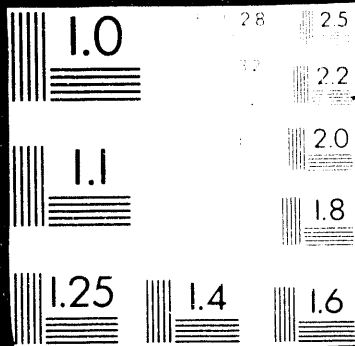


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TESTS OF PROTOTYPE SALT STRIPPER SYSTEM FOR IFR FUEL CYCLE\*

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## TESTS OF PROTOTYPE SALT STRIPPER SYSTEM FOR IFR FUEL CYCLE

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### ABSTRACT

One of the waste treatment steps for the on-site reprocessing of spent fuel from the Integral Fast Reactor fuel cycles is stripping of the electrolyte salt used in the electrorefining process. This involves the chemical reduction of the actinides and rare earth chlorides forming metals which then dissolve in a cadmium pool. To develop the equipment for this step, a prototype salt stripper system has been installed in an engineering scale argon-filled glovebox. Pumping trials were successful in transferring 90 kg of LiCl-KCl salt containing uranium and rare earth metal chlorides at 500°C from an electrorefiner to the stripper vessel at a pumping rate of about 5 L/min. The freeze seal solder connectors which were used to join sections of the pump and transfer line performed well. Stripping tests have commenced employing an inverted cup charging device to introduce a Cd-15 wt % Li alloy reductant to the stripper vessel.

### I. INTRODUCTION

The Integral Fast Reactor (IFR)<sup>1</sup> is an advanced power reactor concept sponsored by the U. S. Department of Energy's Reactor Development Program. The IFR features on-site reprocessing of the U-Pu-Zr fuel in which a pyrochemical electrorefining step is employed to recover the actinides and to separate them from the fission products. The electrorefining step is carried out at 500°C in a steel vessel containing a molten LiCl-KCl electrolyte on top of a pool of molten cadmium. Chopped fuel is anodically dissolved, and the actinides are electrochemically transported to cathodes in the LiCl-KCl salt. The deposited actinides can then be processed to make fresh fuel alloys and returned to the reactor.

Most of the fission products accumulate in the electrorefiner. Periodically, the salt and cadmium from the electrorefiner are treated in a series of steps carried out in equipment external to the electrorefiner; this treatment recovers residual transuranic (TRU) elements, purifies the salt and cadmium for return to the electrorefiner, and concentrates the fission products for conversion to high-level waste forms.

The cadmium is pumped through a sintered metal filter to remove noble metal fission products that accumulate in the cadmium. The filter housing retains a fraction of the cadmium pool and some soluble noble metals and zirconium. The salt also may be pumped through a filter to remove any insoluble impurities. The molten salt discharged from the electrorefiner contains about 1% of the actinides fed with the spent fuel, as well as the alkali metal, alkaline earth, rare earth, and halide fission products.

The salt discharged from the filter is passed through a multi-stage extraction step where it is contacted with a Cd-U solution to remove TRU elements, i.e., Pu, Np, Am, and Cm, from the salt. This leaves most of the rare earths in the salt. After the extraction step, the salt is stripped with a molten Cd-Li alloy to remove essentially all the remaining actinides. Most of the rare earths are also reduced from the salt and dissolved in the cadmium. The stripped salt retains all of the alkali metal, alkaline earth, and halide fission products and three rare earths (yttrium, samarium, and europium) whose chlorides are only partially reduced by lithium. Most of the stripped salt is returned to the electrorefiner, but a small fraction becomes waste and is immobilized in an aluminosilicate matrix, permanent waste form, for disposal.

The cadmium-rare earth solution from the stripper is combined with other metal wastes from the electrorefiner, e.g., cladding hulls, and the cadmium-containing noble metal fission products from the filtration step. These materials are combined and retorted to recover cadmium, which is returned to the process. A matrix metal, e.g., a copper alloy, is added during the retorting process to disperse and immobilize the residue, and form a permanent waste form.

Efforts are underway at Argonne National Laboratory to demonstrate the technology of waste treatment and immobilization for the IFR waste streams. The development work on the stripping step, as well as the pumping and filtration of the molten salt and cadmium, is discussed in this paper. Further details on the overall waste treatment and immobilization technology and development work on the extraction step are presented in companion papers<sup>2,3</sup> at this conference.

