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QUARTERLY PROGRESS REPORT
STUDIES ON MANAGEMENT
OF SELECTED WASTES
JANUARY THROUGH MARCH 1974

Battelle

Pacific Northwest Laboratories
Richland, Washington 99352

APRIL 1974

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Waste Disposal
and Processing

QUARTERLY PROGRESS REPORT
STUDIES ON MANAGEMENT OF SELECTED WASTES
January through March 1974

Compiled by
K. J. Schneider
Nuclear Waste Technology Department

April 1974

BATTELLE
PACIFIC NORTHWEST LABORATORIES
RICHLAND, WASHINGTON 99352

FOREWORD

This is the second quarterly progress report for studies on management of non-high-level wastes being performed by Battelle-Northwest at the AEC's Pacific Northwest Laboratory.

Studies included in this report are:

- Decontamination and densification of chop-leach cladding residues,
- Waste treatment and handling processes, and
- Tritium separation and fixation.

The previous report in this series was BNWL-1806, October through December 1973.

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SUMMARY

DECONTAMINATION AND DENSIFICATION OF CHOP LEACH CLADDING RESIDUES

A literature review of aqueous, molten salt, and melt flux decontamination processes has indicated that waste stream minimization, ease of operation, and decontamination efficiency can probably be optimized by a modified aqueous decontamination process followed by melting. The reuse of metal from decontamination would be possible after long cooling times but economics are not evaluated. A piece of highly irradiated zirconium fuel cladding was obtained to analytically determine transuranic element distribution. The melting of 10, 15, and 20% stainless steel-zirconium alloys in a graphite crucible at 1100°C was demonstrated on a small scale. Temperature arrests in heating and cooling conform to those expected from the phase diagrams.

WASTE TREATMENT AND HANDLING PROCESSES

Equipment required for the scoping studies on pyrolysis of wastes was fabricated and installed during the reporting period. Experimental plans for the scoping studies were completed and experiments at 700°C using six different waste components were conducted. Conceptual design of the experimental facility (pilot plant) was started.

TRITIUM SEPARATION AND FIXATION

Solvent extraction studies have shown that competitive factors negate most of the separation which might be expected. A few studies are continuing using strongly basic solvents.

Calculations have permitted choice of an excitation scheme for preferentially exciting H₂O vapor and causing it to react. A reaction with methane or substituted methane will be tested. The chances appear good that similar excitation paths can be found for other water molecules such as HTO or DHO.

A report on the technology of tritium fixation was issued. Scouting experiments were performed on an organic polymer synthesis.

A report on parametric studies of tritium separation cascades was issued. Computer programs were employed to calculate separative duty as a function of isotopic parameters.

DECONTAMINATION AND DENSIFICATION OF
CHOP-LEACH CLADDING RESIDUES

R. L. Dillon and R. S. Kemper

Fuel element cladding residues from the chop-leach head end process constitute a low density, high surface area metallic waste with a substantial actinide element contamination. The objective of this study is to determine the feasibility of and to develop methods for removal of long half-life transuranics from the cladding residues and for consolidation of the zirconium and stainless steel residues. The study involves:

1. *Selection and laboratory testing of methods for transuranic removal by*
 - a) *aqueous reagents*
 - b) *molten salt reagents*
 - c) *fluxing during melting.*
2. *Consolidation of the fuel element hulls by melting and casting Zircaloy, stainless steel, Inconel or alloys of the three.*
3. *Testing and evaluating the consolidated fuel hulls for long-term waste management.*
4. *Absorption and fixation of tritium into the waste ingots.*
5. *Consolidation of transuranics removed.*

PROCESS SELECTION - B. Griggs

The detailed review of potential decontamination and melting processes for irradiated, fuel contaminated Zircaloy, stainless steel, and Inconel wastes from the chop-leach head end step of fuel reprocessing plants was continued and a report written.⁽¹⁾ The initial phase of this review was previously reported⁽²⁾ and is only summarized here.

Based on typical current fuel designs for light water reactors, the waste stream consists of Zircaloy with 29% and 21% stainless steel plus Inconel for the Pressurized Water Reactor (PWR) and Boiling Water Reactor (BWR), respectively. The trend toward reducing neutron absorbers in the fuel elements will raise the percentage of Zircaloy in the future. The hull

waste stream is contaminated by undissolved fuel particles, fuel-zirconium dioxide corrosion product layer interaction products, and irradiated uranium and thorium impurities in the original metal. Based on 0.1% of the original fuel as contamination on the hull waste stream, overall decontamination factors for the transuranic elements of $10^4 - 10^5$ will be necessary to reduce the Zircaloy-stainless steel-Inconel alloy below 10 nCi/g of transuranic contamination.

The examination of the three proposed decontamination methods, removal by aqueous reagents, molten salts, and melt fluxing, all showed deficiencies when considering the desired objectives of high decontamination factor and minimization of the total waste stream.

Several dilute aqueous solutions containing fluoride are excellent solvents for fuel particles and etchants for Zircaloy, stainless steel, and Inconel.^(3,4) However, none of the aqueous processes is known to dissolve the heavy, corrosion resistant, oxide coating on the Zircaloy (formed by operation in the reactor) without severe attack on the exposed metal. Molten fluoride salts flushed with hydrogen fluoride at about 600°C are known to dissolve the zirconium oxide layer,⁽⁵⁾ metal, fuel oxides, or fission products. For effective use, these reagents require inert atmospheres and must remain anhydrous. Such limitations complicate their use. The use of fluxes on the molten alloy to extract more active impurities from the melt have been examined for molten uranium and other liquid metal systems.^(6,7,8) The removal of transuranium elements at the ppb level from molten zirconium will require a flux with no transuranic contamination and even then will probably produce decontamination factors of only about 10. The use of solid absorbents such as graphite, yttrium oxide, zirconium oxide, or beryllium oxide might be more effective than a flux but will increase the waste stream size substantially unless they can be regenerated and used repeatedly or used as thin coatings on melting vessels.

Detailed examination of the three processes from the standpoint of waste stream minimization and ease of operation shows that the major problem is removing the zirconium dioxide corrosion product layer. Although

the layer can be removed in a fused salt bath, this process will complicate handling procedures and generate a large, hard to recover, waste stream of salt which will cling to the high surface area hulls in large quantities.

Currently it is believed that a modified aqueous process, followed by melting, has greater all around promise of meeting all of the objectives. An aqueous process can be designed to employ volatile (nitric-hydrofluoric acid) or oxidizable (oxalic acid-hydrofluoric acid-hydrogen peroxide) solutions which can be purified substantially by distillation and the residues ignited to oxides and fluorides of the removed contamination, zirconium and stainless steel. Aqueous processes are easy to use simply requiring open vessels for treatment.

One possibility of using the aqueous process would be to pretreat the hulls in a gaseous fluoride, probably hydrogen fluoride, to convert the oxide corrosion product to a labile coating of ZrOF_2 or ZrF_4 which can be removed by the aqueous process. One process investigated for completely oxidizing the cladding operated at about 550°C in oxygen-hydrogen fluoride environment.⁽⁹⁾ Information on this process indicates that hydrogen fluoride is capable of making the zirconium dioxide layer nonprotective at 550°C . This temperature (550°C) has also been reported⁽¹⁰⁾ as the temperature at which hydrogen fluoride reacts with zirconium dioxide to form ZrF_4 and is in the temperature range that hydrogen fluoride reacts with zirconium dioxide in molten alkali fluoride baths.⁽⁵⁾ The vapor pressure of the ZrF_4 at 550°C is about 0.03 mm Hg. Thus, ZrF_4 will not be substantially removed from the basic metal by volatilizing and may slow down the reaction of the volatile fluoride with the base metal.

The waste produced from the gas reaction will depend on the fluoride chemical used. With hydrogen fluoride, water and hydrogen will be produced which will have to be separated from the gas before it can be recycled. The water produced in the ZrO_2 -HF reaction will probably have some contamination from tritium and other fission products that have volatile fluorides at the reaction temperature. If the hydrogen fluoride or other fluoride is not recycled, it could be used to make up the fluoride concentration of the

aqueous etch. The separation of alloys for separate melting would not affect the process applicability. The surface decontaminated and etched material would then be subjected to melting and melt decontamination as desired.

Reuse of Metals

The possible treatment sequence for fuel element cladding and hardware was examined logically on a broader basis than previously, in connection with an evaluation of the possible reuse of metal components. Table 1 and accompanying notes briefly examine the various treatment possibilities for fuel element cladding and hardware. The economics of various schemes was not examined.

The Zircaloy-2 cladding of fuel elements was examined by the ORIGEN⁽¹¹⁾ computer code to determine the activation and fission product inventory after a typical (33,000 MWD/MTU) exposure. The calculation assumes 254 kilograms of Zircaloy-2 per metric ton of uranium in fuel including the "normal" estimated impurities in the Zircaloy. The impurities were estimated by examination of ASTM-B353-71 standard for Zircaloy-2 tubing for nuclear service and from a few specific analyses of Zircaloy material on hand. The effect of uranium and thorium impurities were examined separately. The effect of uranium concentration was linear with respect to the anticipated activation and fission product residues. Increasing the fluence to be more typical for the cladding was also examined and found to have a modest effect on the activation products. The expected activation products in the Zircaloy-2 arising from the metal with all its impurities except uranium and thorium are shown in Table 2 and 3. The fission products from 1 ppm uranium impurity are shown in Table 4 and the activation products from 1 ppm uranium impurity are shown in Table 5. Table 6 shows the activation products expected from 1 ppm of thorium impurity.

Examination of Table 3 shows that even after 100 years cooling Ni-63 and Zr-93-Nb-93 are still present at levels of 16 $\mu\text{ci/g}$ and 0.8 $\mu\text{ci/g}$, respectively. These isotopes are soft beta or gamma emitters and would not present any shielding problems during remanufacture. The reuse of the

TABLE 1. Treatment Logic for Decontamination and Densification of Fuel Hulls

TABLE 1. (Contd) Notes for Treatment Logic for Decontamination and Densification of Fuel Hulls

GENERAL

The changes that may take place in the design parameters of the future are uncertain. The alloys of construction and their relative proportions will probably change as far as practical toward lower neutron absorbing materials in thermal reactors. The most likely base material will probably remain zirconium, however, the specific zirconium alloy may change. The stainless steel and Inconels can probably be used as alloy stock for future alloy of this type.

1. The use of the chop-leach head-end process has been based on the anticipated low cost of separate treatment and burial of cladding hulls and the economy of using stainless steel dissolvers and conventional reprocessing schemes. If the cost of hull disposal is substantially increased or the cost of tritium removal from waste streams requires the alternative head-end processes such as liquid metal clad dissolution, gas phase clad oxidation, or even aqueous dissolving of the clad, the fuel hull waste will be combined with other waste.

2. It is assumed there is currently no alternative to storage for waste streams heavily contaminated with transuranic elements.

3. The disposal of a chemically reactive, high volume, transuranic-contaminated waste streams is assumed to be currently unacceptable.

