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**Technical Progress Report
Private Sector Initiatives Between
the United States and Japan**

January 1990 - December 1990

DOE Research and Development Report

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Rockwell International

Rocketdyne Division

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Rockwell International

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ACRONYMS

ALARA	as low as reasonably achievable
CRIEPI	Central Research Institute for the Electric Power Industry
DOE	Department of Energy
EMF	electromagnetic force
HEPA	high-efficiency particulate air
ICP	inductive coupled plasma
JAPC	Japan Atomic Power Company
KHI	Kawasaki Heavy Industries
LMR	liquid metal reactor
MURR	Missouri University Research Reactor
SC	supercooled
SSFL	Santa Susana Field Laboratory
TRU	transuranic

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INTRODUCTION

The Private Sector Initiatives Program was started in mid-1987 under the cognizance of the Office of the Deputy Assistant Secretary for Reactor Systems, Development Technology (NE-50). The purpose of this program is to utilize industrial ties between U.S. firms and their Japanese counterparts, to enhance U.S. access to advanced Japanese reactor programs, and ultimately, to provide a basis for U.S. vendor participation in advanced Japanese liquid metal reactor (LMR) designs. The program includes participation from the three U.S. vendors: General Electric Company, Rockwell International, and Westinghouse.

Rockwell activities on the Private Sector Initiatives Program were implemented through an existing relationship with Kawasaki Heavy Industries (KHI). Rockwell and KHI reviewed their respective LMR research needs to identify and screen suitable areas for collaborative efforts. These areas were determined to be of mutual benefit and were approved by the Department of Energy (DOE) and Japan Atomic Power Company (JAPC). Rockwell and KHI performed their respective portions of tasks within these selected areas and exchanged results. Agreed-upon exchange areas for the Rockwell/KHI private sector initiatives collaborative efforts are

- Plant-type evaluations
- Shrouded inducer pump
- Inherent safe core
- Passive shutdown heat removal system
- Booster tube steam generator
- Advanced piping
- Transuranic (TRU) partitioning
- Dry sodium removal.

SUMMARY

This annual report for calendar year 1990 describes the efforts performed under the Private Sector Initiatives contract. The report also describes those efforts that have continued with private funding after being initiated under this contract.

Two privately funded, continuing activities are described in Tasks 1 and 2. The first task is the development of the shrouded inducer pump, which is funded by the Japan Atomic Power Company (JAPC), Kawasaki Heavy Industries (KHI), and Rockwell International. The second task is the development of a pyrochemical process, called TRUMP-S, for partitioning actinides from PUREX waste. This effort is funded by the Central Research Institute for the Electric Power Industry (CRIEPI), KHI, and Rockwell International.

TASK 1. SHROUDED INDUCER PUMP

1.1 OBJECTIVE

The objective of this task is to develop a shrouded inducer pump for the Demonstration Fast Breeder Reactor. The expectations are that the pump can be adapted to the requirements of other advanced liquid metal reactor (LMR) plants with little or no design modification. Successful completion of this scale-model pump development phase is expected to lead to a full-scale pump prototype design, fabrication, and test program.

1.2 ACCOMPLISHMENTS

Results of prior year activities are reported in the 1987, 1988, and 1989 Private Sector Initiatives annual reports.^{1,2,3} In 1990 the modified 1/5-scale pump hardware was performance tested at the Kawasaki Heavy Industries (KHI) pump test facility in Akashi, Japan. The original design configuration initially tested had required modification to overcome two performance problems: low-developed head caused by separation in the diffuser and dye removal on the inducer, indicating the potential for cavitation damage. The design modifications selected included a reduction in the tip speed of the inducer, addition of a second blade row (kicker) on the rotor, and an improved diffuser design to diffuse the flow axially before turning it.

Two pump configurations (Figure 1) were developed and tested in the 1/5-scale pump. One configuration, the cylindrical kicker, maintained the same blade tip diameter in the kicker as in the inducer. The second configuration, the bell kicker, used a variable blade tip diameter in the kicker section. The two pump configurations were tested with different diffuser designs. The diffuser system in each pump configuration used a stator downstream of the rotor to remove the swirl flow. In the cylindrical kicker configuration, a bladed annular diffuser was used to slow the axial flow, and then a bladed turn was used to turn the flow from an upward direction to downward. The bell kicker pump configuration used an annular unbladed diffuser section and an unbladed, contoured turn. The bladed diffuser system concept provides for a shorter rotor overhang (the distance between the lower shaft bearing and the center of the rotor), whereas the unbladed diffuser system concept is a simpler mechanical configuration.