## II. BACKGROUND-PRELIMINARY STRIPPING TESTS

In small-scale stripping tests carried out earlier at ANL,<sup>4</sup> it was shown that actinide and rare earth chlorides are rapidly reduced by Cd-Li alloys at 500°C, but in some cases the reduced metals did not completely dissolve in the cadmium. In several runs with LiCl-KCl salt taken directly from a small-scale experimental electrorefiner, about 1 kg of salt was stirred in contact with a Cd-Li alloy having sufficient lithium to reduce the contained actinides and rare earths and produce a final cadmium solution with 0.1 wt % Li. Initially, the salt contained 1.3 wt % Pu, 0.0005% Am, 0.2% U, and 0.4% total rare earths. The plutonium and americium contents were intentionally at least 10 times higher than the TRU content expected in salt that would pass through the salt/metal extraction step. After the salt had been stirred in contact with the Cd-Li alloy at 500°C for 4 to 6 h, the plutonium content in the final salt was less than 1 ppm despite the high initial content. However, only about 30% of the plutonium appeared in the cadmium solution. The missing reduced metals were found to have formed a thin metallic layer on the crucible walls above the salt-metal interface. Because lithium metal is slightly soluble in chloride salts containing LiCl, it was thought that some lithium metal had transferred into the salt and produced particles of metallic actinides in the salt phase. The particles were then forced against the container walls by the action of the agitator.

In the small-scale tests, when an inverted steel cup containing a Cd-Li alloy was immersed in the liquid cadmium, the reduced actinides and rare earths were completely dissolved in the metal phase. This suggested that the lithium should be added gradually to obtain complete dissolution of the reduced metals.

## III. EQUIPMENT DESCRIPTION

After the small-scale tests, a prototype salt stripping system capable of handling uranium and non-radioactive rare earths was installed in a large argon-filled glovebox which also houses an engineering-scale electrorefiner.<sup>5</sup> Using salt from the electrorefiner, several series of tests will be done to obtain design and operating data for a full-scale stripper system, and for the equipment to remove spent salt and cadmium from the electrorefiner. The major components of this system are a stripper vessel, a transfer line/pump assembly, and filtration equipment.

The prototype stripper is a carbon steel vessel, 40 cm in diameter, housed in a heated well adjacent to the electrorefiner. It holds about 90 kg of molten salt (equivalent to a depth of about 65 cm) covering about 150 kg of molten cadmium (equivalent to a depth of about 18 cm); contains a single-shaft mixer with 45° flat paddles in both the salt and cadmium phases; has

baffles to ensure good mixing; and has provisions for investigating various methods of introducing Cd-Li alloys at a gradual and controlled rate.

A sampling device can be inserted into the stripper to obtain simultaneous samples of the salt and cadmium layers. The salt and cadmium samples are drawn into a tantalum tube through a sintered metal frit by opening a valve connected to a pre-evacuated cylinder. A "core" sample of the combined salt and cadmium layers can also be obtained by lowering a tube (that is open at the bottom) into the stripper vessel and then closing a poppet-like valve at the bottom to capture the "core" of material. Other instrumentation includes a contact-electrical-resistance liquid level probe and a reference electrode that measures the redox state of the salt/metal system.

The transfer line/pump assembly can be installed in either the electrorefiner or stripper vessel for transferring salt or cadmium. A schematic of the apparatus set up to transfer salt from the electrorefiner to the stripper is shown in Figure 1. The transfer line between the electrorefiner and the stripper vessel consists of 1/2 inch schedule 80 pipe. It is wrapped with heating cable along its longitudinal axis, and covered with about 2.5 cm of insulation and a stainless steel shroud. The line is pitched to drain away from the vessel from which the liquid is being pumped.