4. The mixture of stainless steel, Inconel and Zircaloy will have to be segregated into three waste streams for processing. This separation can probably be done magnetically.

5. No Reuse - the decontamination of the cladding hulls is for the purpose of removing transuranic contamination to such a degree (<10 nci/g) that the resulting product can be more easily stored. The resulting transuranic and non-transuranic waste streams must be minimized to produce only a small increase in total mass and a large reduction in total volume and chemical reactivity. The transuranic contaminated waste stream should be a very small fraction of the total. The process considered most likely to accomplish this result at present is a gas phase oxide metathesis followed by an

aqueous bath for decontamination and descaling followed by final decontamination by a calcium fluoride slag during melting. The salts will have to be purified and reused to reduce waste volume. If no decontamination is performed, the waste stream will presumably require permanent storage of the total waste stream.

6. Reuse - the reuse of the metallic waste stream requires that no impurities detrimental to reuse be added to the waste stream. Thus, the different alloys of the waste stream must be handled independently, possibly by different processes. The fact that all of the stream will be stored may reduce the decontamination factors from those required for nontransuranic waste.

The decontamination process for the Zircaloy will be the same as nonreuse case because it is desirable to remove the oxide from the Zircaloy prior to melting. The stainless steel may not have transuranic contamination and in any event might be processed by aqueous decontamination processes. The Inconel similarly may respond satisfactorily to aqueous processes. The waste stream must eventually be decontaminated prior to reuse, thus this decision is based on intervening storage problems. The amount of fuel contamination is estimated at less than 0.3% of the cladding hulls.

7. The estimated density of current cladding hulls is 1.1 kg/liter with surface to mass ratios of 4.5 cm²/g. This large surface to mass ratio makes storage of the Zircaloy material sensitive to combustion unless carefully controlled environments are employed. The easiest way to store the low density hulls would be under water or cast in concrete which will keep the temperature low. The high

surface area and the fact that the fuel contamination is largely on the surface of the hulls would promote the leaching of transuranics and fission products by water and thus they must be in a closed system which itself will become contaminated with transuranic elements in time.

8. Inasmuch as this material is not to be recovered for reuse, the separation of the storage medium and the hulls does not have to be considered.

9. The storage of this material must consider the separation of the hulls from the storage medium for eventual decontamination. The corrosion in storage must be carefully considered because material that is converted to oxide will probably not be recovered.

10. The melting of an oxide covered mixture of Zircaloy, stainless steel and Inconel may present a few problems in volatilization of radioactivity such as tritium and other constituents volatile at about 1100°C. Any slag formed on the melt would constitute a separate transuranic waste stream. The melting equipment and materials of construction would be simpler than for a straight zirconium melting operation at 1850°C minimum.

11. The presently proposed decontamination involves the treatment of the oxide covered metal in a volatile fluoride to transform the oxide into a fluoride or oxyfluoride so that it can be removed in a subsequent dilute aqueous process.

12. The problems of volatile radioactive elements from the melt would be similar but more severe than in note 10 because the stainless steels would have to be heated to about 1550°C and the Zircaloy to 1850°C.

The oxides and fuel contamination on the surface would become part of the melt, which may affect the corrosion resistance of the stored ingot. In any event the oxide and other impurities would have to be removed during recovery and probably require extensive processing to evolve a usable alloy in the case of zirconium. It will be necessary to use a cold wall type of melting device to avoid contamination of the zirconium with crucible material.

13. The transuranic contaminated waste from the decontamination process is the dehydrated residue of fluorides and oxides of dissolved zirconium, stainless steel and Inconel. The transuranic waste stream from the melting processes would consist of slags and fluxing salts (CaF₂) or electrolysis products of the flux if it can be adequately purified by electrolysis.

14. The corrosion resistance of the oxygen and transuranic element contaminated Zircaloy will not be as good as the original Zircaloy.

15. The advantages of densification are reduced storage volume, reduced shipping volume and shielding, reduced corrosion surface area, better heat transfer, and some decontamination. The only disadvantage is that the material may have to be remelted later to change its alloying before reuse.

16. The melting of a stainless steel-zirconium-Inconel alloy at about 1100°C has been demonstrated. This can be done in a graphite crucible by induction and hopefully under a suitable flux to achieve a final D.F. of about 10 for the transuranic contamination which will be present in the alloy from original uranium and thorium contamination.

17. The ability to do a final decontamination on the melt to remove uniformly distributed transuranics and not add objectionable contamination to the melt from the standpoint of final reuse is uncertain. The higher melting temperatures for the separate alloys coupled with a desire to keep things like carbon, fluorine, and oxygen out of the melt may restrict melting techniques to a cold wall device without a flux. Decontamination of the melt under such restrictions may be minimal.

Alloying the melt to produce an ingot that can be directly processed after storage may be desirable. Similarly it may be desirable to cast the alloys in an ingot that can be directly used in subsequent melting and alloying processes such as the consumable arc furnace.

18. The alloy stability and corrosion resistance of the three alloys which have been contaminated by their surface oxide and fuel contamination is not known.

19. The removal of large (0.3%) amounts of fuel contamination during melt processing will require a very high D.F. It is doubtful this high a decontamination factor can be achieved to allow subsequent use of the material as non-transuranic contaminated which will complicate rather extensive subsequent processing.

20. Even if one can achieve an acceptable decontamination in 19, the oxygen and other impurities in the melt must be removed before reuse. A technique for doing this, short of complete reprocessing for zirconium, is not apparent. Stainless steels and Inconels may be deoxygenated by suitable oxygen gettering techniques.

21. The storage of densified material will present few problems. The storage of the decontaminated hulls presents a problem in corrosion and inflammability which will require cool storage and oxide removal before melting.

22. Storage of these ingots should not present major problems unless the guidelines change or the decontamination cannot be achieved to meet the current standards. The storage of hulls will require cool, high volume storage and may contain more than 10 nCi/g of transuranic contamination.

23. & 24. The refabrication of these alloys will have to be done in a radioactive facility even if the transuranic content of these alloys has been reduced below 10 nCi/g. The attached tables of ORIGEN computer runs for a Zircaloy-2 with normal expected impurities show several long half life materials such as ⁶³Ni and ⁹³Zr which though they present little dose rate problem will require control of atmosphere, etc., to prevent ingestion or inhalation. It would thus be desirable to do as little refabrication work as can be done to acquire a satisfactory product. For example, if directly extrudable billets were made in 18 of the proper alloy, the tubing could be extruded with a minimum of handling. The actual fabrication of fuel will require radioactive facilities in any event and the only changes will be in the handling of the final fuel element.

25. The waste streams from fuel manufacture will be slightly radioactive from the cladding. The insolubility of zirconium dioxide in reactor coolant should not add any new radioactivity to the reactor system.