Test results for the two configurations are shown in Figures 2 and 3. Pump head data as a function of pump discharge flow are shown for three rotor speeds. In Figures 2 and 3, the normal operating range is shaded. The basic requirements for the pump are a discharge flow of 9.2 m³/min (2,430 gpm) with a pump head of 80 m (262 ft) maximum and 53.3 m (175 ft) minimum. The data in Figure 2 show that the cylindrical kicker design exhibits a stable performance in the required region with margin on both the high and low flow sides.

The bell kicker design also exhibits stable performance in the required region, but shows no margin at the 100% flow, minimum head condition ($N = 86\%$, $Q = 9.2 \text{ m}^3/\text{min}$ on Figure 3). The head falls off rapidly beyond this point due to cavitation. The bell kicker was also found to have a lower efficiency (82.6%) than the cylindrical kicker, which has a 97% efficiency. This results in an

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Figure 1. Cylindrical Kicker and Bell Kicker Pump Configurations

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Figure 2. Head-Flow Performance Curves for Inducer Pump with Cylindrical Kicker

Figure 3. Head-Flow Performance Curves for Inducer Pump with Bell Kicker

overall pump efficiency for the bell kicker configuration of 70.9% compared to 74.4% for the cylindrical kicker configuration.

Contrary to expectations, the bladed diffuser configuration tested in the cylindrical kicker pump was found to have approximately 40% larger head loss than the unbladed diffuser configuration tested in the bell kicker pump. Using the unbladed diffuser configuration with a cylindrical kicker could further improve the efficiency of the cylindrical kicker pump.

1.3 PROGRAM SUMMARY

The shrouded inducer pump is a small, compact pump, which can make a major contribution toward achieving a compact reactor assembly for an economic LMR design. Tests performed in 1990 have demonstrated a design configuration that meets the requirements set forth for the scale-model pump development phase. The test results also point to some design configuration improvements, which could further improve the pump efficiency. It is proposed that the design be further modified to institute these improvements into the selected pump configuration.

TASK 2. TRANSURANIC PARTITIONING

2.1 OBJECTIVE

The overall objective of the Rockwell International/Kawasaki Heavy Industries/Central Research Institute for the Electric Power Industry (Rockwell/KHI/CRIEPI) program is to develop a process to recover 99% of the remaining transuranic (TRU) materials (Np, Pu, Am, Cm) from PUREX waste to simplify nuclear waste management. The purpose of the program is to separate the high-level nuclear waste into a TRU-rich stream and a TRU-depleted stream to reduce waste management costs. The actinide product can then be transmuted into fission products or stored. This can markedly reduce the amount of actinides in the waste requiring long-term isolation or repository emplacement and can supply significant quantities of fissile and fertile materials for power production during transmutation.

2.2 ACCOMPLISHMENTS

2.2.1 Alpha Laboratory and Actinide Data

An Alpha Laboratory had been constructed in 1989 at Rockwell's Santa Susana Field Laboratory (SSFL) for the purpose of conducting the actinide tests on the TRUMP-S program (as reported in Ref. 3). However, due to public pressure to terminate activities at the laboratory with radioactive materials, Rockwell decided to complete contractual commitments, including Stage 1 of TRUMP-S, and then terminate work with actinide materials at Santa Susana. Since it appeared likely that the TRUMP-S project would continue into Stage 2, it was decided to seek an alternate site at which both Stage 1 and Stage 2 TRUMP-S test activities could be conducted, so equipment would not need to be relocated between the two stages of the project.

Bids from qualified organizations were received in late 1989 and early 1990, resulting in the January 1990 selection of the Columbia campus of the University of Missouri as the new site for conducting the TRUMP-S actinide tests. The University of Missouri at Columbia was selected since the campus includes:

1. Large operating research reactor [Missouri University Research Reactor (MURR)] with the associated facilities and personnel required to perform the required development
2. Required licenses in place (license amendments were required)
3. Strong nuclear engineering program
4. Required space and commitment at MURR to install the Alpha Laboratory with minimal downtime.

