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MANUAL FOR THE PREPARATION OF SIMULATED
FUEL REPROCESSING WASTE SOLUTION

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HEALTH PHYSICS DIVISION
and
CHEMICAL TECHNOLOGY DIVISION

MANUAL FOR THE PREPARATION OF SIMULATED FUEL
REPROCESSING WASTE SOLUTION

William J. Lacy

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MANUAL FOR THE PREPARATION OF SIMULATED FUEL
REPROCESSING WASTE SOLUTION

O.O ABSTRACT

Preparation of synthetic wastes from the processing of aluminum-clad natural uranium, aluminum-enriched uranium alloy, zirconium-uranium, stainless steel-UO₂, and stainless steel-UO₂-MgO fuel elements is described. These preparations will be used in waste disposal studies.

MANUAL FOR THE PREPARATION OF SIMULATED FUEL
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1.0 INTRODUCTION

This report gives the compositions and methods of preparing simulated radiochemical process wastes of various types for use in waste disposal studies. For much of the research on waste disposal, simulated wastes are entirely satisfactory. The use of actual wastes would involve unnecessary personnel radiation exposure. For some years the wastes of interest will probably be the aqueous raffinates from solvent extraction processes. Only the potentially high-activity-level wastes are considered here. Wastes from the Purex (HNO_3), TBP-25 (Al-HNO_3), STR (Zr-HF-Al), Darex (stainless steel- HNO_3), and SIR (stainless steel- H_2SO_4) processes are described.

Obviously, the wastes from a given plant's solvent extraction effluent vary to some extent from day to day, and the chemical compositions given are nominal values. No attempt was made to add the minute amounts of various chemicals or corrosion products, that may or may not be important, in a particular test or experiment. For example, traces of tributyl phosphate (TBP), TBP radiolysis degradation products, and stable or radioactive fission products are not added. The synthetic wastes described contain more than 99 per cent of the bulk salts present.

Assumptions made are: fuels will be irradiated to 4000 megawatt days per ton at a neutron flux of 5×10^{13} n/cm²/sec and decayed 100 days after discharge

from the reactor; the burnup for U-235 enriched fuel elements will be 53 per cent; and the energy distribution is approximately 50 per cent gamma and 50 per cent beta.² One gram of U-235 fissioned will produce approximately 1 Mwd of heat.

2.0 WASTE FROM PROCESSING OF ALUMINUM CLAD

NATURAL URANIUM FUEL ELEMENTS

The Purex process* is used in recovering uranium and plutonium from irradiated aluminum-clad natural uranium fuel elements, such as those of the Hanford and Savannah River reactors. The cladding is removed mechanically or by dissolution in caustic. The uranium is then dissolved in nitric acid, using mercury as a catalyst, and the uranium and plutonium extracted from the nitric acid solution with tributyl phosphate (TBP) in a kerosene type diluent. Nitric acid is the salting agent. The uranium and plutonium are eventually separated from each other and further purified. The waste of interest is the aqueous raffinate remaining after the first extraction; it contains the bulk of the fission products and therefore has a high level of radioactivity. It is evaporated to reduce the volume to be stored, recover nitric acid, and decrease sodium hydroxide required for neutralization prior to storage in mild steel tanks.

*In the older Redox process the solvent was hexone (methyl isobutyl ketone), and the salting agent was aluminum nitrate. The advantages of the Purex process are the higher flash point and lower volatility of the TBP and the ready recoverability, by distillation, of the nitric acid salting agent so that less waste storage volume is required. The Redox aluminum-bearing wastes are neutralized with caustic prior to storage in mild steel tanks.