TABLE 2. Activation Products From Zircaloy-2 Plus Impurities

Basis: Irradiation at 2.88×10^{13} n/cm²/sec avg. for 1100 days

ELEMENT CONCENTRATIONS, GRAMS BASIS = MT OF U CHARGED TO REACTOR													
	CHARGE	DISCHARGE	1.MO	3.MO	6.MO	12.MO	36.MO	60.MO	120.MO	1200.MO	12000.MO	120000.MO	
H	3.15E+01	3.15E+01	3.15E+01	3.15E+01	3.15E+01	3.15E+01	3.15E+01	3.15E+01	3.15E+01	3.15E+01	3.15E+01	3.15E+01	
HE	0.	2.00E-02	2.00E-02	2.00E-02	2.00E-02	2.00E-02	2.00E-02	2.00E-02	2.00E-02	2.00E-02	2.00E-02	2.00E-02	
LI	0.	9.69E-03	9.69E-03	9.69E-03	9.69E-03	9.69E-03	9.69E-03	9.69E-03	9.69E-03	9.69E-03	9.69E-03	9.69E-03	
BE	0.	6.69E-05	6.69E-05	6.69E-05	6.69E-05	6.69E-05	6.69E-05	6.69E-05	6.69E-05	6.69E-05	6.69E-05	6.69E-05	
B	7.56E-02	6.36E-02	6.36E-02	6.36E-02	6.36E-02	6.36E-02	6.36E-02	6.36E-02	6.36E-02	6.36E-02	6.36E-02	6.36E-02	
C	3.42E+01	3.43E+01	3.43E+01	3.43E+01	3.43E+01	3.43E+01	3.43E+01	3.43E+01	3.43E+01	3.43E+01	3.43E+01	3.43E+01	
N	1.02E+01	1.02E+01	1.02E+01	1.02E+01	1.02E+01	1.02E+01	1.02E+01	1.02E+01	1.02E+01	1.02E+01	1.02E+01	1.02E+01	
O	2.54E+02	2.54E+02	2.54E+02	2.54E+02	2.54E+02	2.54E+02	2.54E+02	2.54E+02	2.54E+02	2.54E+02	2.54E+02	2.54E+02	
F	0.	2.13E-07	2.13E-07	2.13E-07	2.13E-07	2.13E-07	2.13E-07	2.13E-07	2.13E-07	2.13E-07	2.13E-07	2.13E-07	
NE	0.	2.18E-09	2.18E-09	2.18E-09	2.18E-09	2.18E-09	2.18E-09	2.18E-09	2.18E-09	2.18E-09	2.18E-09	2.18E-09	
NA	0.	2.71E-08	1.69E-10	1.69E-10	1.69E-10	1.69E-10	1.69E-10	1.69E-10	1.69E-10	1.69E-10	1.69E-10	1.69E-10	
MG	0.	1.02E-04	1.02E-04	1.02E-04	1.02E-04	1.02E-04	1.02E-04	1.02E-04	1.02E-04	1.02E-04	1.02E-04	1.02E-04	
AL	8.88E+00	8.88E+00	8.88E+00	8.88E+00	8.88E+00	8.88E+00	8.88E+00	8.88E+00	8.88E+00	8.88E+00	8.88E+00	8.88E+00	
SI	1.53E+01	1.53E+01	1.53E+01	1.53E+01	1.53E+01	1.53E+01	1.53E+01	1.53E+01	1.53E+01	1.53E+01	1.53E+01	1.53E+01	
P	0.	9.89E-05	9.89E-05	9.89E-05	9.89E-05	9.89E-05	9.89E-05	9.89E-05	9.89E-05	9.89E-05	9.89E-05	9.89E-05	
S	0.	2.03E-08	2.10E-08	2.12E-08	2.12E-08	2.12E-08	2.12E-08	2.12E-08	2.12E-08	2.12E-08	2.12E-08	2.12E-08	
CL	0.	1.17E-14	1.17E-14	1.17E-14	1.17E-14	1.17E-14	1.17E-14	1.17E-14	1.17E-14	1.17E-14	1.17E-14	1.17E-14	
AR	0.	3.48E-11	3.48E-11	3.48E-11	3.48E-11	3.48E-11	3.48E-11	3.48E-11	3.48E-11	3.48E-11	3.48E-11	3.48E-11	
K	0.	1.18E-12	1.14E-12	1.14E-12	1.14E-12	1.14E-12	1.14E-12	1.14E-12	1.14E-12	1.14E-12	1.14E-12	1.14E-12	
CA	0.	1.60E-05	1.60E-05	1.60E-05	1.60E-05	1.60E-05	1.60E-05	1.60E-05	1.60E-05	1.60E-05	1.60E-05	1.60E-05	
SC	0.	4.74E-06	3.51E-06	2.21E-06	1.17E-06	4.53E-07	2.59E-07	2.59E-07	2.59E-07	2.59E-07	2.59E-07	2.59E-07	
TJ	6.33E+00	6.33E+00	6.33E+00	6.33E+00	6.33E+00	6.33E+00	6.33E+00	6.33E+00	6.33E+00	6.33E+00	6.33E+00	6.33E+00	
V	0.	2.98E-01	3.05E-01	3.10E-01	3.11E-01	3.11E-01	3.11E-01	3.11E-01	3.11E-01	3.11E-01	3.11E-01	3.11E-01	
CR	2.54E+02	2.54E+02	2.54E+02	2.54E+02	2.54E+02	2.54E+02	2.54E+02	2.54E+02	2.54E+02	2.54E+02	2.54E+02	2.54E+02	
MN	6.38E+00	6.19E+00	6.19E+00	6.19E+00	6.20E+00	6.20E+00	6.22E+00	6.24E+00	6.25E+00	6.26E+00	6.26E+00	6.26E+00	
FE	3.43E+02	3.44E+02	3.44E+02	3.44E+02	3.44E+02	3.44E+02	3.44E+02	3.44E+02	3.44E+02	3.43E+02	3.43E+02	3.43E+02	
CO	2.54E+00	2.51E+00	2.51E+00	2.50E+00	2.49E+00	2.47E+00	2.42E+00	2.38E+00	2.31E+00	2.24E+00	2.25E+00	2.30E+00	
NI	1.40E+02	1.40E+02	1.40E+02	1.40E+02	1.40E+02	1.40E+02	1.40E+02	1.40E+02	1.40E+02	1.40E+02	1.40E+02	1.40E+02	
CU	6.36E+00	6.29E+00	6.29E+00	6.29E+00	6.29E+00	6.30E+00	6.30E+00	6.30E+00	6.30E+00	6.37E+00	6.43E+00	6.43E+00	
ZN	0.	3.40E-02	3.40E-02	3.40E-02	3.40E-02	3.40E-02	3.40E-02	3.40E-02	3.40E-02	3.40E-02	3.40E-02	3.40E-02	
SR	0.	2.22E-01	2.21E-01	2.21E-01	2.21E-01	2.20E-01	2.20E-01	2.20E-01	2.20E-01	2.20E-01	2.20E-01	2.20E-01	
Y	0.	6.61E-03	2.84E-03	1.38E-03	4.71E-04	5.47E-05	1.15E-04	1.48E-09	1.31E-09	1.42E-10	3.23E-20	0.	
ZR	2.49E+05	2.49E+05	2.49E+05	2.49E+05	2.49E+05	2.49E+05	2.49E+05	2.49E+05	2.49E+05	2.49E+05	2.49E+05	2.49E+05	
NR	0.	7.34E-01	6.85E-01	4.66E-01	2.07E-01	3.23E-02	9.74E-05	1.22E-04	2.16E-04	1.92E-03	1.90E-02	1.89E-01	
MO	0.	1.20E+01	1.24E+01	1.31E+01	1.37E+01	1.41E+01	1.41E+01	1.41E+01	1.41E+01	1.41E+01	1.41E+01	1.41E+01	
TC	0.	3.24E-10	3.31E-10	3.31E-10	3.31E-10	3.31E-10	3.31E-10	3.31E-10	3.31E-10	3.31E-10	3.30E-10	3.21E-10	
CD	9.39E-03	1.63E-07	1.60E-07	1.60E-07	1.60E-07	1.60E-07	1.60E-07	1.60E-07	1.60E-07	1.60E-07	1.60E-07	1.60E-07	
SN	3.65E+03	3.65E+03	3.65E+03	3.65E+03	3.65E+03	3.65E+03	3.65E+03	3.65E+03	3.65E+03	3.65E+03	3.65E+03	3.65E+03	
SB	0.	1.25E+00	1.25E+00	1.25E+00	1.24E+00	1.24E+00	1.23E+00	1.22E+00	1.21E+00	1.21E+00	1.22E+00	1.22E+00	
TE	0.	1.84E-02	1.93E-02	2.12E-02	2.37E-02	2.82E-02	4.13E-02	4.92E-02	5.76E-02	6.08E-02	6.08E-02	6.08E-02	
TA	0.	2.03E-04	2.09E-04	2.18E-04	2.29E-04	2.40E-04	2.48E-04	2.48E-04	2.48E-04	2.48E-04	2.48E-04	2.48E-04	
W	1.27E+01	1.12E+01	1.12E+01	1.12E+01	1.12E+01	1.12E+01	1.12E+01	1.12E+01	1.12E+01	1.12E+01	1.12E+01	1.12E+01	
TOTALS	2.54E+05	2.54E+05	2.54E+05	2.54E+05	2.54E+05	2.54E+05	2.54E+05	2.54E+05	2.54E+05	2.54E+05	2.54E+05	2.54E+05	

TABLE 3. Activation Products From Zircaloy-2 Plus Impurities
Basis: Irradiation at 2.88×10^{13} n/cm²/sec avg. for 1100 days

		NUCLIDE RADIOACTIVITY, CURIES											
		BASIS = MT OF U CHARGED TO REACTOR											
	CHARGE	DISCHARGE	1.MO	3.MO	6.MO	12.MO	36.MO	60.MO	120.MO	1200.MO	12000.MO	120000.MO	
H 3	0.	5.37E-03	5.35E-03	5.30E-03	5.22E-03	5.08E-03	4.54E-03	4.05E-03	3.06E-03	1.92E-05	0.	0.	
BE 10	0.	6.50E-08	6.50E-08	6.50E-08	6.50E-08	6.50E-08	6.50E-08	6.50E-08	6.50E-08	6.50E-08	6.50E-08	6.48E-08	
C 14	0.	2.08E-01	2.08E-01	2.08E-01	2.08E-01	2.08E-01	2.08E-01	2.08E-01	2.07E-01	2.05E-01	1.84E-01	6.19E-02	
NA 24	0.	2.34E-01	5.11E-16	0.	0.	0.	0.	0.	0.	0.	0.	0.	
P 32	0.	2.60E-04	5.95E-05	3.11E-06	3.72E-08	5.32E-12	2.22E-27	9.28E-43	0.	0.	0.	0.	
AR 39	0.	1.48E-17	1.47E-17	1.47E-17	1.47E-17	1.47E-17	1.46E-17	1.46E-17	1.44E-17	1.14E-17	1.12E-18	9.49E-29	
K 43	0.	1.41E-07	2.15E-17	4.97E-37	0.	0.	0.	0.	0.	0.	0.	0.	
CA 45	0.	1.09E-03	9.62E-04	7.45E-04	5.07E-04	2.36E-04	1.09E-05	5.09E-07	2.37E-10	0.	0.	0.	
CA 47	0.	6.78E-06	6.44E-08	5.80E-12	4.95E-18	3.61E-30	0.	0.	0.	0.	0.	0.	
SC 46	0.	1.44E-01	1.12E-01	6.76E-02	3.18E-02	7.03E-03	1.68E-05	4.02E-08	1.13E-14	0.	0.	0.	
SC 47	0.	2.32E-01	4.94E-04	2.26E-09	4.20E-17	1.49E-29	0.	0.	0.	0.	0.	0.	
SC 48	0.	2.22E-02	2.18E-07	2.11E-17	2.01E-32	0.	0.	0.	0.	0.	0.	0.	
CR 51	0.	1.20E+03	5.63E+02	1.23E+02	1.27E+01	1.33E-01	1.64E-09	2.01E-17	3.36E-37	0.	0.	0.	
MN 54	0.	2.16E+01	2.02E+01	1.75E+01	1.42E+01	9.37E+00	1.76E+00	3.31E-01	5.08E-03	0.	0.	0.	
FE 55	0.	1.73E+02	1.69E+02	1.62E+02	1.51E+02	1.32E+02	7.77E+01	4.56E+01	1.20E+01	4.55E-10	0.	0.	
FE 59	0.	1.35E+01	8.43E+00	3.30E+00	8.09E-01	4.85E-02	6.29E-07	8.16E-12	4.94E-24	0.	0.	0.	
CO 58	0.	1.33E+02	9.86E+01	5.45E+01	2.24E+01	3.80E+00	3.13E-03	2.58E-06	5.01E-14	0.	0.	0.	
CO 60	0.	3.01E+02	2.98E+02	2.91E+02	2.82E+02	2.64E+02	2.03E+02	1.56E+02	8.06E+01	5.69E-04	0.	0.	
NI 59	0.	5.47E-02	5.47E-02	5.47E-02	5.47E-02	5.47E-02	5.47E-02	5.47E-02	5.47E-02	5.47E-02	5.42E-02	5.02E-02	
NI 63	0.	8.60E+00	8.59E+00	8.58E+00	8.50E+00	8.53E+00	8.40E+00	8.28E+00	7.97E+00	4.05E+00	4.59E-03	0.	
CU 64	0.	1.71E+02	1.13E-15	0.	0.	0.	0.	0.	0.	0.	0.	0.	
ZN 65	0.	6.86E-02	6.30E-02	5.30E-02	4.09E-02	2.44E-02	3.09E-03	3.91E-04	2.23E-06	0.	0.	0.	
SR 89	0.	3.77E+01	2.51E+01	1.12E+01	2.30E+00	2.90E-01	1.71E-05	1.01E-09	2.69E-20	0.	0.	0.	
SR 90	0.	9.09E-04	9.07E-04	9.03E-04	8.98E-04	8.87E-04	8.44E-04	8.03E-04	7.10E-04	7.71E-05	1.76E-14	0.	
Y 90	0.	1.39E+03	5.09E-01	9.04E-04	8.98E-04	8.87E-04	8.44E-04	8.04E-04	7.10E-04	7.71E-05	1.76E-14	0.	
Y 91	0.	9.90E+01	6.92E+01	3.38E+01	1.15E+01	1.34E+00	2.43E-04	4.42E-08	1.97E-17	0.	0.	0.	
ZR 93	0.	1.05E-01	1.05E-01	1.05E-01	1.05E-01	1.05E-01	1.05E-01	1.05E-01	1.05E-01	1.05E-01	1.05E-01	1.05E-01	
ZR 95	0.	2.99E+04	2.16E+04	1.13E+04	4.26E+03	6.08E+02	2.51E-01	1.04E-04	3.62E-13	0.	0.	0.	
NB 93M	0.	7.29E-03	7.70E-03	8.52E-03	9.74E-03	1.21E-02	2.11E-02	2.93E-02	4.63E-02	1.04E-01	1.05E-01	1.05E-01	
NB 94	0.	1.30E-13	1.30E-13	1.30E-13	1.30E-13	1.30E-13	1.30E-13	1.30E-13	1.30E-13	1.30E-13	1.30E-13	1.30E-13	
NB 95	0.	2.89E+04	2.69E+04	1.83E+04	8.12E+03	1.27E+03	5.34E-01	2.21E-04	7.68E-13	0.	0.	0.	
NR 96	0.	1.34E+01	3.69E-09	2.78E-28	0.	0.	0.	0.	0.	0.	0.	0.	
MO 99	0.	3.67E-06	1.92E-04	5.22E-16	7.43E-26	1.50E-45	0.	0.	0.	0.	0.	0.	
TC 99M	0.	3.20E-06	1.83E-09	4.99E-16	7.10E-26	1.44E-45	0.	0.	0.	0.	0.	0.	
TC 99	0.	5.56E-12	5.70E-12	5.70E-12	5.70E-12	5.70E-12	5.70E-12	5.70E-12	5.70E-12	5.70E-12	5.68E-12	5.52E-12	
CD115	0.	1.68E-03	1.30E-07	7.81E-16	3.64E-28	0.	0.	0.	0.	0.	0.	0.	
SN117M	0.	1.16E+04	2.57E+03	1.26E+02	1.37E+00	1.62E-04	3.17E-20	6.20E-36	0.	0.	0.	0.	
SN119M	0.	2.27E+01	2.09E+01	1.76E+01	1.37E+01	8.25E+00	1.09E+00	1.44E-01	9.08E-04	0.	0.	0.	
SN121M	0.	3.36E-01	3.36E-01	3.35E-01	3.35E-01	3.33E-01	3.27E-01	3.21E-01	3.07E-01	1.35E-01	3.68E-05	0.	
SN121	0.	2.06E+03	1.48E-05	7.56E-22	0.	0.	0.	0.	0.	0.	0.	0.	
SN123	0.	4.35E-01	3.68E-01	2.62E-01	1.58E-01	5.74E-02	9.99E-04	1.74E-05	6.95E-10	0.	0.	0.	
SN125	0.	3.60E+00	3.82E-01	4.28E+03	5.10E-06	7.22E-12	0.	0.	0.	0.	0.	0.	
SB124	0.	4.65E+00	3.27E+00	1.62E+00	5.64E-01	6.84E-02	1.48E-05	3.19E-09	2.19E-18	0.	0.	0.	
SB125	0.	4.47E+01	4.38E+01	4.19E+01	3.93E+01	3.46E+01	2.07E+01	1.24E+01	3.43E+00	3.16E-10	0.	0.	
SB126	0.	2.18E-01	4.03E-02	1.38E-03	8.70E-06	3.48E-10	8.86E-28	2.26E-45	0.	0.	0.	0.	
TE125M	0.	1.62E+01	1.65E+01	1.66E+01	1.60E+01	1.43E+01	8.58E+00	5.13E+00	1.42E+00	1.31E-10	0.	0.	
TA182	0.	9.80E-02	8.15E-02	5.65E-02	3.26E-02	1.08E-02	1.33E-04	1.62E-06	2.69E-11	0.	0.	0.	
W181	0.	3.13E-01	2.70E-01	1.99E-01	1.27E-01	5.14E-02	1.38E-03	3.71E-05	4.38E-09	0.	0.	0.	
W185	0.	4.59E+01	3.46E+01	1.97E+01	8.48E+00	1.57E+00	1.83E-03	2.14E-06	1.00E-13	0.	0.	0.	
W187	0.	1.22E+03	7.66E-07	3.03E-25	0.	0.	0.	0.	0.	0.	0.	0.	
SUBTOT	0.	7.73E+04	5.25E+04	3.05E+04	1.30E+04	2.35E+03	3.23E+02	2.28E+02	1.06E+02	4.65E+00	4.53E-01	3.21E-01	
TOTAL	0.	7.90E+04	5.25E+04	3.05E+04	1.30E+04	2.35E+03	3.23E+02	2.28E+02	1.06E+02	4.65E+00	4.53E-01	3.21E-01	