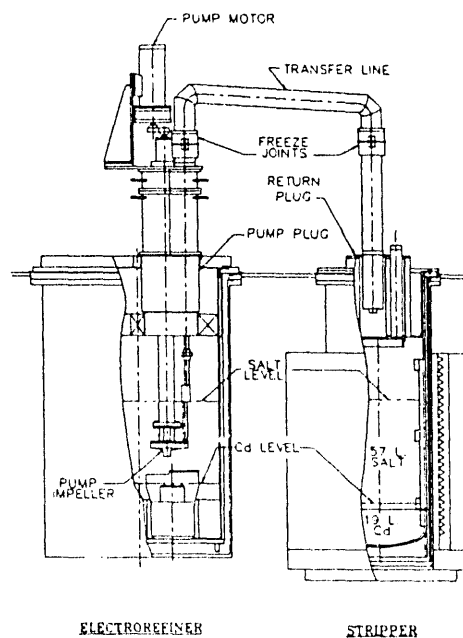


Fig. 1. Stripper Arrangement for Pumping Salt from Electrorefiner

Freeze joint assemblies are used to make the connections between the transfer line and the pump discharge line, as well as the stripper supply line, as shown in Figure 2. Silver solder (50 wt % Ag, 15.5% Cu, 16.5% Zn, and 18% Cd; melting point 630°C) is placed in the annular cup. The tubes are joined by melting the solder, inserting the skirt on the small (upper) tube in the molten solder, and allowing the solder to freeze. The primary liquid seal is provided by the metal-to-metal joint, with any leakage flowing back into the feed vessel through the annulus in the large (lower) tube.

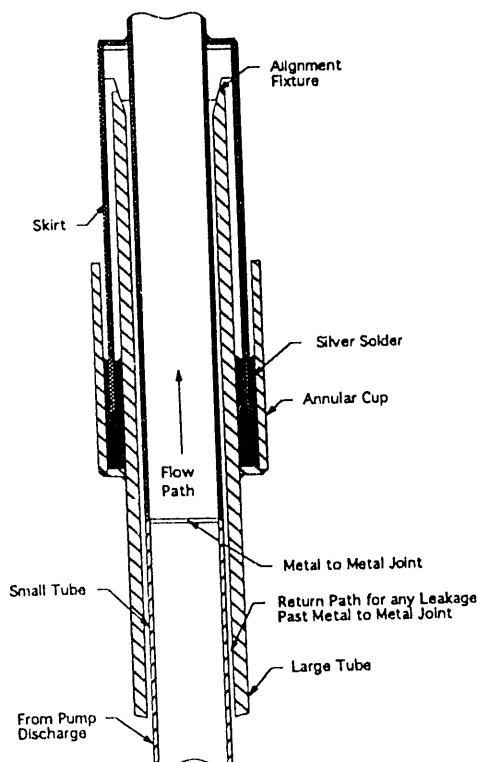


Fig. 2. Freeze Seal Connector

The pump is similar to a centrifugal sump pump but has several features to adapt it to high temperature service. The impeller section of the pump is shown in Figure 3. The lower end of the pump shaft is supported by a cast-iron journal bearing. To compensate for differential thermal expansion between the pump shaft and discharge line, which are supported on the same plate, the discharge line is slip-fit into the impeller housing.

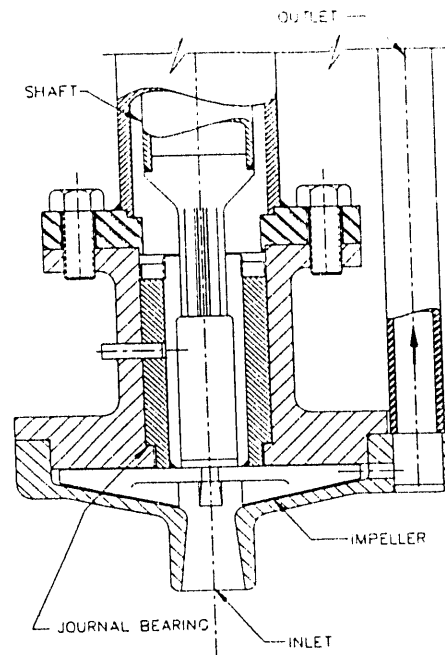


Fig. 3. High Temperature Pump-Impeller Section

A filter assembly can also be installed in the pump discharge line so that the pump can be used to circulate salt or metal from either the stripper or the electrorefiner through a cylindrical sintered metal filter (pore size of 13 and 35  $\mu\text{m}$ ). A schematic of the filter assembly is shown in Figure 4. The pumped fluid flows up the central tube to the top of the filter assembly, through the sintered metal filter, and then returns back to the supply vessel down through an annulus in the freeze seal assembly. The filter assembly has instrumentation designed to measure the liquid head and to estimate the flow rate of fluid through the unit by means of a force probe.