Simulated Purex waste that has been concentrated by evaporation but not neutralized is prepared from the following chemicals, which are dissolved and then diluted to one liter:

<u>Chemical</u>	<u>Molecular Wt.</u>	<u>Amt. Used per Liter</u>	<u>Final Conc., M</u>
$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	404	202 g	0.5
Na_2SO_4	142	21.3 g	0.15
H_2SO_4	98	35.5 ml of 18 M	0.60
HNO_3	63	353 ml of 15.6	5.5

The nominal composition of a typical Purex waste is:

	Volume gal/ton U	Specific Gravity	Concentration, Moles						Fission Products Grams/L
			H^+	Na^+	Fe^{+++}	NO_3^-	SO_4^{--}	OH^-	
Original	1280	1.01	1	Trace	Trace	1	0.03	0	0.25
Evaporated Concentrate	125	1.20	7.0	0.3	0.5	7.3	0.75	0	2.50
Neutralized Concentrate	132	1.18	0	5.3	0.034	7.1	0.10	5.0	2.50

The radioactivity of the neutralized concentrate is 1300 curies/gal, and the energy emitted is 22.7 Btu/hr gal. The actual Purex waste contains traces of tributyl phosphate and of tributyl phosphate radiolysis products (DBP, H_3PO_4 , butanol, and hydrocarbons).

3.0 WASTE FROM PROCESSING OF ALUMINUM-ENRICHED URANIUM ALLOY FUEL ELEMENTS

The TBP-25 process is used in separating and decontaminating enriched uranium from uranium-aluminum alloy fuel elements; for example, the fuel of the Materials Testing Reactor (MTR) at the Idaho Chemical processing Plant. The fuel is dissolved in nitric acid, with mercuric nitrate as catalyst, and the uranium is extracted with 5 per cent tributyl phosphate in a kerosene-type diluent; both nitric acid and aluminum nitrate are salting agents. The aluminum wastes are not neutralized and are stored in stainless steel tanks.

Simulated first extraction column waste is prepared from the following chemicals:

<u>Chemical</u>	<u>Molecular Wt.</u>	<u>Amt. Used per Liter</u>	<u>Final Conc., M</u>
$\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	375	600 g	1.6
$\text{Fe}_2(\text{SO}_4)_3 \cdot x\text{H}_2\text{O}$	400	0.8 g*	0.002
$(\text{NH}_4)_2\text{SO}_4$	132	2.64 g	0.02
$\text{Hg}(\text{NO}_3)_2$	325	3.25 g	0.01
NH_4NO_3	80	2.4 g	0.03
HNO_3	63	33 ml of 15.6 M	0.5

* To be added as $\text{Fe}_2(\text{SO}_4)_3$

The chemicals are dissolved in water and diluted to one liter.

The average chemical composition of a typical Idaho Chemical Processing Plant (ICPP) TBP-25 process waste solution is:

Volume	0.22 to 0.11 gal/g U-235		
Specific gravity	1.29	NO_3^-	5.5 <u>M</u>
H^+	0.5 <u>M</u>	SO_4^{--}	0.02 <u>M</u>
Al^{3+}	1.6 <u>M</u>	Fe^{3+}	0.002 <u>M</u>
Hg^{++}	0.01 <u>M</u>	NH_4^+	0.05 <u>M</u>

The radioactivity of the waste is 2500 to 5100 curies/gal; the energy is 44 to 88.6 Btu/hr. gal, or 12.5 to 26 watts/gal.

4.0 WASTE FROM PROCESSING OF ZIRCONIUM URANIUM FUEL ELEMENTS

With the increasing need for a more corrosion-resistant and a more thermally stable fuel element, the reactor designers turned to zirconium and stainless steels (304 and 347) for fuel components. The Submarine Thermal Reactor (STR) of the Nautilus has a zircaloy (98% Zr - 2% Sn) clad zirconium-enriched uranium alloy core. The choice of dissolution reagents is very limited with this corrosion-resistant material, and the elements are currently being dissolved in hydrofluoric acid. The uranium (IV) is oxidized to U(VI) by chromate and extracted with 10 per cent tributyl phosphate. During this oxidizing step the Al(III) complexes the free fluoride ion.