TABLE 4. Fission Products to Be Expected From 1 ppm of Uranium Impurity in Zircaloy Fuel Cladding
Basis: Exposure at 2.88×10^{13} n/cm² avg. for 1100 days

ELEMENTAL ACTIVITY, CURIES BASIS = . MT OF U CHARGED TO REACTOR												
	CHARGE	DISCHARGE	1.MO	3.MO	6.MO	12.MO	36.MO	60.MO	120.MO	1200.MO	12000.MO	120000.MO
H	J.	1.25E-04	1.25E-04	1.24E-04	1.22E-04	1.18E-04	1.06E-04	9.45E-05	7.13E-05	4.47E-07	0.	0.
ZN	J.	5.03E-06	9.38E-11	3.26E-20	2.11E-34	0.	0.	0.	0.	0.	0.	0.
GA	0.	2.22E-04	1.35E-10	4.68E-20	3.04E-34	0.	0.	0.	0.	0.	0.	0.
GE	0.	1.77E-03	6.02E-24	0.	0.	0.	0.	0.	0.	0.	0.	0.
AS	0.	7.77E-03	1.10E-09	4.74E-21	4.24E-38	0.	0.	0.	0.	0.	0.	0.
SE	J.	6.57E-02	5.03E-08	5.03E-08	5.03E-08	5.03E-08	5.03E-08	5.03E-08	5.03E-08	5.03E-08	4.98E-08	4.52E-08
BR	J.	3.06E-01	1.93E-10	6.69E-23	1.37E-41	0.	0.	0.	0.	0.	0.	0.
KR	0.	5.59E-01	1.19E-03	1.18E-03	1.16E-03	1.12E-03	9.85E-04	8.66E-04	6.29E-04	1.95E-06	0.	0.
RB	J.	9.34E-01	1.64E-05	1.72E-06	5.83E-08	6.89E-11	2.00E-12	2.00E-12	2.00E-12	2.00E-12	2.00E-12	2.00E-12
SR	J.	9.78E-01	7.57E-02	3.80E-02	1.68E-02	8.54E-03	7.38E-03	7.03E-03	6.21E-03	6.74E-04	1.54E-13	0.
Y	0.	1.56E+00	1.06E-01	5.57E-02	2.41E-02	9.65E-03	7.38E-03	7.03E-03	6.21E-03	6.74E-04	1.54E-13	0.
ZR	J.	7.88E-01	1.77E-01	9.24E-02	3.49E-02	4.98E-03	2.29E-06	2.28E-07	2.27E-07	2.27E-07	2.27E-07	2.26E-07
NB	0.	1.97E+00	2.29E-01	1.55E-01	6.86E-02	1.07E-02	4.55E-06	6.46E-08	9.98E-08	2.26E-07	2.27E-07	2.26E-07
MO	J.	1.63E+00	1.56E-04	4.25E-11	6.04E-21	1.22E-40	0.	0.	0.	0.	0.	0.
TC	J.	1.82E+00	1.51E-04	2.08E-06	2.08E-06	2.08E-06	2.08E-06	2.08E-06	2.08E-06	2.08E-06	2.07E-06	2.01E-06
RU	J.	8.83E-01	2.82E-01	1.66E-01	1.06E-01	6.78E-02	1.69E-02	4.26E-03	1.35E-04	0.	0.	0.
RH	J.	1.36E+00	2.82E-01	1.66E-01	1.06E-01	6.78E-02	1.69E-02	4.26E-03	1.35E-04	0.	0.	0.
PD	J.	1.24E-01	2.73E-08	2.73E-08	2.73E-08	2.73E-08	2.73E-08	2.73E-08	2.73E-08	2.73E-08	2.73E-08	2.73E-08
AG	J.	1.49E-01	1.59E-03	8.38E-04	5.51E-04	3.95E-04	5.33E-05	7.21E-06	4.84E-08	0.	0.	0.
CD	J.	1.10E-02	8.14E-05	3.26E-05	9.19E-06	2.72E-06	2.14E-06	1.94E-06	1.51E-06	1.75E-08	7.77E-28	0.
IN	0.	2.08E-02	6.18E-07	1.99E-07	5.60E-08	4.46E-09	1.78E-13	7.11E-18	7.18E-29	0.	0.	0.
SN	J.	6.76E-01	2.05E-03	1.21E-03	7.28E-04	2.65E-04	4.88E-06	2.18E-07	1.16E-07	1.16E-07	1.15E-07	1.08E-07
SB	J.	1.18E+00	2.07E-03	1.89E-03	1.75E-03	1.53E-03	9.17E-04	5.49E-04	1.52E-04	2.30E-07	2.29E-07	2.15E-07
TE	J.	1.45E+00	1.78E-02	7.85E-03	3.43E-03	1.35E-03	3.87E-04	2.28E-04	6.30E-05	5.81E-15	0.	0.
I	J.	2.27E+00	1.37E-02	7.05E-05	3.34E-08	6.30E-09	6.30E-09	6.30E-09	6.30E-09	6.30E-09	6.30E-09	6.30E-09
XE	0.	1.57E+00	7.53E-03	2.20E-05	9.71E-08	2.14E-12	4.93E-31	1.14E-49	0.	0.	0.	0.
CS	J.	1.31E+00	4.92E-02	4.54E-02	4.28E-02	3.84E-02	2.64E-02	2.30E-02	1.37E-02	1.57E-03	4.13E-08	4.12E-08
BA	0.	1.24E+00	6.75E-02	1.67E-02	1.46E-02	1.45E-02	1.38E-02	1.32E-02	1.17E-02	1.47E-03	1.36E-12	0.
LA	0.	1.21E+00	6.07E-02	2.25E-03	1.60E-05	8.10E-10	5.33E-27	3.51E-44	0.	0.	0.	0.
CE	J.	1.12E+00	2.85E-01	1.68E-01	1.11E-01	6.76E-02	1.14E-02	1.91E-03	2.21E-05	0.	0.	0.
PR	J.	9.29E-01	2.06E-01	1.34E-01	1.05E-01	6.75E-02	1.14E-02	1.91E-03	2.21E-05	0.	0.	0.
ND	0.	2.16E-01	1.64E-02	3.66E-04	1.22E-06	1.36E-11	2.09E-31	3.22E-51	0.	0.	0.	0.
PM	J.	2.68E-01	2.25E-02	1.86E-02	1.63E-02	1.40E-02	8.22E-03	4.84E-03	1.29E-03	5.87E-14	0.	0.
SM	J.	1.09E-01	2.21E-04	2.19E-04	2.19E-04	2.18E-04	2.14E-04	2.11E-04	2.03E-04	9.89E-05	7.60E-08	0.
EU	J.	5.04E-02	1.26E-02	2.98E-03	2.26E-03	2.03E-03	1.45E-03	1.13E-03	7.81E-04	1.52E-05	1.77E-22	0.
GD	J.	1.28E-03	4.55E-06	3.82E-06	2.94E-06	1.74E-06	2.15E-07	2.65E-08	1.42E-10	0.	0.	0.
TB	0.	6.17E-04	2.47E-04	1.33E-04	5.53E-05	9.55E-06	8.51E-09	7.58E-12	1.79E-19	0.	0.	0.
OY	J.	1.13E-04	5.69E-09	2.28E-14	1.83E-22	1.18E-38	0.	0.	0.	0.	0.	0.
HQ	J.	3.15E-05	8.68E-09	1.78E-10	1.78E-10	1.78E-10	1.78E-10	1.78E-10	1.77E-10	1.68E-10	1.00E-10	5.53E-13
TOTALS	J.	2.68E+01	1.92E+00	1.38E+00	6.56E-01	3.78E-01	1.24E-01	6.76E-02	4.13E-02	4.51E-03	3.07E-06	2.90E-06

