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TESTING OF PYROCHEMICAL CENTRIFUGAL CONTACTORS*

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ABSTRACT

A centrifugal contactor that performs oxidation and reduction exchange reactions between molten metals and salts at 500°C has been tested successfully at Argonne National Laboratory (ANL). The design is based on contactors for aqueous-organic systems operating near room temperature. In tests to demonstrate the performance of the pyrocontactor, cadmium and LiCl-KCl eutectic salt were the immiscible solvent phases, and rare earths were the distributing solutes. The tests showed that the pyrocontactor mixed and separated the phases well, with stage efficiencies approaching 99% at rotor speeds near 2700 rpm. The contactor ran smoothly and reliably over the entire range of speeds that was tested.

I. INTRODUCTION

Argonne National Laboratory is investigating the electrometallurgical treatment of the spent nuclear fuels that exist in the Department of Energy complex. Electrorefining is a key step in this treatment. In the electrorefining step, fission products are separated from the actinides and build up in the molten chloride salt used as the electrolyte. Periodically, this salt is removed from the electrorefiner, and the accumulated fission products are recovered from the salt, immobilized in zeolite, combined with glass, and converted into a stable waste form. The salt can then be recycled to the electrorefiner for reuse. One of the steps in the waste treatment process involves the oxidation, reduction, or extraction of the "dirty" salt using continuous multistage countercurrent contactor. The objective of the testing program described in this paper was to determine the performance of a pyrochemical centrifugal contactor (pyrocontactor) with a single

stage for the oxidation, reduction, and extraction processes, and then develop a continuous countercurrent pyrocontactor with multiple stages.

II. TEST EQUIPMENT AND PROCEDURES

The pyrocontactor for treating molten salt is similar to the centrifugal contactor used for aqueous-organic solvent extraction processes.¹ However, the pyrocontactor operates at 500°C and handles liquid metal (~7.8 g/mL density) and molten salt (~1.65 g/mL density), while a conventional contactor processes an aqueous phase (~1.1 g/mL density) and organic phase (~0.8 g/mL) near room temperature. For high-temperature extractions with radioactive and fissile materials, the centrifugal contactor has several advantages over other types of contacting equipment. These advantages are (1) a compact size that minimizes the use of expensive hot-cell space; (2) low holdup and fast startup and shutdown that provide greater nuclear criticality safety; (3) a simple design that is relatively easy to build, operate, maintain, and replace; (4) a flexible design that allows a wide range of phase ratios, feed and product stream locations, and number of stages as dictated by the process; and (5) an overall system that can be controlled and adjusted quickly and reliably.

The pyrocontactor that was tested is shown schematically in Fig. 1. It consisted of a hollow rotor and a stationary housing. The equipment was made of Type 304 stainless steel. The heavy (metal) and light (salt) phases enter the contactor housing and flow down the annulus between the housing and rotor, where the fluids are mixed by vanes on the spinning rotor. Stationary vanes at the bottom direct the two-phase liquid dispersion toward the rotor inlet. As the liquids flow upward inside the rotor,

they are separated by the centrifugal force generated by the spinning rotor. The separated fluids pass through the overflow and underflow weirs at the top of the rotor and leave the contactor through their respective outlets.