Simulated first extraction column waste is prepared from the following chemicals:

<u>Chemical</u>	<u>Molecular Wt.</u>	<u>Amt. Used per Liter</u>	<u>Final Conc., M</u>
$\text{Zr}(\text{OH})_4 \cdot 12\text{H}_2\text{O}$	375	206 g	0.55
$\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	375	273 g	0.75
$\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	400	6.4 g	0.016
$\text{Sn}(\text{mossy})$	119	1.43	0.012
HF	20	107 ml of 28 <u>M</u>	3.0
HNO_3	63	83 ml of 15.6 <u>M</u>	1.3

The radioactivity of actual waste would be 100 curies/gal; the energy emitted would be 1.71 Btu/hr. gal or 0.5 watt/gal. Preparation of STR type waste solution from the chemical compounds listed above is not difficult. However, the zirconium hydroxide may be more or less difficult to dissolve, depending upon the temperature used in drying.

Preparation of synthetic waste from zirconium fluoride was unsatisfactory because of the difficulty of dissolving ZrF_4 in the small excess of hydrofluoric acid.* An alternate procedure for preparation, therefore, is to dissolve 50 g of Zircaloy-2 in 340 ml of 2.37 M HF to provide the zirconium and tin. To this is added the nitric acid and water solutions of the other salts, and the entire solution diluted to one liter volume. The nitric acid and salts should not be added until all the Zircaloy-2 is in solution. The hydrofluoric acid should be added to the Zircaloy slowly. Since aqueous hydrofluoric acid readily attacks glass, a polyethylene or

* A word of warning about aqueous hydrofluoric acid is in order. If it contacts the skin, no sensation of pain can be felt at first; after several hours a slight tingling occurs, which rapidly becomes worse until the pain is very severe. A tough, sandy coagulated area of skin forms at the burn, and the destruction of tissue under this area may continue for several days. The burned area heals very slowly.

magnesium beaker which may be heated on a hot plate must be used. Although warm water will attack magnesium, warm HF will not do so because of the formation of a protective film. The waste must be stored in a polyethylene bottle.

Zirconium as a powder or sponge metal has a very great tendency to spontaneous ignition in air at low temperatures. The combustion hazard is greatly increased when 16 per cent or less moisture is present. The U. S. Bureau of Mines reported that dry zirconium powder (90% of 6 μ size or smaller) can ignite explosively and spontaneously when dispersed in air at room temperature. No hazard exists during the dissolution of zirconium or Zircaloy-2 if hydrofluoric acid is added slowly at first. Care should be taken because of displacement of hydrogen by the metal.

The actual composition of first extraction column waste would be:

Volume	2500 gal	Sn ⁺⁺	0.012 <u>M</u>
Specific gravity	1.216	Cr ³⁺	0.016 <u>M</u>
H ⁺	2.14 <u>M</u>	NO ₃ ⁻	3.59 <u>M</u>
Al ³⁺	0.75 <u>M</u>	F ⁻	3.00 <u>M</u>
Zr ⁴⁺	0.55 <u>M</u>		

The radioactivity would be 100 curies/gal and the energy emitted would be 1.71 Btu/hr. gal or 0.5 watt/gal.

5.0 WASTE FROM PROCESSING OF STAINLESS STEEL-UO₂ FUEL ELEMENTS

The Darex process is used in processing uranium fuel elements containing stainless steel, like types 304 and 347, which readily dissolved

in dilute aqua regia. The chloride is removed to a level of 30 ppm by distillation before solvent extraction of the uranium with TBP. Examples of fuel elements to be processed by this method are the Army Package Power Reactor (APPR), which has a stainless steel jacket and sintered enriched uranium dioxide - stainless steel core; and the Yankee Atomic Power Reactor, which has a stainless steel jacket and slightly enriched UO_2 core.

Simulated waste is prepared from the following chemicals:

<u>Chemical</u>	<u>Molecular Wt.</u>	<u>Amt. Used per Liter</u>	<u>Final Conc., M</u>
$Ni(NO_3)_2 \cdot 6H_2O$	291	19.8 g	0.075
$Fe(NO_3)_3 \cdot 9H_2O$	404	275 g	0.68
$Cr(NO_3)_3 \cdot 9H_2O$	400	64.0 g	0.16
$Al(NO_3)_3 \cdot 9H_2O$	375	46.1 g	0.123
HCl	36.5	1 ml of 1.0 M	0.001
HNO_3	63	189 ml of 15.6 M	2.95

The nitrate salts readily dissolve in water. The acid should be diluted, mixed with the solution of salts, and diluted to one liter. The appearance of the waste solution will be dark blue-black because of the chromium nitrate.