The TRUMP-S Alpha Laboratory was constructed in the basement of MURR so that all actinides used in the program have multiple containment (i.e., gloveboxes, Alpha Laboratory, MURR basement, and MURR facility). This approximately 6-m by 8-m alpha facility has been designed

and operated to meet all requirements for plutonium containment. A view of the Alpha Laboratory from the basement is shown in Figure 4 with the emergency exit at the left and the airlock entrance on the right. The TRUMP-S Alpha Laboratory dedication plaque (April 24, 1990) is on the emergency exit door

All personnel that work at the MURR facility must wear radiation dosimeter badges to monitor total whole body radiation exposure. Personnel working in the Alpha Laboratory must also wear dosimeter rings to monitor the radiation exposure received by the hands while working in the glove-box. In addition, when americium is in the gloveboxes, personnel must also wear dosimeters that monitor radiation dosages received. MURR's ALARA (as low as reasonably achievable) policy is to assure that on-the-job exposures are only a small fraction of allowable exposures. The Alpha Laboratory is operated according to strict written procedures, which have been prepared by the various operating departments but approved by facility Health Physics and program management. The Alpha Laboratory became operational in July 1990 and actinide data have been reported.⁴⁻⁶ A description of the laboratory and a summary of the actinide data are given in Sections 2.2.1.1 through 2.2.1.4.

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Figure 4. TRUMP-S Alpha Laboratory at the Missouri University Research Reactor Facility

2.2.1.1 Gloveboxes

The TRUMP-S test program uses three gloveboxes located in the Alpha Laboratory: the argon box, the air box, and the inductive coupled plasma (ICP) spectrometer box.

The three gloveboxes are located in an "L" shape in the center of the laboratory (Figures 5 and 6). The support equipment is located around the edge of the room.

The argon box is used to conduct the electrochemical experiments that are the objective of the program. The air box is used to prepare aqueous samples for analysis in the ICP box and to clean and dry equipment for use in the argon glovebox. The ICP box is used to analyze the chemical composition of various aqueous samples.

All TRUMP-S gloveboxes are maintained at negative pressure (-0.5 to -1.5 in. of water) to the Alpha Laboratory pressure. Pressure in the gloveboxes can be monitored both within and outside

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Figure 5. Glovebox Arrangement Viewed from DriTrain/NiTrain

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Figure 6. Alpha Laboratory Layout

the laboratory; the MURR Facility Control Room, which is manned around the clock 7 days a week, is automatically notified whenever glovebox pressure is not maintained.

The argon in the argon glovebox is continuously purified by circulating the atmosphere through a vacuum atmosphere DriTrain and NiTrain, which removes traces of water, oxygen, nitrogen, and hydrogen that might diffuse into the glovebox or be formed by the removal equipment. High-efficiency particulate air (HEPA) filters on the argon circulation lines to and from the argon glovebox assure actinide particulates removal before the argon is purified. The atmosphere is maintained routinely at about 0.01-ppm oxygen, <1-ppm H₂O, and <10-ppm N₂ and H₂.

2.2.1.2 Analytical

Samples can be analyzed by ICP with a sensitivity of 0.1 to 100 ppm depending on the actinide being analyzed. Americium sensitivity (<0.05 ppm) is much greater than the other actinides. The reproducibility of ICP analysis is about $\pm 1\%$ when fully utilized.

Frequently the electrochemical end points can be used to determine concentration. If competing reactions are negligible, the concentration can be determined to <0.1 mg coulometrically. For small weights (mg range), this is generally a more accurate method for determining concentration than by weighing the amounts.

2.2.1.3 Experimental

The electrochemical cells used correspond to the following:

The reference electrode consisted of a Ag wire in contact with AgCl dissolved in molten LiCl-KCl eutectic. A lithium aluminum electrode was used as a secondary reference electrode. The reference electrode, active metal electrode, and Li/Al electrode were contained within a 2-in.-diameter alumina crucible partially filled with LiCl-KCl eutectic.

NpCl₃, PuCl₃, and UCl₃ were prepared in situ by reaction of CdCl₂ in molten LiCl-KCl with the appropriate actinide metal dissolved in molten Cd at 550°C to produce an approximate 1 wt. % solution of actinide trichloride in the molten salt. For some uranium electromotive force (EMF) measurements, preparation of UCl₃ was carried out by electroreduction of UCl₄ within the cell itself. The LiCl-KCl eutectic was purchased in a highly purified form from Anderson Physics Laboratory. The measured oxygen content of the LiCl-KCl eutectic was 15 ± 12 ppm. AgCl was also purchased from Anderson Physics laboratory and had emission spectrographic impurities of approximately 3 ppm. The actinides used were supplied as pure metals under contract from the Department of Energy (DOE).