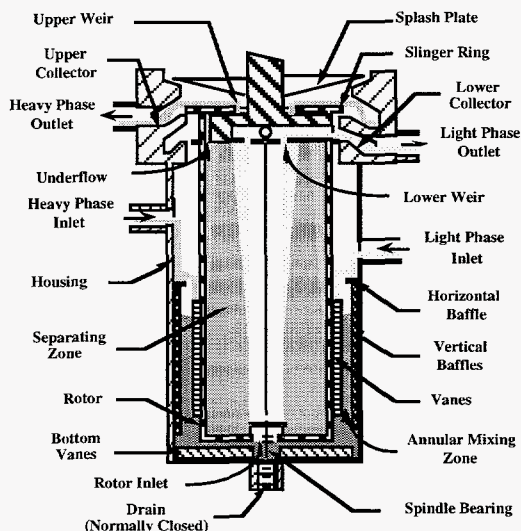


Fig. 1. Schematic of the Pyrocontactor

The rotor had a 4-cm O.D. and 7.46-cm length; the I.D. of the stationary housing was 6.35 cm. The internal rotor volume of 85 mL provided a residence time of about 5 s to separate the phases at the maximum flow rates of 500 mL/min for both the salt and metal. The maximum flow rates are high enough that the pyrocontactor could process 500 kg of waste salt in 12 h, which is equivalent to treatment of 1 metric ton of spent fuel with 3% burnup. For the liquid cadmium metal and chloride salt system, the diameters for the light- and heavy-phase weirs were 1.6 and 2.2 cm, respectively. The metal and salt inlets to the housing were below the lower collector ring but above the horizontal baffle on the housing. At speeds above 2100 rpm, the rotor provided sufficient pumping head that the metal and salt could be discharged at a level such that they could flow by gravity to the next stages in a multistage contactor bank. The splash plate prevented the release of liquid droplets and reduced the escape of cadmium and salt vapors.

The basic contactor design¹ was modified in several ways for high-temperature service. The hollow drive shaft was 3.2-cm O.D., 2.7-cm I.D., and 45.0-cm long. To protect the drive train from high temperatures, the upper bearings were 20 cm above the rotor and were insulated from the heat zone by heat shields and the hollow shaft. A spindle bearing made of tungsten was installed at the rotor bottom to provide additional support. The first natural frequency of vibration for the drive shaft was calculated to be 5800 ± 1200 rpm, which was well above the maximum operating speed of 2700 rpm.

Adequate mixing of aqueous-organic phases in centrifugal contactors can usually be achieved by the Couette flow between a smooth rotor and housing. However, the molten cadmium-salt system required more mixing power because of the high fluid densities, large density difference, and high interfacial tension between the metal and salt phases. Mixing vanes on the rotor and baffles on the housing were installed to provide the extra mixing power needed to ensure adequate mixing of the molten salt and metal in the annulus.

A diagram of the complete pyrocontactor test system is shown in Fig. 2. The test system included a single-stage pyrocontactor, feed tanks, raffinate tanks, treatment tanks, and transfer lines. These apparatuses were constructed of Type 304 stainless steel. The temperatures of the tanks, transfer lines, and contactor were maintained around 500°C by electrical resistance heaters. Orifices in the feed line controlled the flow rates to the contactor. The feed tanks were suspended from the glovebox ceiling by load cells that continuously measured the weight of the tank and contents. Each feed tank could hold up to 27 L liquid, which corresponds to 210 kg cadmium metal and 45 kg of the LiCl-41.5 mol% KCl eutectic salt. Metal and salt flow rates to the contactor were calculated from the load cell signals by a data acquisition system (DAS). These feed rates are time averaged over the test duration and are estimated to be accurate to within $\pm 15\%$. The glovebox containing these apparatuses had an argon atmosphere that was continuously purified to maintain the oxygen and water content below 15 ppm. This was necessary to avoid oxidation of the metal and salt solutions and to prevent corrosion of the stainless steel equipment by the salt.