#### IV. TESTING PLAN

Initially, salt will be pumped from the electrorefiner to the stripper vessel. Cadmium is then added to the stripper vessel. During the stripper tests, lithium in the form of a Cd-Li alloy is added to the cadmium in a controlled manner, while the salt and metal are being stirred. The rates of disappearance of uranium and rare earths from the salt and their appearance in the cadmium phase are determined by periodically taking filtered samples of both phases and then analyzing them. The uranium and rare earths can be oxidized back into the salt by adding  $\text{CdCl}_2$  to the salt.

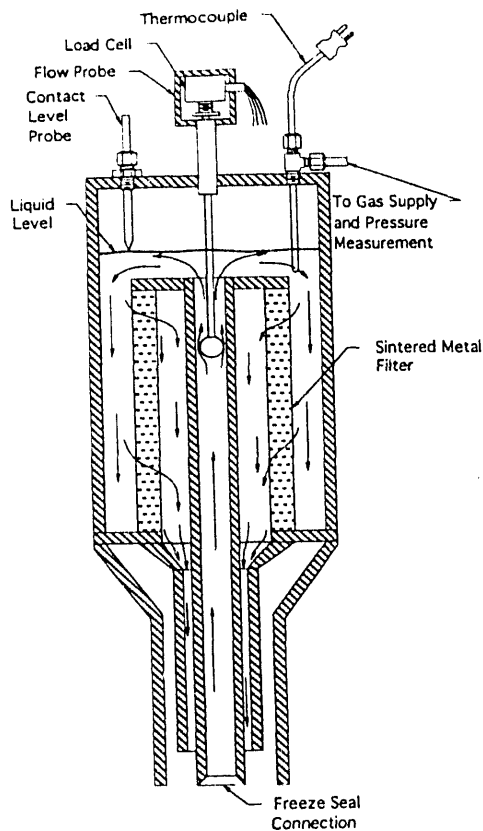


Fig. 4 Filter Assembly

One of the main purposes of the testing is to demonstrate an effective method for adding lithium to cadmium so that the reduced metals are completely dissolved in cadmium. Several tests will be made to investigate the effects of mixing and different methods of introducing Cd-Li alloys on the rate and extent of uranium and rare earth removals from salt. To avoid the formation of undissolved metallic compounds, three lithium addition methods may be explored: (1) immersion of an inverted cup holding a Cd-Li alloy below the salt-cadmium interface. The alloy will melt and be dispersed into the cadmium and react at the salt-cadmium interface; (2) injection of molten Cd-Li alloy from a tube placed in the stripper vessel; and (3) anodic dissolution of lithium from a solid Cd-Li alloy immersed in the salt.

Four pump filter tests are planned. The first two will be done in the stripper after the uranium and rare earths have been completely transferred to the cadmium. First salt and then cadmium will be filtered to collect insoluble materials, i.e., oxides, nitrides, or carbides of uranium and rare earths. The third and fourth filtrations will be done in the electrorefiner to collect and identify salt- and metal-suspended solids. The collection of molybdenum and ruthenium, which were introduced during the electrorefining of uranium alloys, is of special interest.

## V. INITIAL TEST RESULTS

After an extensive series of equipment handling operations and pretesting in a glovebox mockup was completed, the prototype salt stripping system was transferred into the glovebox. Subsequently a series of pumping tests was carried out to transfer salt from the electrorefiner to the stripper vessel. A photograph of this equipment in the glovebox during these tests is shown in Figure 5. Some difficulties were encountered when the pump would suddenly "lock up" and stop after short periods of operation. These difficulties were resolved by adjusting the clearances of the gear drive assembly, modifying the bearing housing assembly, and installing replacement bearings. Then 90 kg of LiCl-KCl salt (containing 1.22 wt % U, 0.71 wt % Nd, 0.45 wt % Ce and 0.11 wt % Y as chlorides) at 500°C was successfully transferred from the electrorefiner in several short pumping trials. The pumping rate was calculated from the change in salt level as determined by a single-point electrical resistance contact probe and previous calibrations of the stripper vessel with water. The level readings were also compared to the output from load cells that supported the stripper vessel. The pumping rate ranged from 4.0 to 5.5 L/min. at a speed of 800 rpm. The liquid head was about 1.5 m. The pump performance with molten salt was similar to its performance during out-of-box pretests with water.