TABLE 5. Activation Products From 1 ppm Uranium Impurity in Zircaloy-2
Basis: Irradiation at 2.88×10^{13} n/cm²/sec avg. for 1100 days

ELEMENTAL ACTIVITY, CURIES												
BASIS = MT OF U CHARGED TO REACTOR												
	CHARGE	DISCHARGE	1.MO	3.MO	6.MO	12.MO	36.MO	60.MO	120.MO	1200.MO	12000.MO	120000.MO
TL	0.	3.56E-11	3.83E-11	4.63E-11	5.80E-11	8.38E-11	1.97E-10	2.97E-10	4.38E-10	2.26E-10	2.25E-11	3.26E-10
PB	0.	9.89E-11	1.06E-10	1.29E-10	1.61E-10	2.33E-10	5.46E-10	8.25E-10	1.22E-09	6.25E-10	4.84E-10	2.24E-08
BI	0.	9.89E-11	1.06E-10	1.29E-10	1.61E-10	2.33E-10	5.46E-10	8.25E-10	1.22E-09	6.25E-10	4.84E-10	2.24E-08
PO	0.	1.62E-10	1.74E-10	2.11E-10	2.64E-10	3.82E-10	8.96E-10	1.35E-09	2.00E-09	1.02E-09	7.05E-10	3.19E-08
AT	0.	1.23E-14	6.98E-15	2.43E-15	2.09E-15	2.23E-15	2.74E-15	3.29E-15	4.85E-15	8.99E-14	1.90E-11	3.06E-09
RN	0.	9.89E-11	1.06E-10	1.29E-10	1.61E-10	2.33E-10	5.46E-10	8.25E-10	1.22E-09	6.24E-10	2.44E-10	9.82E-09
FR	0.	1.30E-14	7.75E-15	3.30E-15	3.11E-15	3.52E-15	5.12E-15	6.74E-15	1.08E-14	1.27E-13	1.93E-11	3.06E-09
RA	0.	9.89E-11	1.06E-10	1.29E-10	1.61E-10	2.33E-10	5.46E-10	8.25E-10	1.22E-09	6.24E-10	2.63E-10	1.29E-08
AC	0.	6.45E-14	6.26E-14	6.47E-14	7.45E-14	9.46E-14	1.73E-13	2.50E-13	4.32E-13	2.77E-12	4.12E-11	3.32E-09
TH	0.	1.25E-07	8.28E-08	8.28E-08	8.28E-08	8.29E-08	8.32E-08	8.35E-08	8.39E-08	8.34E-08	8.40E-08	9.90E-08
PA	0.	1.27E-07	1.07E-07	1.08E-07	1.08E-07	1.08E-07	1.08E-07	1.08E-07	1.09E-07	1.31E-07	2.63E-07	3.03E-07
U	1.75E-07	4.93E+00	2.70E-03	6.02E-06	8.15E-07	7.99E-07	7.42E-07	6.90E-07	5.80E-07	2.05E-07	2.52E-07	2.79E-07
NP	0.	4.90E+00	6.23E-04	4.86E-06	4.86E-06	4.86E-06	4.86E-06	4.86E-06	4.86E-06	4.84E-06	4.60E-06	2.18E-06
PU	0.	1.24E-01	2.80E-02	2.78E-02	2.75E-02	2.68E-02	2.45E-02	2.23E-02	1.78E-02	6.06E-04	1.98E-04	1.11E-04
AM	0.	4.99E-02	3.60E-05	4.33E-05	5.42E-05	7.54E-05	1.56E-04	2.28E-04	3.82E-04	8.50E-04	2.06E-04	2.00E-06
CM	0.	9.46E-03	8.44E-03	6.66E-03	4.72E-03	2.51E-03	6.77E-04	5.53E-04	4.54E-04	1.59E-05	1.25E-07	4.53E-08
BK	0.	2.89E-09	9.77E-10	8.54E-10	6.98E-10	4.66E-10	9.30E-11	1.85E-11	3.29E-13	2.62E-20	2.53E-20	1.77E-20
CF	0.	2.71E-11	2.61E-11	2.53E-11	2.47E-11	2.34E-11	1.87E-11	1.52E-11	1.04E-11	2.50E-12	4.40E-13	3.62E-17
ES	0.	8.60E-13	7.17E-13	1.76E-13	1.22E-14	3.56E-17	1.01E-27	2.51E-38	0.	0.	0.	0.
TOTALS	1.75E-07	1.00E+01	3.98E-02	3.45E-02	3.22E-02	2.94E-02	2.53E-02	2.31E-02	1.86E-02	1.48E-03	4.10E-04	1.16E-04

TABLE 6. Activation Products to Be Expected From 1 ppm of Thorium Impurity in Zircaloy Fuel Cladding
Basis: Exposure at $2.88 \cdot 10^{13}$ n/cm²/sec avg. for 1100 days

ELEMENTAL ACTIVITY, CURIES												
BASIS = MT OF U CHARGED TO REACTOR												
	CHARGE	DISCHARGE	1.MO	3.MO	6.MO	12.MO	36.MO	60.MO	120.MO	1200.MO	12000.MO	120000.MO
TL	0.	1.15E-05	1.21E-05	1.40E-05	1.62E-05	2.02E-05	3.01E-05	3.45E-05	3.68E-05	1.64E-05	8.81E-07	1.86E-06
PB	0.	3.19E-05	3.37E-05	3.88E-05	4.51E-05	5.59E-05	8.34E-05	9.57E-05	1.02E-04	4.52E-05	9.62E-06	6.05E-05
BI	0.	3.19E-05	3.37E-05	3.88E-05	4.51E-05	5.59E-05	8.34E-05	9.57E-05	1.02E-04	4.52E-05	9.62E-06	6.05E-05
PO	0.	5.22E-05	5.52E-05	6.36E-05	7.38E-05	9.17E-05	1.37E-04	1.57E-04	1.67E-04	7.30E-05	9.47E-06	5.98E-05
AT	0.	1.90E-08	1.75E-08	1.74E-08	1.96E-08	2.52E-08	4.38E-08	6.24E-08	1.09E-07	9.42E-07	8.88E-06	5.87E-05
RN	0.	3.18E-05	3.37E-05	3.88E-05	4.50E-05	5.59E-05	8.33E-05	9.56E-05	1.02E-04	4.42E-05	7.25E-07	1.19E-06
FR	0.	1.95E-08	1.80E-08	1.80E-08	2.03E-08	2.60E-08	4.51E-08	6.43E-08	1.12E-07	9.52E-07	8.89E-06	5.87E-05
RA	0.	3.19E-05	3.37E-05	3.88E-05	4.51E-05	5.59E-05	8.34E-05	9.57E-05	1.02E-04	4.52E-05	9.63E-06	5.99E-05
AC	0.	6.15E-08	6.19E-08	6.56E-08	7.34E-08	9.01E-08	1.51E-07	2.09E-07	3.43E-07	1.63E-06	9.58E-06	5.93E-05
TH	2.77E-08	1.83E+01	5.65E-04	1.31E-04	5.16E-05	5.58E-05	8.32E-05	9.55E-05	1.02E-04	4.52E-05	9.69E-06	6.01E-05
PA	0.	1.83E+01	8.15E+00	1.75E+00	1.73E-01	1.71E-03	6.91E-07	6.91E-07	6.91E-07	6.89E-07	6.76E-07	5.59E-07
U	0.	6.18E-03	4.77E-04	2.18E-04	2.18E-04	2.18E-04	2.15E-04	2.13E-04	2.08E-04	1.50E-04	1.07E-04	1.03E-04
NP	0.	1.18E-03	5.21E-08	1.03E-09	1.03E-09	1.03E-09	1.03E-09	1.03E-09	1.03E-09	1.03E-09	1.03E-09	1.03E-09
PU	0.	4.19E-06	4.17E-06	4.16E-06	4.15E-06	4.14E-06	4.07E-06	4.00E-06	3.84E-06	1.88E-06	3.88E-09	1.43E-09
AM	0.	3.43E-08	4.42E-11	6.46E-11	9.48E-11	1.54E-10	3.78E-10	5.81E-10	1.01E-09	2.32E-09	5.57E-10	8.23E-13
CM	0.	5.42E-09	4.83E-09	3.76E-09	2.59E-09	1.26E-09	1.64E-10	1.08E-10	8.74E-11	3.86E-12	2.92E-14	5.03E-15
BK	0.	4.35E-17	1.47E-17	1.28E-17	1.05E-17	7.01E-18	1.43E-18	2.57E-19	2.65E-20	6.79E-32	6.79E-32	6.79E-32
CF	0.	2.00E-19	2.04E-19	2.08E-19	2.14E-19	2.19E-19	1.83E-19	1.57E-19	1.14E-19	3.65E-20	6.16E-21	3.75E-25
TOTALS	2.77E-08	3.66E+01	8.15E+00	1.75E+00	1.74E-01	2.32E-03	8.04E-04	8.88E-04	9.26E-04	4.70E-04	1.85E-04	5.84E-04

zirconium alloys would require careful control and dedicated facilities to avoid personnel contamination. If Zircaloy-4 comes into extensive use, the Ni-63 content would become very minor. However, the Zr-93-Nb-93 has a very long half-life and cannot be chemically separated. The presence of Zr-93-Nb-93 in any zirconium alloy will make it perpetually radioactive but reusable if desired.

The most active fission products in Table 4 and the transuranic elements in Tables 5 and 6 would be substantially reduced during decontamination of the melt because they are all very electro-positive elements. Presumably decontamination for storage and reuse would not have to be as complete as it would be storage as nontransuranic (<10 nCi/g).

The reuse of zirconium alloys requires a corrosion resistant material with the proper mechanical properties. To obtain these properties, chemical contamination of the alloys during cleaning and melting must be avoided. The oxide surface layers must be removed to avoid oxygen contamination and the melt must be made in a cold wall crucible excluding all reactive atmospheres and chemicals. It is possible that a calcium fluoride flux might be used during melting if the fluorine contamination can be held down. The reuse of the stainless steel and Inconel alloys will require longer cooling times because of the higher Co-60, Ni-63 and Ni-59 content of the irradiated alloys. The high energy gamma emissions of Co-60 will require shielding unless aged for 50 to 100 years.

An initial sample of highly irradiated Zircaloy fuel cladding was obtained as two 1-inch pieces of tubing from UO_2 - PuO_2 fuel rod. This tubing will be used to analytically verify the calculations of transuranic element concentrations in the clad metal and its distribution. The initial analytical methods will involve surface etching to various depths with the residue dissolved and analyzed by mass spectrometer and alpha energy analyses.