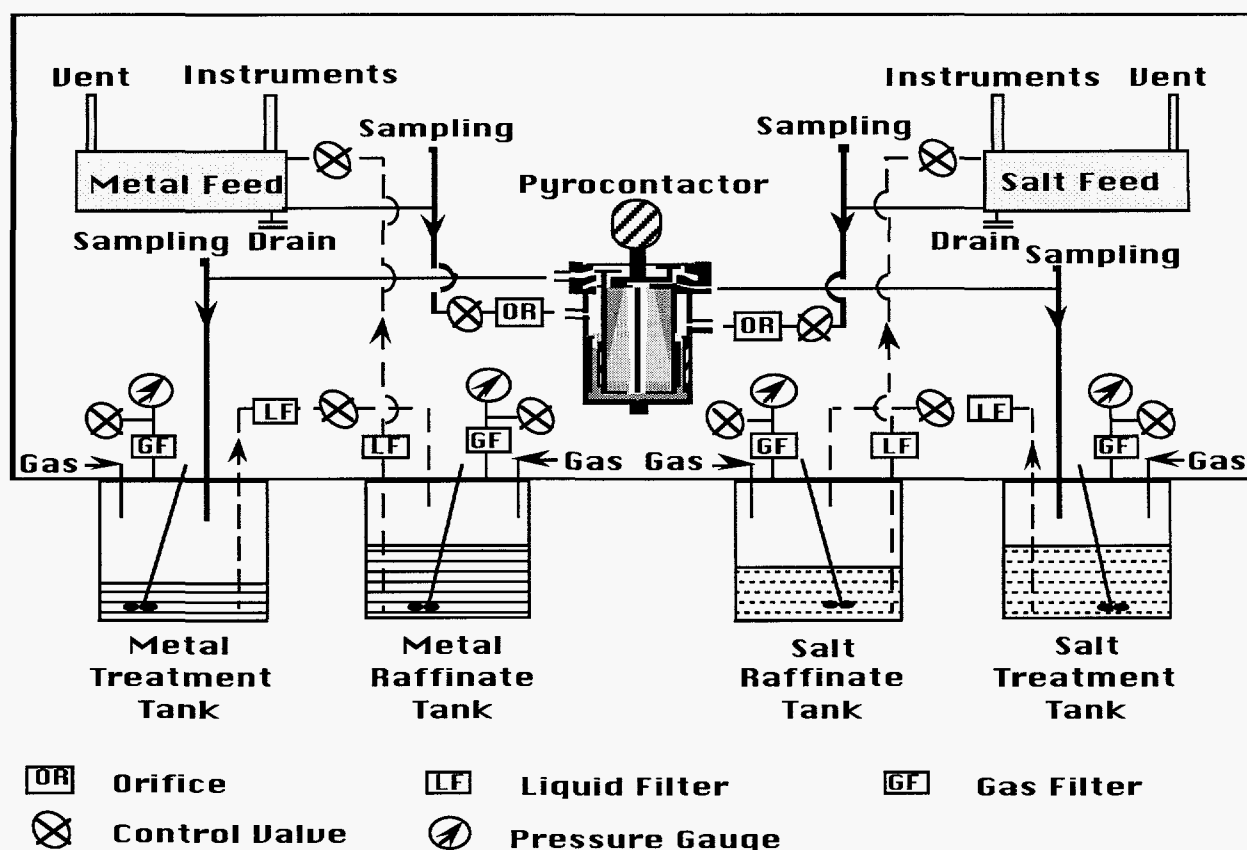


Fig. 2. Schematic of the Pyrocontactor Test System

Prior to a run, the salt and metal feed solutions were prepared, mixed in the treatment tanks, and then transferred by gas pressure to the raffinate tanks through sintered metal filters to ensure that the liquids contained no particles that could interfere with flow through the contactor. The filtered fluids were then transferred to the feed tanks by gas pressure. During a run, salt and metal flowed from the feed tanks to the spinning pyrocontactor by gravity. The feed rates were controlled by orifices in the feed lines. The effluents from the contactor were collected in their respective treatment tanks. The rotor speeds were varied between 2100 rpm and the maximum speed of 2700 rpm. Samples of the metal and salt feeds and effluents were collected from the transfer lines entering and leaving the contactor during the test. The weight fractions of each species in the feed and effluent samples were determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES). The analytical data are accurate to within $\pm 10\%$. The metal and salt feed rates were converted to mole fractions and used as input data to the XTRACT code^{2,3} to calculate the theoretical equilibrium concentrations in the effluents. The extraction efficiency was then determined from the

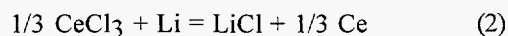
ratio of the measured amount of an element being transferred to the theoretical amount that could have been transferred. When the uncertainties in the determination of feed rates and chemical analysis of samples are accounted for, the extraction efficiencies are estimated to be accurate to within approximately $\pm 20\%$.

