																		
$H_2C_2O_4$ M									1.0	0.105			1.0		0.1	0.107			0.105
$C_6H_8O_6$ M						1.0													
$C_6H_6O_6$ M										0.024						trace			0.021
HNO_3 M	3.44									2.26		15.7			1.5	1.65			2.16
Np mgm/L										<10						trace		trace	trace
Hydrazine wt/o		35								trace									
HNO_3 wt/o												68							12.97
H_2O wt/o		65	100		100		28.6	100			20	32		100					85.78
Ascorbic Acid wt/o				100															0.35
Oxalic Acid wt/o							71.4												0.90
$Np(C_2O_4)_2 \cdot 6H_2O$ wt/o											80								
NpO_2 wt/o																	100		
CO_2 vol/o																		11.62	
CO vol/o																		11.62	
H_2O vol/o																		76.76	

CONVERSION OF NEPTUNIUM NITRATE TO NEPTUNIUM OXIDE - OXALATE ROUTE

FIGURE 2

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1	DOE Rocky Flats Area Office, Golden, CO
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