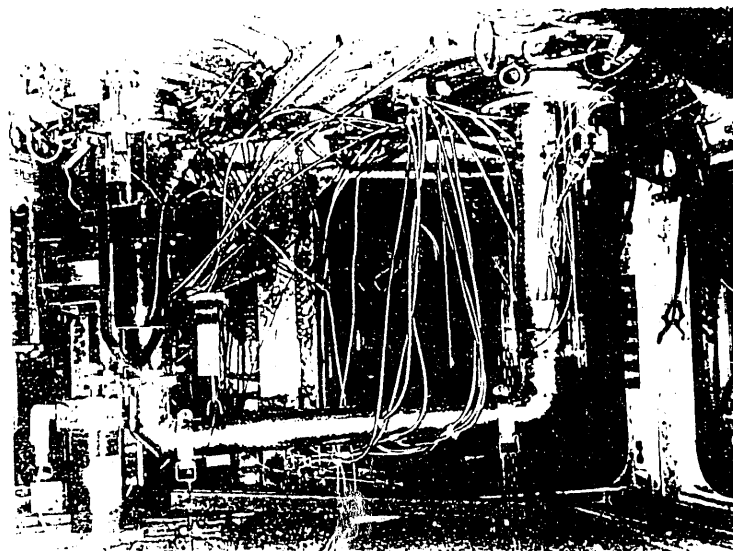


Fig. 5. Photograph of Salt Transfer Equipment

During the high-temperature tests, the freeze seals and transfer line also performed extremely well. The seals were easily made and also disassembled when the furnaces surrounding the seals were heated to about 700°C. There were no problems with alignment, sticking, or binding. (The argon glovebox atmosphere impurities, oxygen and water, measure <1 ppm.)

Some further development is needed to ensure that the pump will be capable of reliably pumping for longer periods of time, as will be required for future tests of filtration equipment. (During the above described pumping trials, the longest period of continuous pump operation was about 5 min.) Three modifications will be made to improve pump performance: (1) installing new upper bearings with more clearance; (2) replacing the gear drive with a more compliant belt drive, and (3) drilling a hole in the lower portion of the housing surrounding the shaft to ensure that the lower bearing is always flooded and hence lubricated.

## VI. STRIPPING EXPERIMENTS

After the salt was transferred to the stripper vessel, the pump and transfer line assemblies were disassembled and removed from the stripper vessel and electrorefiner. The top plug assembly was placed on the stripper vessel. This assembly has a port for an agitator and various other ports for charging materials, sampling salt and metal, and introducing other instrumentation. A total of 150 kg of cadmium (250-500 g pieces) was then charged through a loading chute into the stripper vessels equivalent to a depth of about 18 cm.

In the first series of stripping experiments, an inverted cup device was used to charge a Cd-15 wt % Li alloy into the cadmium layer at the bottom of the stripper vessel. The charging device consisted of an inverted cup on a rod. The cup contained the Cd-Li alloy which had been cast into it. A schematic of the stripper vessel set-up for these experiments is shown in Figure 6. The Cd-15 wt % Li alloy is a liquid at the 500°C temperature of the stripper contents and is lighter than cadmium. When the cup was immersed into the cadmium, the Cd-Li alloy melted and the lithium reductant was released slowly into the cadmium layer and then came into contact with the salt at the salt-cadmium interface. After insertion of the cup with the alloy, the mixer was turned on, and samples were taken after various time periods. The uranium and rare earth behavior during these first stripper tests (S1 and S2) is shown in Figure 7.