2.2.1.4 Actinide Results and Discussion

At equilibrium, the experimental cell potential (E) is related to the mole fraction of the actinide trichloride (X_{MCl_3}) by the following form of the Nernst equation.

$$E = E_{\text{MCl}_3}^{\circ} - \frac{RT}{nF} \ln X_{\text{MCl}_3} - \frac{RT}{nF} \ln \gamma_{\text{MCl}_3} - E_{\text{AgCl}}^{\circ} + \frac{RT}{F} \ln X_{\text{AgCl}} + \frac{RT}{F} \ln \gamma_{\text{AgCl}} \quad (1)$$

In Eq. 1, unit activity coefficients have been assumed for AgCl⁶ and unit activities for the pure metals. $E_{\text{MCl}_3}^{\circ}$ refers to the standard potential in a hypothetical supercooled (SC) liquid reference state where $X_{\text{MCl}_3} = 1$ and $\gamma_{\text{MCl}_3} = 1$. Other symbols in Eq. 1 have their usual meaning.

At low concentrations of MCl_3 and at constant temperature, the activity coefficient (γ_{MCl_3}) and hence the last four terms in Eq. 1 will be nearly constant with respect to X_{MCl_3} . Extrapolation of measured values of E to $X_{\text{MCl}_3} = 1$ then permits the calculation of $E_{\text{MCl}_3}^\circ$. Values of E versus $\log X_{\text{MCl}_3}$ for various temperatures have been plotted from the data obtained and the resultant graphs are shown in Figure 7 for U, Np, and Pu. Values of E versus temperature (T) are given in Figure 8. Least square lines were calculated relating the cell voltage to X_{MCl_3} and T . These are shown in Figures 7 and 8. From the least square lines in Figure 7, values of the cell voltage E were extrapolated to $X_{\text{MCl}_3} = 1$ to yield standard potentials, $E_{\text{MCl}_3}^\circ$. The slope of the line gives n , the theoretical number of electrons that must be added to the actinide ion to obtain the metal. Correlation of the observed value of n and the theoretical ($n = 3$) is a measure of the quality of the data. These potentials are listed in Table 1 along with values for the free energy of formation ($\Delta G_f^\circ = -nFE^\circ$). From Table 1 note that the standard potentials are ordered as follows:

$$E_{\text{Pu(III)/Pu(O)}}^\circ > E_{\text{Np(III)/Np(O)}}^\circ > E_{\text{U(III)/U(O)}}^\circ$$

This ordering of standard potentials is opposite to that measured by Martinot et al.^{7,8,9} in the LiCl-KCl eutectic.

A comparison of data obtained here and in the literature in the LiCl-KCl eutectic at about 450°C is shown in Figure 9. For uranium, agreement between data reported here and in the literature is quite good (within 30 mV). For plutonium, data from this laboratory agrees well with that of

Table 1. Values of E° , ΔG_f° , and γ for Actinide Trichlorides

Actinide	Temperature (°C)	n^a	E° (V)	ΔG_f° (exp) k joules	ΔG_f° (thermal) ^b k joules	γ
UCl_3	400	3.03	2.527	-731.4	-695.6	1.7×10^{-3}
	425	3.01	2.513	-727.5	-691.4	2.0×10^{-3}
	450	2.98	2.499	-723.5	-687.1	2.3×10^{-3}
	500	-	2.472	-715.5	-678.9	3.4×10^{-3}
NpCl_3	400	2.95	2.724	-788.5	-730.0	$(10^{-5})^c$
	425	2.97	2.708	-783.9	-726.0	$(10^{-4})^c$
	450	2.99	2.692	-779.2	-721.9	$(10^{-4})^c$
	500	2.98	2.660	-769.8	-713.6	$(10^{-4})^c$
PuCl_3	400	2.89	2.834	-820.4	-783.9	1.5×10^{-3}
	425	2.93	2.817	-815.5	-779.6	2.1×10^{-3}
	450	2.95	2.800	-810.5	-775.5	3.0×10^{-3}
	500	-	2.766	-800.7	-767.0	5.3×10^{-3}

^aFrom slope of extrapolations indicating the number of electrons required to convert the halide to metal.

^bHypothetical supercooled (SC) liquid standard state.

^cSome neptunium thermal data used to derive ΔG_f° (thermal) were estimated; it is believed that these estimated data are in error sufficiently to give erroneous γ s for NpCl_3 and γ_{NpCl_3} should be 10^{-3} as found for U and Pu.

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Figure 7. Dependence of Actinide Cell Voltage on Concentration

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Figure 8. Dependency of Cell Voltage on Temperature

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Figure 9. Comparison of Actual Data With the Literature

Campbell and Leary.¹⁰ The potentials measured by Martinot and Duyckaerts for Np⁸ and Pu⁹ are much lower than those obtained in this work or by Campbell and Leary. One possible reason for the discrepancy between our work and that of Martinot and Duyckaerts may be the quality of the atmosphere above the molten salt bath that contained 10-ppm O₂ for Martinot and Duyckaerts' work with Pu, but only 0.01-ppm O₂ for the work reported here.