Plans For Next Period

The construction and testing of laboratory equipment to evaluate decontamination processes on a small scale will be completed and some process parameters investigated. The analyses of irradiated tubing samples will be initiated.

ZIRCALOY-STAINLESS STEEL ALLOY EVALUATION - R. G. Nelson

Consolidation of Zircaloy cladding hulls by melting with the other stainless steel hardware in the fuel bundle could result in the combination of two waste materials into a single storable ingot having a minimum volume. By combining stainless steel with Zircaloy, low melting eutectic mixtures are formed which can lead to simplified melting processes.

Initial laboratory studies of Zircaloy-2 type 316 stainless steel melts reported previously⁽²⁾ were extended to examine the melting and cooling curves of the 10, 15, and 20% stainless steel alloys in graphite crucibles heated by a resistance furnace. The objectives of these experiments were to determine what temperatures would be required to melt these compositions, to observe the extent of crucible reactions, and to observe the relative ingot quality. Table 7 is a summary of the results of these melting experiments.

It appears that compositions in the range of 15 to 20% stainless steel can be melted in graphite at a temperature of 1100°C or possibly slightly less. Greater crucible wear and carbon pickup will occur in the 20% stainless steel range. Temperatures for melting in the 10% stainless steel range will be slightly over 1100°C. The importance of oxygen contamination of the melt can be seen in the first two runs where incomplete melting occurred even at 1400°C. Carbon pickup in the ingots and their chemical properties have not been determined.

Plans For Next Period

Continued evaluation of low temperature melting alloys of stainless steel-Zircaloy-Inconel may be resumed in fiscal year 1975 depending on interest in potential reuse of the individual alloys.

TABLE 7. Summary of Zircaloy-2 - Stainless Steel Melting Experiments

Run Number	Composition wt% Stainless Steel	Charge	Temperature °C of Slope Change		Remarks
			Heating	Cooling	
1-7-74	20	Chopped stainless steel sheet and 1/4 in. diameter Zr-2 pellets; 150 grams total charge weight	Not Recorded	Not Recorded	56 grams of unmelted Zr-2; bridged ~1400°C maximum temperature; poor vacuum; Zr had black oxide; no cracks; considerable crucible reaction.
1-14-74	15	193 grams of pre-alloyed non-homogeneous arc buttons	Not Recorded	903 926	90 grams melted; balance was bridged; poor vacuum; Zr had black oxide; cracked, very little crucible reaction; maximum temperature 1125°C.
1-15-74	15	57 grams of pre-alloyed non-homogeneous arc buttons; 90 grams melted material from 1-14-74 run; 58 grams of bridged material from 1-14-74 run; 205.5 grams total.	906 924	903 926	Approximately 30 grams of unmelted material; improved vacuum; Zr was gold colored; cracked ingot; some crucible reaction near the solid-liquid interface; maximum temperature 1093°C.
1-16-74	15	114 grams non-homogeneous arc button	897 926	903 923 926	Added graphite lid to crucible; excellent melt 100% melted; little if any crucible reaction; non wetting convex meniscus. No discoloration; vacuum 1-2 microns; no cracks; maximum temperature 1104°C.
1-18-74	10	150 grams homogeneous arc button	926 965 980	902 926 952	With graphite lid; approximately 15 grams of unmelted material; no cracks; no discoloration; very little if any crucible reaction; Vacuum 0.35-0.40 microns, maximum temperature 1080°C.
1-21-74	20	150 grams homogeneous arc buttons	925 962 974	927 1062	

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WASTE TREATMENT AND HANDLING PROCESSES

V. L. Hammond

The principal objective of this program is to evaluate current practice and to develop alternative processes for safe and economical volume reduction of low-level contaminated solid wastes (hereafter called alpha wastes) from 10- to 50-fold as a precursor to long-term storage and/or disposal. This objective will be pursued via (1) a phased program of bench-scale research activities leading to and supporting experimental-facility development activities; and (2) by systematic and continuing analytical studies to assess developments in "current practice" for alternative, candidate techniques for processing alpha wastes prior to long-term storage and/or disposal.

The PNL experimental program will initially emphasize pyrolysis as a means for reducing the volume (and weight) of combustible solid wastes. It is also anticipated that there may be significant advantages obtainable by combining pyrolysis with incineration and/or molten salt treatment methods.

BENCH-SCALE RESEARCH AND DEVELOPMENT - L. K. Mudge, G. F. Schiefelbein, and G. L. Roberts

Fabrication of the equipment for the scoping studies was completed during the reporting period. The reactor components are pictured before assembly in Figure 1 and in Figure 2. Descriptions of the equipment were given in the previous quarterly report.

The reactor is heated by glass tape that has a maximum operating temperature of 1650°F. The receiver is heated by a resistance furnace that can be operated continuously at 1850°F. Gases can be added to the reactor to give downflow or upflow operation during gasification and can be preheated before addition to the reactor in a coil heat exchanger located in a tube resistance furnace.

Thermocouples are located on the inside wall of the alumina liner in the reactor at 1 in. from the bottom of the reaction zone and 1 in. from the top of the reaction zone, in the ash (or slag) receiver, and in the inlet

FIGURE 1. Reactor and Components

FIGURE 2. Pyrolysis Equipment Assembly

and outlet gas lines to the reactor. The total volume of gas generated in the reactor is determined by a wet test meter. Volumes are recorded at about 1 min intervals to determine the rate of gas evolution throughout the experiment. Condensibles in the gas stream are removed in a water cooled condenser; halides and sulfur-containing gases are removed for analysis in a caustic bubble scrubber. A glass beaker is inserted in the condensate receiver to allow easy removal of the condensate for weighing and analysis.

Conditions for the first five sets of experiments in the scoping studies are shown in Table 8. The experiments in set 1 are the same as originally planned with six constituents commonly found in alpha wastes used individually as feed. Set 2 experiments differ from the original plan in that only one constituent and two fluxing agents will be used rather than three of each. The main purpose of the flux is to remove halogens from the generated gas. This can be evaluated by use of PVC and the two fluxes shown which are active chloride getters. Two concentrations of each flux will be used corresponding to a 15% excess and a 100% excess of the amount required for stoichiometric chloride retention. Further experimentation with fluxes will probably be necessary in the set 6 experiments to evaluate the behavior of the flux during gasification and the form for flux addition, e.g., as large distinct pellets or as a powder on the surface of the waste material. In set 3 experiments, two feed sizes in addition to the size used in set 1 experiments will be evaluated, which is probably the limit of effectiveness for evaluation of the 1.78-inch I.D. reactor. Sets 4 and 5 experiments are essentially the same as outlined in the original plan. Set 6 experiments will probably include the fluxing experiments discussed above, experiments with steam addition, experiments for verification of results in sets 1 through 5, experiments with different feed mixtures, and experiments at different heating rates.

The simulated feed composition that will be used in the set 4 and 5 experiments is shown in Table 9. The simulated feed as defined for this study is a combination of the individual constituents evaluated in set 1

TABLE 8. Experimental Conditions for Scoping Studies - Pyrolysis Tests

Set No.	Exp. No.	Feed	Size	Temp., °C	Gas Addition	Flux
1	1 (a)	Rubber Tubing	-5/8"+1/4"	700	None	None
1	2 (a)	PVC				
1	3 (a)	Tygon				
1	4 (a)	Polyethylene				
1	5 (a)	Neoprene				
1	6 (a)	50 wt % paper- 50 wt % cloth				
2	7	PVC	-1/4"+14 mesh			1gNa ₂ CO ₃ /g PVC
2	8					1.7gNa ₂ CO ₃ /g PVC
2	9					0.75g NaOH/g PVC
2	10					1.3gNaOH/g PVC
3	11					None
3	12	Polyethylene				
3	13	Polyethylene	-1"+5/8"			
3	14	PVC				
4	15		To be determined	500		
4	16			900		
4	17	Polyethylene		500		
4	18	Polyethylene		900		
4	19	Neoprene		500		
4	20	Neoprene		900		
4	21	Simulated		500		
4	22			700		
4	23			900		
5	24			500 (b)	Air, 100 ml/min (c)	
5	25			700 (b)		
5	26			900 (b)		
5	27			Determined by air rate	Air, 400 ml/min (c)	
5	28				Air, 200 ml/min (c)	
5	29				Air, 100 ml/min (c)	
6	Conditions to be selected on basis of results in Sets 1 through 5.					

- (a) Experiments completed
 (b) Temperature for pyrolysis
 (c) Air started after pyrolysis is complete

experiments and is not a simulation of an actual alpha waste stream. Simulation of a waste stream is somewhat complicated because the day-to-day compositions are quite likely to vary considerably. The feed composition shown in Table 9 will be used in an attempt to define the interaction of waste constituents that were tested individually, and thus ascertain how variation of feed composition will affect the system.

TABLE 9. Simulated Feed Composition

<u>Constituent</u>	<u>% By Weight</u>
Rubber Tubing	15
Neoprene	15
PVC	20
Tygon	10
Polyethylene	10
Tissue Paper	15
Rags	15

The scoping studies experiments are designed to evaluate the effects of feed composition, feed size, fluxing agents, temperature, and air flow rate on processing rates and output compositions. Data that are being obtained for full evaluation of the system performance include:

- for the feed: calorific value, size distribution, bulk density, moisture content, composition, and ash content
- for the residue: calorific value, bulk density, ash content, and thermal stability as determined by differential thermal analysis
- for the gaseous effluent: volume per unit weight of feed, generation rate, composition (volume % H_2 , CH_4 , C_2H_4 , C_2H_6 , CO , CO_2 , N_2 , O_2 , and HCl), and particulates
- for the condensate: weight and calorific value of the oil fraction, and percent total organics for the aqueous fraction

In addition to the above information, the flow rate and temperatures in and out of the reactor will be obtained to determine overall mass and energy balances. With the above information, gas cleaning requirements can be developed and volume reduction and residue stability will be obtained. Corrosive attack on the reactor components by the gases generated will be noted.

Individual feed materials for the scoping studies were prepared and characterized. Results of the characterization of the feed materials are summarized in Table 10.

TABLE 10. Measured Characteristics of Feed Materials to Pyrolysis Unit

Feed Material	Wt% Moisture	Wt% Ash	Calorific Value		Bulk Density g/cm ³	Size in.
			kcal/g	Btu/lb		
50 wt% paper 50 wt% rags	3.4	0.77	3.98	7170	0.079	3/4
Latex Rubber Tubing	0.099	1.42	10.42	18,760	0.462	+1/5-5/8
Polyvinyl Chloride	0.043	1.83	4.98	8970	0.744	3/16 rod 5/8 long
Tygon	0.044	0	6.13	11,030	0.402	+1/4-5/8
Neoprene	0.16	28.5	4.83	8690	0.776	+1/4-5/8
Polyethylene	0	0.013	11.00	19,790	0.356	5/8 squares 1/8 to 1/4 thick

The moisture content in the feed materials was obtained by drying a weighed quantity of material in a drying oven for 2 hr at 100°C and determining the weight loss. The ash content in the feed materials was determined from the weight loss of feed samples upon heating to 950°C in air for 2 hr. An adiabatic calorimeter pressurized to 30 atm with oxygen was used to determine the heat of combustion (calorific value) of the different

materials. The bulk density of the material was determined by weighing the quantity of material required to fill a 500 ml graduated cylinder.