The uranium content of the salt that was transferred from the electrorefiner to the stripper was 1.22 wt %, as determined for a sample taken two days after the transfer. Analyses of salt samples taken approximately a month later (after cadmium had been added to the stripper vessel but before Test S1 was started) indicated uranium contents of 0.90, 0.95, and 0.80 wt %. This drop is assumed to be associated with the reactions of the uranium chloride with impurities (oxygen, moisture, etc.) in the cadmium or on the interior surfaces of the

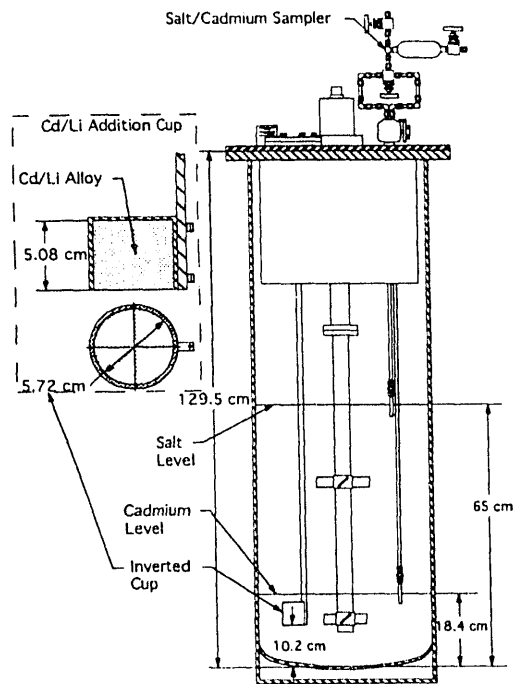


Fig. 6. Schematic of Stripper Vessel

stripper vessel. The drop in uranium concentration corresponds to 22-34% of the uranium in the original salt, but is relatively small on an absolute scale, i.e., 270-370 g uranium.

There was no comparable drop in the concentration of Nd (0.71-0.70 wt %), Ce (0.45-0.40 wt %), or Y (0.11 wt %). The range of values for the rare earths appeared to be within the estimated analytical uncertainty. The samples were analyzed using inductively coupled plasma - atomic emission spectrometry (ICP-AES) with an estimated uncertainty of  $\pm 10\%$ . The results are also consistent with thermodynamic predictions. Only uranium is expected to react with oxygen because it is thermodynamically the most stable oxide of the above in the presence of cadmium.

Stripper Test S1 was carried out at 500°C and a stirrer speed of 150 rpm. Filtered samples of the salt and metal phases were taken at approximately 1, 2, 6 and 24 h. As seen from the data, the uranium concentration of the salt decreased to a final value of 0.41-0.49 wt % within 6 h. Within the same time period of 6 h, the uranium concentration of the cadmium had increased and reached its final level of 0.31-0.32 wt %. The uranium material balance at the end of Test S1 shows 83% accounted for in salt and metal samples (40% in the salt, 43% in the metal), based on 1.22 wt % uranium in the original transferred salt. Coupled with the 22-34% originally converted to oxides, this yields a