If this trend of increasing standard potential with increasing atomic number continues, the potential of americium could approach that of the lanthanides. This would result in americium being the most difficult actinide of the dominant actinides present in nuclear waste or spent nuclear fuel to separate in pyrochemical processing. Measurements of thermodynamic properties of americium are planned.

An examination of the temperature dependence of the free energy of formation can yield enthalpies and entropies of formation for the trivalent actinides. The data clearly show a linear dependence of ΔG_f° on temperature. A least squares fit of the free energy versus absolute temperature (K) is expressed by the following equations:

$$\Delta G_f^\circ = \Delta H_f^\circ - \Delta S_f^\circ T$$

$$\text{UCl}_3, \Delta G_f^\circ = -838.4 + 0.158 T \text{ (kJ/mol)}$$

$$\text{NpCl}_3, \Delta G_f^\circ = -914.1 + 0.187 T \text{ (kJ/mol)}$$

$$\text{PuCl}_3, \Delta G_f^\circ = -953.0 + 0.197 T \text{ (kJ/mol)}$$

Activity coefficients, γ_{MCl_3} , were calculated by comparison of the standard free energy as determined by electrochemical methods to a reference free energy derived from thermal measurements. The reference state chosen was the SC liquid actinide trichloride. Activity coefficients were calculated from the following equation:

$$RT \ln \gamma_{\text{MCl}_3} = \Delta G_{f(\text{electrochem})}^\circ - \Delta G_{f(\text{SCliq})}^\circ \quad (2)$$

The standard free energy of formation of the SC liquid was determined from published values of $\Delta G_{f(\text{crystal})}^\circ$ ¹¹ and calculated values of the free energy of fusion, $\Delta G_{\text{fusion}}^\circ$. For uranium and plutonium, $\Delta G_{\text{fusion}}^\circ$ was calculated using published values of thermal data. Reliable thermodynamic values for the calculation of $\Delta G_{\text{fusion}}^\circ$ of NpCl₃ were not found in the literature and were estimated.

Value of free energies of formation from thermal data and activity coefficients for UCl₃ and PuCl₃ are listed in Table 1. The activity coefficients, which are typically 10⁻³, indicate very non-ideal solution behavior, which is consistent with complexation of UCl₃ and PuCl₃ in the molten salt electrolyte observed by others. These large negative deviations from ideality have also been calculated from electrochemical measurements by Martinot.¹²

2.2.2 Preliminary Actinide/Rare Earth Separation Estimates

Preliminary estimates of actinide/rare earth separation efficiency and product purity have been made using the measured data for the rare earths and actinides other than americium, for which the data were estimated.

An initial indication of actinide/rare earth separation efficiency can be obtained from the potential diagram given in Figure 10, which shows the equilibrium potential/concentration relationship around the inert cathode. In electrode deposition from a salt solvent, the element with the least negative deposition potential for its chloride compound will deposit before an element with a more negative deposition potential. This elemental deposition will continue until the concentration decreases sufficiently so that comparable deposition potentials are observed for the two elements. Thereafter the elements will be codeposited.

In this preliminary estimate, all elements will be assumed to behave as an $n = 3$ electron electrochemical reaction. In Figure 10, the rare earth element with the closest potential to americium (the most negative potential actinide) is gadolinium. Based on potential alone, this would make gadolinium the most difficult rare earth to separate from the actinides. When concentrations in the PUREX waste starting material are taken into consideration, neodymium is the most abundant rare earth present with a concentration approximately 20 times higher than gadolinium. This will place

Figure 10. MCl_3 Potentials Versus Concentration

neodymium's potential more positive than gadolinium and make neodymium the most abundant rare earth in the cathode deposit product.

Calculations were made for the electrochemical separation of the elements using an inert anode. The results are presented in Figure 11. Three nines (99.9%) removal of the actinides studied can be accomplished on a single pass with approximately 8% rare earths in the deposit.

2.3 PROGRAM SUMMARY

The TRUMP-S process is feasible and cost effective, but electrochemical property data for americium is still required to determine actinide separation efficiency.

Electrochemical data for U, Np, and Pu has been obtained and is summarized in this report. The experimental chloride-free energies of formation were measured along with activity coefficients of the chloride in LiCl-KCl electrolyte. Activity coefficients will be very important in obtaining good separation of actinides from rare earths.

Preliminary actinide/rare earth separation estimates indicate that a greater than 99% removal of the actinides can be accomplished on a single pass with only 8% of rare earths in the actinide stream.

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Figure 11. Codeposition of Rare Earths with Actinides in an Electrodeposition Using an Inert Anode

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