In each test run in set 1, the reactor was heated to 700°C prior to addition of the feed which was added in about 40 g batches. The gas evolved was collected for analysis in a sample bag after passing through a wet test meter to determine the volume of gas generated. Condensibles from each of the three batches of feed used in the run were removed and collected in a water-cooled condenser. A separate gas sample was collected for two of the batches in each of the runs. For each feed addition, pyrolysis was complete in about 5 minutes.

The calorific value of the residue and condensates and the chloride and sulfur analysis of the scrub solutions are not obtained yet. Partial results for the experiments already conducted are shown in Table 11.

TABLE 11. Results of Pyrolysis Experiments at 700°C

<u>Characteristics</u>	<u>Feed Material</u> <u>Paper Rags</u>	<u>Latex Rubber Tubing</u>	<u>PVC</u>	<u>Tygon</u>	<u>Neoprene</u>	<u>Polyethylene</u>
% wt reduction	79.9	98.5	86.9	92.7	56.4	99.7
% volume reduction (uncompacted)	62.6	98.21	9.1	79.1	(308)	99.0
% volume reduction (compacted)	80.4	99.05	59.3	93.2	(123)	99.0
Total wt of condensate, g	36.4	67.8	1	4	5	5
Condensate density	1.08	0.89	--	--	--	0.95
Total wt of residue, g	18.3	2.4	20.9	10.7	34.9	0.2
Total wt of feed reacted, g	90.9	160	160	146.9	80.0	76.2

() -- indicates a volume increase

The percent weight reduction in each experiment was determined from the weight of the feed added to the reactor and the weight of residue that remained in the reactor. The Neoprene that was used in the experiments had a high ash content, as noted in Table 10, which results in the low weight reduction figure.

The volume reduction was determined from the volume of residue obtained 1) as dumped from the reactor (uncompacted) and 2) after tamping of the residue in the graduate (compacted). Neoprene actually swells when subjected to heat, resulting in a volume increase when pyrolyzed at 700°C. A volume increase was also noted when neoprene was incinerated at 950°C to determine its ash content.

Two phases were present in the run with paper and rags as feed. The oil phase volume was less than 2 milliliters, and the aqueous phase appeared to contain considerable quantities of organics. The condensates obtained with the other feeds were not water soluble. A heavy, viscous tar was all that was collected with tygon, neoprene, and polyvinyl chloride (PVC) feeds. The condensate obtained with the other feed materials was similar to water in fluidity. The presence of the large fraction of condensate represents a troublesome and significant disposal problem for a process employing pyrolysis of this type of feed materials. Methods for handling the condensate and off-gas will be evaluated later in the program. Other processing conditions that can reduce the amount of condensate will be evaluated.

Differential thermal analysis (DTA) of the different feed materials and their carbonaceous residue (char) was utilized to evaluate reactivity. Results obtained in argon for tygon and for char obtained by pyrolysis of tygon at 700°C are shown in Figures 3 and 4. Results obtained by running the char in air are shown in Figure 5. Results for all feed materials showed that the degradation processes occurred at temperatures less than 400°C. Degradation of neoprene and latex were exothermic at about 360°C, while all other material tested degraded endothermically. The char from all feed materials was stable in argon but ignited in air at temperatures

FIGURE 3. DTA for Tygon in Argon

FIGURE 4. DTA in Argon for Char from Tygon

FIGURE 5. DTA in Air for Char from Tygon

above 400°C. Results shown in Figures 4 and 5 illustrate the reactivity of tygon char in argon and air and is typical for char residues from all the different feed materials.

The average gas compositions obtained on pyrolysis of the different feed materials are shown on a dry basis in Table 12. Chloride and sulfur compounds were removed from the gas stream prior to gas analysis. The amounts of chloride and sulfur are being determined by analysis of the scrub solution. Two separate 12 liter samples of the evolved gases were taken for each feed material pyrolyzed, and the concentrations in Table 12 are the average of the separate samples. Good agreement was obtained between samples for all feed except paper and rags which could not be averaged.

TABLE 12. Average Compositions of Pyrolysis Gases

Feed	Composition of Scrubbed Gas, Volume %							
	H ₂	CO ₂	C ₂ H ₄	C ₂ H ₆	CH ₄	CO	C ₃ H ₈	Unknowns
Latex	11.9	0.6	11.2	5.0	38.9	0.4	6.6	25.4
PVC	40.3	0.7	12.7	6.1	27.5	2.8	6.4	3.5
Neoprene	23.8	6.2	15.1	7.0	35.4	2.6	6.8	3.1
Tygon	24.0	3.9	17.6	4.9	30.4	10.9	5.4	2.9
Polyethylene	8.9	0.07	35.1	7.1	23.9	--	12.6	12.3
Paper-Rags (1)	20.9	10.2	5.5	1.3	9.4	52.0	0.7	--
(2)	23.7	28.9	2.3	1.3	7.7	35.4	0.7	--

Difficulty was encountered in pushing the paper and rags into the heated zone of the reactor during addition of the batches for the No. 2 sample. Some of the feed was found unreacted in the upper cool zone of the reactor which indicates that the refuse for the No. 2 sample was pyrolyzed at a temperature lower than 700°C. One would expect a greater yield of CO₂ and a lower yield of CO at the lower temperature than at 700°C. Results from the first sample are considered to be typical for pyrolysis of paper and cloth at 700°C.

The unknowns noted in Table 12 are primarily C_4 and C_5 hydrocarbons. The gas chromatograph is not calibrated to determine the concentration of these gases. The equipment and procedure used for gas analysis are described below.

A Hewlett-Packard model 7624A gas chromatograph was used for gas analysis with the off-gas samples being taken from the sample stream after passing through a flow meter to measure total volume through the sample system and a drierite column to remove moisture from the sample. Approximately 1.0 to 1.5 liters of sample volume was passed through the sample system before valving a gas sample into the columns. A heated gas sampling valve equipped with 0.5 cm^3 sample loops directed the sample into the columns.

The off-gas composition is such that obtaining complete resolution of all components on a single column in a reasonable length of time is difficult. Therefore, the dual-columns-in-series arrangement described below and shown schematically in Figure 6 was adopted.

The components of the product gas are completely separated in two columns in series, with a switching valve between the columns to allow the second column to be bypassed. The first column in the series is a 10 feet x 1/8 inch stainless steel Porapak Q^(a) (50-80 mesh), and the second column is a 10 feet x 1/8 inch Molecular Sieve 5A^(b) (60-80 mesh). The switching valve allows the CO_2 , ethane, ethylene, propane and higher boiling compounds to be eluted directly from the Porapak column to the detector, thus bypassing the molecular sieve column required for separation of oxygen and nitrogen.

The carrier gas used has a composition of 8.5% H_2 and 9.15% He. This mixture increased the sensitivity to H_2 by a factor of two over what could be obtained with pure He as the carrier gas.

-
- a. Trademark, Waters Corporation
 - b. Trademark, Linde Corporation

FIGURE 6. Schematic of Gas Chromatograph Column Switching Valve

Calibration curves were prepared using analytically certified gas mixtures prepared by the Matheson Company. All peak areas were integrated by a Hewlett-Packard Model 3370 electronic integrator. A sample obtained with the equipment is shown on Figure 7. About 10 minutes are required for each sample analysis.

EXPERIMENTAL FACILITY DESIGN - R. Walter, R. Sullivan

Conceptual design of the alpha waste pyrolysis experimental facility was begun in March. This facility will be used to demonstrate and evaluate the pyrolysis and/or pyrolysis-incineration process as a method of reducing the volume of low-level contaminated solid wastes prior to final storage or disposal. The processing equipment for use in the facility is being designed to enable the handling of both contaminated and non-contaminated waste. A decision will be made when the non-contaminated waste studies are complete to determine if the study should be continued, operation of the facility will be performed in glove using contaminated waste. The contaminated waste would consist of either alpha waste or waste with tracer levels of actinide elements or other suitable tracers.

Emphasis was placed on the development of design data for the facility during this time period. Preliminary data on the pyrolytic reactor and the auxiliary hardware are currently being generated.

FIGURE 7. Sample Chromatograph

TRITIUM SEPARATION AND FIXATION

L. L. Burger

The objectives of this program are to identify potential tritium separations methods and methods of fixing the tritium in forms suitable for storage.

The separations study aims to calculate separative work and costs in terms of separation parameters to identify overall separations needs. Solvent extraction, molecular excitation, and other techniques that appear attractive are to be evaluated in experimental tests.

The fixation study will define the concentrations and amounts of tritiated water wastes and examine methods of immobilizing HTO for permanent storage.

SOLVENT EXTRACTION - R. D. Scheele, L. L. Burger

The effect of hydrogen bonding on the physical properties of water and aqueous solutions is well known. Since hydrogen bonding is somewhat stronger when heavy isotopes of hydrogen are substituted for ^1H , it is reasonable to expect solubility changes where deuterated or tritiated water is substituted for normal water. This demonstrated by the solubility in water of both inorganic and organic compounds.

Likewise, H_2O is more soluble than D_2O in nearly all organic compounds that have been measured. Typical are data shown in the following table:

TABLE 13. Isotope Effect in the Solubility of Water in Organic Liquids

$t = 20^\circ\text{C} - 25^\circ\text{C}^{(1)}$

<u>Compound</u>	<u>Solubility, Mole Percent</u>		
	<u>H_2O</u>	<u>D_2O</u>	<u>$\text{H}_2\text{O}/\text{D}_2\text{O}$</u>
Furfural	21.4	17.9	1.19
EtOAc	13.3	11.7	1.13
sec-BuOH	70.5	67.6	1.04
Et_2O	(5.3)	(4.7)	1.16
Et_3N	--	--	1.43

Of the compounds reported, triethylamine shows the largest effect. No data exist for the strongly polar oxygen-containing compounds.

A second informative measurement is the shift of the infrared O-D or O-H stretching frequencies when water is dissolved in organic liquids. This shift (referred to the frequency in the unbonded molecule) is a direct measure of the H-bond strength. Thus, for the unsymmetric molecule DOH, the O-H stretching frequency varies from 3707 cm^{-1} in the vapor to 3374 in Et_3N ⁽²⁾ for the configuration D-O-H--X. Here X represents the hydrogen bonding polar group in the organic molecule. Table 14 lists some literature values of potential solvents.