III. OXIDATION, REDUCTION, AND EXTRACTION TESTS

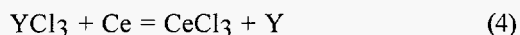
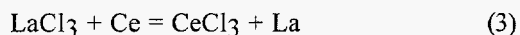
The first test series conducted was the oxidation of Ce by CdCl_2 :



The second test series was the reduction of CeCl_3 by Li:



The third test series was the separation of lanthanum from the chloride salts containing LaCl_3 and YCl_3 by contacting the salt with Ce:



This is possible because the separation factor for yttrium with respect to cerium is much higher than the separation factor for lanthanum, as shown in Table I. Here, the separation factor (SF) is defined as:

$$\text{SF}_{\text{La}} = [\text{La}/\text{LaCl}_3] [\text{CeCl}_3/\text{Ce}] \quad (5)$$

$$\text{SF}_{\text{Y}} = [\text{Y}/\text{YCl}_3] [\text{CeCl}_3/\text{Ce}] \quad (6)$$

Table I. Separation Factors for the Pyrocontact or Test Salt

Pyrocontactor Test Salt	
Elements	SF
Ce (Basis)	1
La	2
Y	88

At equilibrium, lanthanum prefers the metal phase while the yttrium prefers the salt. Calculated results⁴ shown in Table II suggest that when a 1 at.% Ce-Cd extractant is used, about 6% of the yttrium and 74% of the lanthanum are extracted from the test salt.

Table II. Calculations for One-Stage Extraction of Pyrocontactor Test Salts with Cerium

Extractant Concentration, mol% in LiCl-KCl	1 at.% Ce-Cd	
	Pyrocontactor Test Salt	
	Solutes in Salts	Percent Extracted
1.0	YCl_3	6
0.1	LaCl_3	74

IV. TEST RESULTS AND DISCUSSION

Several startup tests were run to establish proper operating conditions and demonstrate control of salt and metal flow through the contactor. Results from these tests indicated that the contactor should be operated at speeds above 1800 rpm to achieve effective phase separation. After the startup tests, oxidation, reduction, and extraction tests were done to measure extraction efficiency and demonstrate process performance.

A. Cerium Contacted with Cadmium chloride

In the first series, cadmium containing dissolved cerium was contacted with LiCl-KCl containing CdCl_2 . Two tests were conducted using a metal feed of 0.21 wt% Ce-Cd and a salt feed of the eutectic salt with 3.57 wt% CdCl_2 . During each of

these tests, the feed rates and the contactor speeds were varied. Samples of metal and salt feeds and effluents were collected. The test conditions, the measured data for the feeds and effluents, and the calculated results are summarized in Table III. The measured chemical compositions for the feed and effluent were determined from the chemical analysis of the collected samples by ICP-AES and the metal and salt feed rates. The theoretical values for the effluent samples were calculated from the feed values using the XTRACT code. The material balances were determined by dividing the sum of the compositions for each element measured in the metal and salt effluent samples by the overall compositions of the element in both the metal and salt feed samples. The extraction efficiency was determined from the ratio of the measured transfer of an element to the theoretical transfer of the element.