The composition of an actual extraction column aqueous raffinate would be:

Volume	0.5 gal/g U-235	Cr^{3+}	0.16 M
Specific gravity	1.28	Al^{3+}	0.123 M
H^+	2.94 M	NO_3^-	6.1 M
Ni^{2+}	0.075 M	Cl^-	30 ppm
Fe^{3+}	0.68 M		

The radioactivity would be about 1000 curies/gal and the energy would be 17.6 Btu/hr. gal.

6.0 WASTE FROM PROCESSING OF STAINLESS STEEL- UO_2 -MgO FUEL ELEMENTS

The fuel element of the Submarine Intermediate Reactor (SIR) of the Sea Wolf has a stainless steel cladding over a sintered UO_2 -MgO core. The stainless steel is dissolved in boiling 4-6 M H_2SO_4 , and nitric acid is then added to dissolve the core. The nitrate-sulfate solution is extracted with 10 per cent tributyl phosphate to recover the uranium.

Simulated first extraction column aqueous waste is prepared from the following chemicals:

<u>Chemical</u>	<u>Molecular Wt.</u>	<u>Amt. Used per Liter</u>	<u>Final Conc., M</u>
$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	404	28 g	0.07
$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	291	2.3 g	0.008
$\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	400	13 g	0.018
$\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	256	4.4 g	0.017
$\text{Mn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	119	.119 g	0.001
H_2SO_4	98	28 ml of 18 M	0.51
HNO_3	63	131 ml of 16 M	2.35

All these salts go into an aqueous solution readily. After the salts are dissolved the acids are added, and the solution is diluted to volume. It is best to weigh out each salt and dissolve it separately in a small volume of water before mixing them together. The color of the solution is dark blue-black.

Actual waste would have the following composition:

Volume	0.35 gal/g U-235	Mg ⁺⁺	0.017 <u>M</u>
Specific gravity	1.09	Mn ⁺	0.001 <u>M</u>
H ⁺	3.52 <u>M</u>	NO ₃ ⁻	2.8 <u>M</u>
Fe ³⁺	0.066 <u>M</u>	SO ₄ ⁻⁻	0.51 <u>M</u>
Cr ³⁺	.018 <u>M</u>		
Ni ²⁺	0.008 <u>M</u>		

The radioactivity would be 1500 curies/gal, and the energy emitted 25.0 Btu/hr. gal or 7.5 watts/gal.

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APPENDIX

In the power reactor waste types cited, no mention is made of the concentration of the many important radioactive fission products present or of the contribution to the total activity represented by a particular radioactive fission product for some given time after fission. This would be of general interest in making up a tagged fuel reprocessing waste solution. Therefore, a copy of the graph by Koontz and Jarrett⁽⁹⁾ is shown in Figure I. The total radioactivity of the spent fuel in curies, as a function of cooling time for various periods of operation of a natural uranium reactor at a one-watt-power level, is shown in Figure I. The simple equation for this curve is:

$$\text{Total F. P. activity in curies} = 1.44 P \left[(t - T_0)^{-0.2} - t^{-0.2} \right]$$

P = power level in watts

T₀ = number of days exposure in reactor

t = total number of days cooling exposure

Figure II is a schematic flow sheet for a typical first cycle solvent extraction process. In this flow sheet arrangements are shown by which the three principal fractions, uranium, plutonium, and the fission products, may be separated from each other. The flow sheet also indicated the sources of radioactive waste products.

FIRST CYCLE OF A TYPICAL SOLVENT-EXTRACTION PROCESS

Figure 2.

Figure 1. Activity of Fission Products in Natural Uranium fuel, Following T Days Exposure in Reactor.

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