TABLE 14. Fundamental O-H Stretching Bands for Bonded Unsymmetric Water Molecules⁽²⁾

Solvent	ν, cm^{-1}	Solvent	ν, cm^{-1}
Vapor	3707	Pyridine	3405
CCl_4	3662		
$\text{ClCH}_2\text{CH}_2\text{Cl}$	3635	Et_3N	3514
Tetrahydrofuran	3524	D_2O (ice)	3300
D_2O	3407		

These and other indicators of strong solvent-water interactions suggest that solvents may be found that produce a hydrogen isotope separation. If no bonding differences existed in the organic phase, then one should expect the separation characteristic of the relative vapor pressures. These are:⁽³⁾

$t^\circ\text{C}$	$P_{\text{H}_2\text{O}}/P_{\text{HDO}}$	$P_{\text{H}_2\text{O}}/P_{\text{HTO}}$
25°	1.075	1.095
50°	1.053	1.065
100°	1.026	1.030

However, the solubility will be expected to be low except where strong bonding is observed. Hence, the most promising solvent candidates would be expected to be those that form exceptionally strong bonds with water.

In our first experiments deuterium was used in place of tritium, and absorption spectroscopy employed to determine the isotope ratios. Since we are interested in compounds which form strong bonds with water, we chose initially two classes of compounds to study, those containing the phosphoryl group and the amines. For the phosphorus compounds, the alkyl phosphates, the alkyl phosphonates, the alkyl phosphine oxides, and the alkyl hydrogen phosphates were chosen because of their highly polar P=O group. The first three are in increasing order of their polarity, and the last exhibits a possibility of isotopic exchange and solvent extraction simultaneously.

Preliminary experiments indicated no separation of D₂O (or HDO) from H₂O using TBP (tributylphosphate), but a slight preferential extraction of D₂O using DBBP (dibutylbutylphosphonate). Subsequent tests did not confirm the latter result. Extractions with methyl isobutyl ketone, dimethoxy-pentane, and trioctylphosphine oxide (TOPO) also showed no separation. Triethylamine gave a small preferential extraction for H₂O.

It appears that opposing effects in solution tend to nearly balance the H-bond energy differences noted above. In an attempt to clarify the picture, extractions were carried out:

1. At low temperatures where water structure is a maximum (heavy isotopes produce greater structure).
2. At temperatures above about 60° where these highly polar solvents begin to assume "normal" temperature-solubility phenomena.
3. Extractions of strong acids and weak acids (heavy isotopes give lower ionization constants).

Tritiated water was substituted for H₂O-D₂O mixtures in an attempt to improve the analytical accuracy.

Difficulty in recovery of all the water from the organic phase has placed uncertainties on the analyses. Experiments involving extraction of nitrous acid by DBBP showed a preferential extraction of tritium with a separation factor of 1.3. This is the largest effect observed to date. Isotope separation in the extraction of water by DBBP remains questionable. A slight preferential extraction of tritium may exist.

MOLECULAR EXCITATION - J. R. Morrey, L. L. Burger

There are a number of ways in which optical excitation might be used to separate hydrogen isotopes. First, molecules containing tritium may be excited directly to unstable electronic states. Second, they may be excited to stable states whose potential energy surfaces intersect with unstable electronic states allowing for free dissociation processes. This process is called predissociation. Third, molecules containing tritium and in the ground electronic state may be vibrationally excited into sufficiently high levels to react readily and preferentially with other molecules to form products which are chemically separable from water. Fourth, a combination of vibrational excitation to affect selectivity and electronic excitation to affect dissociation might be used. All of these processes are under evaluation.

The molecules, H_2O , HDO , HTO , and D_2O are all nonlinear in the ground state. H_2O and D_2O exhibit C_{2v} symmetry; the others, C_s symmetry. H_2O has an 1A_1 ground state. The first electronic excited state results from the promotion of a $1b_1$ electron into a $4a_1$ orbital, generating a 1B_1 state. An optical transition between 1A and 1B , is represented by a continuous absorption band with a broad maximum at 1,655Å. There is photochemical evidence that this transition results in the dissociation of water into $\text{H} + \text{OH}$ free radicals, suggesting that the potential surface of the excited state is either entirely repulsive or has an unsymmetrical equilibrium position.

If ultraviolet lasers were available, water could be dissociated directly with no energy loss to surrounding molecules. It is possible that THO could be dissociated efficiently and selectively by this process under the proper experimental conditions. What these conditions might be will depend on the electronic spectrum of THO , which would require measurement. The basic problem, however, is that no suitable lasers are currently available to execute this scheme.

The second scheme, i.e., predissociation, would require higher frequency lasers than the first and would be more inefficient. On this basis, both have been tentatively ruled out.

It is conceivable that the combination of two lasers, one having the proper infrared frequency and the other the proper visible or near ultra-violet frequency, could be used to selectively excite and dissociate THO or that vibrational excitation in the ground state would lead to selective reactions. The conditions that must be met are stringent. They are listed below:

1. Photons from a tuned laser must have an energy resonant with the tritium containing molecules, and only those molecules.
2. Either the energy of the absorbed photons must be sufficient to overcome an activation energy of the tritium-containing molecules in reaction or there must be an efficient process by which multiple-photon excitation can occur to attain the requisite energy without exciting competing molecules.
3. The scheme must not allow significant exchange of energy from the excited tritium-containing molecules to hydrogen and deuterium containing molecules.
4. The reaction product must not exchange tritium atoms with deuterium and hydrogen at rates faster than the time required to physically separate the reaction product.

To satisfy conditions (1) and (2) we must carefully search the myriad of possible ground and hot-band vibration-rotation transitions of the tritium-containing molecules, and the competing isotopic molecules. To accomplish this requires a knowledge of spectroscopic constants for the molecules being investigated and computer programs to generate vibration-rotation spectra and to search among the lines for transitions meeting conditions (1) and (2).

A computer program was written to calculate spectroscopic constants for planar C_{2v} and C_s molecules H_2O , HDO , D_2O , THO , TDO , H_2S , HDS , D_2S , T_2S , THS , given molecular configuration and masses.

Another computer program has been written to calculate the vibration-rotation spectra of C_{2v} or C_s planar molecules, taking into account the selection rules and state weighting factors.

Our search for multiple excitation schemes has uncovered several possibilities for H_2O . Using discretely chosen single laser frequencies, it is possible to excite molecules into vibrational modes up to 80 kilocalories above ground state. Some of these schemes are expected to be exceedingly efficient processes because theoretically the molecules can attain their highest energies before collision with other molecules is likely to occur, thus making reaction probable upon first collision.

There is reason to believe the comparable schemes will emerge for other H, D, T containing molecules. The scheme finally settled on for the first experiment will use a laser frequency of 8735.00 wave numbers to excite the water molecule from the (000) $\rightarrow$ (210) transition and subsequently the (210) $\rightarrow$ (042) transition. In this notation the integers are the quantum numbers for the ν_1 , ν_2 , and ν_3 frequencies. The rotational lines to be excited are $(2_0) \rightarrow (2_{-2})$ and the $(2_0) \rightarrow (2_{-2})$ levels. Here the integer is the rotational quantum level, and the subscript is the τ value for asymmetric rotators.

Data from Bluhm, Yardley, Young, and Harris has been reported to us which describes a method whereby a tunable infrared laser can be used in a resonant two-photon pumping scheme to generate tunable infrared radiation in the vacuum ultraviolet, reportedly with extremely good photon efficiency. This method may be an appropriate means of electronically exciting tritiated molecules, thus allowing for predissociation or reaction.

TRITIUM FIXATION - J. L. Ryan, E. C. Martin, L. L. Burger

A report "The Technology of Fixation of Tritium" BNWL-1807 has been written. This report defines the sources of tritium in the fuel cycle and considers the present approach to the handling of tritium wastes then assesses means of controlling, fixing, and storing tritiated wastes.

Under present practices, large volumes of low concentration tritium wastes are produced at both the reactor and reprocessing site. Most of the fission produced tritium will appear at the reprocessing plant where modified flowsheets could produce smaller volumes of higher concentration wastes.

Known methods of complexing water and hydrogen are reviewed, and the merits of each considered. Some other disposal methods, e.g., deep well, are briefly considered for reference. Clays, molecular sieves, Portland cement, organic compounds, and hydrides all appear to have merit depending on the nature (principally the degree of dilution) of the tritium waste. Recommendations for further studies were made.

PARAMETRIC STUDIES OF TRITIUM SEPARATION CASCADES - D. H. Lester

The basic goals of the first parametric studies were (a) to examine the relative effects of decontamination factor, enrichment factor and concentration levels on a tritium separation cascade; (b) produce generalized curves so that the basic parametric effects could be readily examined; and (c) examine the importance of simultaneous deuterium separation. An interim report, BNWL-B-326 has been written which is summarized here.

Computer programs are now in place which can be used to calculate parametric effects in ideal cascade separations of tritium-hydrogen. Calculations of total flow and separative duty have been made as a function of decontamination factors and enrichment factors for a range of separation constants (α 's). Plant shapes (head-to-product flow ratios versus stage number) have also been calculated.

In addition, an initial investigation of the simultaneous separation of naturally occurring deuterium has been carried out. The deuterium separation was calculated in the same fashion as suggested by Ribnikov and Pupezin.⁽⁴⁾ The deuterium calculations are done assuming that an ideal cascade for tritium is also ideal for deuterium at the same time (i.e., constant heads separation factor, equal concentrations for both streams fed to a stage).

The results show that the total flow (which is indicative of cascade power consumption) for tritium-hydrogen changes very rapidly with separation constant, from greater than 3000 to 200 moles/mole feed for a separation constant less than 2 for decontamination factors (DF) between 10^4 and 10^8 . The change in total flow with separation factor is relatively small for separation factors above 2. Total flow at a given enrichment factor is relatively insensitive to feed concentration from 4×10^{-11} to 1×10^{-8} . A dramatic difference is observed between the effect of DF and the effect of enrichment factor or plant shape: A change in DF at a given enrichment factor results in a reduction of the number of stripping stages with no change in the recycle ratios of the plant. A change in enrichment factor for a given DF has an overall, large impact on plant shape and size. Increasing enrichment factors results in large increase of pumping and capital costs, while DF mainly affects capital costs. A check on the assumptions used for deuterium predictions indicates that the assumption is not very accurate. More sophisticated handling of H, D, T is indicated.

- The interim conclusions are:

Unless there is great advantage to producing tritiated D_2O , high enrichment factors are not recommended because of high capital and operating expense. A maximum enrichment factor of 10^2 is indicated.

- Large DF's do not instill large penalties in plant design (10^6 or 10^8 may not be unreasonable).
- Incremental increases of separation constants above 5 result in rapidly diminishing incremental decreases of the number of stages (capital cost) and in the total flow (operation cost).
- A more sophisticated treatment of the deuterium-tritium interaction is needed. The results of a more detailed analysis may alter some of the above conclusions.

PLANS FOR NEXT PERIOD

Solvent extraction results that appear interesting will be rechecked using HTO tracer. A test of the molecular excitation method is planned

using the water-methane reaction. In spite of excitation of the OH bond, the reaction may require a catalyst. In this event, another organic molecule may be chosen.

It is planned to test the acetaldehyde synthesis using D_2O . When the yields and product of step number two are improved, then a tritiated sample will be prepared for observation.

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