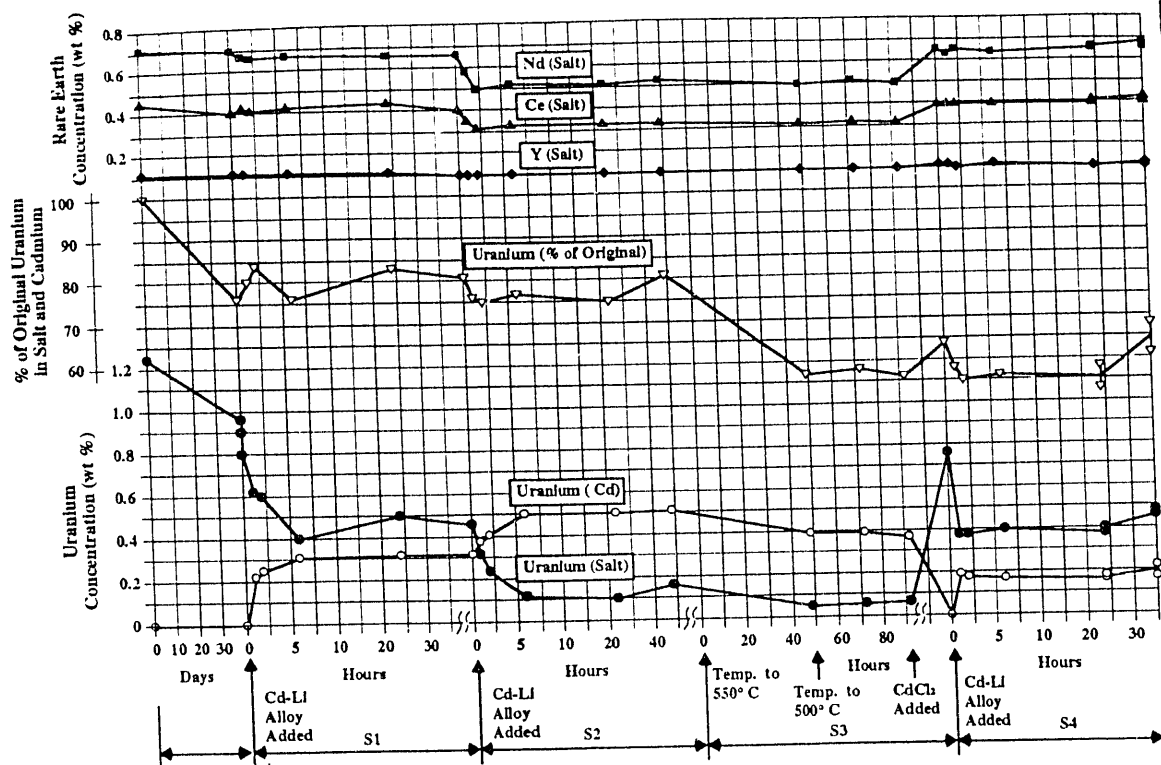


Fig. 7 Uranium and Rare Earth Behavior, Stripper Tests S1-S4

uranium material balance for S1 of 105-117%. During S1, the mixer stopped turning sometime between the 6 and 24 h samples.

Test S2 was started after replacement of a failed lower ball bearing on the agitator shaft with a Graphitar sleeve bearing. Like Test S1, Test S2 was carried out at 500°C with a stirrer speed of 150 rpm. A second inverted cup of Cd-15 wt % Li alloy was placed in the stripper vessel, which contained the salt and cadmium at the concentrations achieved at the end of Test S1. During Test S2, the uranium concentration in the salt decreased from a starting value of 0.46 wt % to a final value of 0.09-0.15 wt % within 6 h. The uranium concentration in the cadmium also reached its final value of 0.50-0.51 wt% in 6 h. The uranium material balance at the end of Run S2 remained at about the same level, i.e., 81% accounted for in the salt and metal samples (12% in salt, 69% in metal) and 22-34% as oxides.

The concentration of neodymium, cerium, yttrium in the salt during S1 and S2 is also shown in Figure 7. During Test S1, while the concentration of uranium was still relatively high in the salt, there was little or no decrease of these rare earth chlorides in the salt, and no appreciable increase in the cadmium metal phase.

During Test S2, neodymium and cerium started to transfer from the salt to the cadmium. Approximately 20% of the neodymium and 20% of the cerium were transferred from the salt to the cadmium. The neodymium and cerium concentrations in the salt and cadmium stabilized after only 2 h. The yttrium still remained in the salt. The material balance for tests S1 and S2 was 87% for neodymium and 74% for cerium. The behavior of the rare earths was as expected based on the equilibrium distribution of uranium and the rare earths. The transfer of uranium and rare earths between the salt and cadmium is dictated by the amount of reductant (Li) or oxidant ( $\text{CdCl}_2$ ) added, and the equilibrium between uranium and the rare earths as explained in a companion paper.<sup>2</sup>

The total amount of uranium, neodymium and cerium transferred from the salt to the cadmium during Runs S1 and S2 was in reasonable agreement with the cumulative amount of Cd-Li alloy added during these two tests. Sufficient Cd-Li alloy was added to transfer a total of about 5.6 g moles of uranium and rare earths, whereas, 4.6 g moles or 82% was actually transferred.

One of the purposes of Test S3 was to determine if raising the stripper temperature to 550°C would change



the uranium concentration of the metal samples, e.g., possibly dissolve any undissolved intermetallic uranium compounds that might be in the stripper. This test was carried out at a mixer speed of 300 rpm, i.e., twice that of the previous tests. At the end of Test S3, sufficient  $\text{CdCl}_2$  (1.6 kg) was added to the stripper vessel to oxidize the uranium and rare earths that were reduced by the Cd-Li alloy additions during Tests S1 and S2. The purpose of this addition was to determine if the stripper contents could be restored to the original starting conditions. The uranium and rare earth behavior during Tests S3 and S4 is shown in Figure 7.

Increasing the stripper temperature to 550°C did not increase the uranium content of the cadmium. In fact, the opposite occurred: the amount of uranium in the cadmium and salt decreased. The resultant material balance of uranium in the salt and cadmium decreased from about 81% to about 56-59%, based on the original uranium in the salt charged to the stripper vessel. This result may be due to increased reaction at the higher temperature of uranium with the stripper vessel wall. In a separate experiment, a thin layer of U-Fe intermetallic (thought to be  $\text{FeU}_2$ ) was found upon metallographic examination of a laboratory-scale electrorefiner crucible used in earlier tests.<sup>6</sup>

When the  $\text{CdCl}_2$  was added to the stripper, the uranium concentration in the salt increased to 0.76 wt %, which corresponds to 64% of the uranium contained in the original salt charge (1.22 wt %). With an assumed oxide content of 22-34%, the overall uranium material balance at the end of Test S3 was 86-98%.

Test S4 was the start of a series of Cd-Li alloy additions intended to reduce the uranium and rare earth concentrations in the salt to very low levels. During this test, the addition of the cup of Cd-15 wt % Li alloy lowered the uranium concentration in the salt from about 0.76 wt % to about 0.40 wt% in the first hour. Subsequent samples taken up to 24 h after the cup addition, were also about this level. This achievement of an apparent stable level within the first hour was much faster than Tests S1 and S2, but is not necessarily surprising since the mixer speed was doubled, to 300 rpm. The level of uranium in the salt that was achieved, (~0.40 wt %) is about the same as that achieved in Test S1 after the addition of the same quantity of Cd-Li alloy. The amount of uranium in both the salt and metal samples during Test S4 has remained at about the same level as Test S3, (55-65%) implying that no further conversion of uranium to oxides or some other inert form had occurred.

As discussed earlier, about 20% of the neodymium and 20% of the cerium were transferred from the salt to the cadmium during Tests S1 and S2. When  $\text{CdCl}_2$  was added at the end of Test S3, the levels of Nd and Ce in the salt were restored to essentially their starting values and remained at these levels during Test S4. During all of the tests (S1-S4), yttrium remained in the salt at the same level.

## VII. SUMMARY

A prototype salt stripper system, consisting of a stripper vessel, transfer line and pump assembly, and filter assembly has been tested in a glovebox mockup and installed in an engineering-scale argon-filled glovebox. A series of pumping trials was successful in transferring 90 kg of LiCl-54 wt % KCl salt at 500°C from the electrorefiner to the stripper vessel. The pumping rate ranged from 4.0 to 5.5 L/min. at a speed of 800 rpm. The liquid head was about 1.5 m. Further development work is necessary to extend the pumping duration.

The freeze seal connections and transfer line performed extremely well during the pumping trials. The seals were easily made and disassembled.

Tests have commenced with the prototype salt stripping system. In this system, the transfer of uranium and rare earths between the salt and the cadmium is dictated by the amount of reductant (Li) or oxidant ( $\text{CdCl}_2$ ) added, and the equilibrium between uranium and the rare earths. Several tests have been carried out to date at 500°C and mixing speeds of 150 and 300 rpm. An inverted cup charging device was used to introduce Cd-15 wt % Li alloy to the stripper vessel containing the LiCl-KCl salt with uranium, neodymium, cerium, and yttrium chlorides. Stable concentrations of uranium and rare earths were achieved in 6 h or less at 150 rpm and 1 h at 300 rpm.

Before the stripping tests started, the uranium content of the transferred salt decreased due to reaction of uranium chloride with impurities in the cadmium charge or on the interior surfaces of the stripper vessel. The uranium content of the salt and metal samples also decreased when the stripper vessel was heated to 550°C. However, the uranium material balance appears to have stabilized after these initial effects.

Cadmium chloride was added to the stripper after two additions of Cd-Li alloy. The  $\text{CdCl}_2$  addition reversibly transferred the uranium, cerium, and neodymium (which had transferred to the cadmium) back to the salt. In all the tests to date, yttrium has remained with the salt.

A series of Cd-15 wt % Li alloy additions with the inverted cup charging device has commenced. This series is intended to reduce the uranium and rare earth content of the salt to very low levels to demonstrate the salt stripping step in the reference process for treating waste salt. After the stripping tests, a series of tests will be initiated to evaluate the pumping and filtration of the salt and cadmium phases.

## VIII. ACKNOWLEDGMENT

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