Table III. Summary for Ce-CdCl₂ Tests

Run Number	Run 1		Run 2				
			Run 2A		Run 2B		
Contacteur Speed, rpm	2680	2390	2670	2400	2100	2680	2400
Metal Feed Rate, mL/min	358	358	374	374	374	374	374
Salt Feed Rate, mL/min	483	483	225	225	225	651	651
Total Feed Rate, mL/min	841	841	599	599	599	1025	1025
Metal-to-Salt Volume Ratio	0.74	0.74	1.66	1.66	1.66	0.57	0.57
Metal Feed, mol/min							
Ce, measured	0.0299	0.0279	0.0292	0.0271	0.0292	0.0292	0.0292
Salt Feed, mol/min							
Cd, measured	0.04	0.039	0.0195	0.0185	0.0169	0.0507	0.0507
Ce, measured	0.00028	0.00045	0.0	0.0	0.0	0.0	0.0
Metal Effluent, mol/min							
Ce, measured	0.0004	0.00797	0.0173	0.0167	0.0271	0.0	0.00875
Ce, calculated	0.00272	0.00171	0.0158	0.0145	0.0176	0.0	0.0
Salt Effluent, mol/min							
Cd, measured	N/A ^a	0.07	0.00859	0.0175	0.0132	0.0325	0.0401
Cd, calculated	0.0	0.0	0.0	0.0	0.0	0.00695	0.00695
Ce, measured	N/A ^a	0.0165	0.0162	0.0141	0.00318	0.0284	0.0164
Ce, calculated	0.0275	0.0267	0.0133	0.0126	0.0116	0.0291	0.0291
Cerium Material Balance, %	N/A ^a	86	115	114	104	97	86
Stage Efficiency, %							
Metal Phase	108	76	90	83	18	100	70
Salt Phase	N/A ^a	61	122	112	28	97	56

^aN/A=none available.

The results of extraction efficiencies versus contactor speeds are shown in Fig. 3. The error bars shown are the estimated experimental error of $\pm 20\%$ based on the arithmetic means of the results. The arithmetic means of the efficiencies at contactor speeds of 2100, 2400, and 2680 rpm were 23, 76, and 103%, respectively. The uncertainties of the chemical analysis are $\pm 10\%$, and those of the feed rate are $\pm 15\%$, so the extraction efficiency data can be considered reliable since most of the data fell within $\pm 20\%$ of the mean values. The fact that the material balance data fell within $100 \pm 15\%$ further supports the validity of the data. Figure 4 shows that the extraction efficiencies increased as the contactor speeds increased for all runs in this test series. In summary, the pyrocontactor ran smoothly throughout these tests. It mixed and separated liquid metal and salt at 500°C as designed without any operating problem. Excellent extraction efficiencies were obtained at contactor speeds near 2700 rpm.

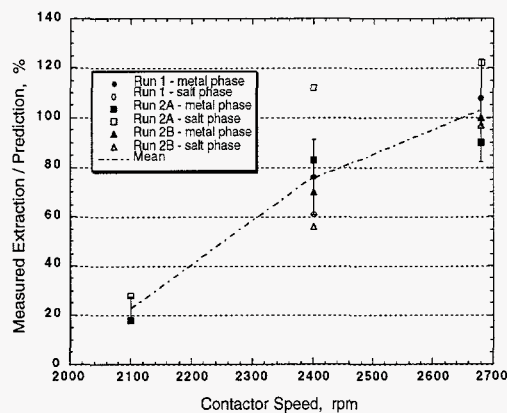


Fig. 3. Extraction Efficiency vs. Contacteur Speed for Ce-CdCl₂ Tests

B. Lithium Contacted with Cerium Chloride

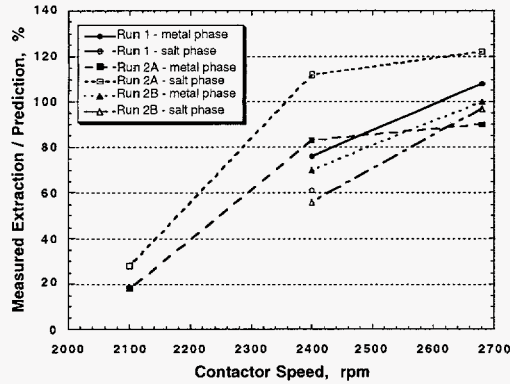


Fig. 4. Qualitative Trends of Extraction Efficiency vs. Contactor Speed for Ce-CdCl₂ Tests

In the second test series, salt containing CeCl₃ was contacted with cadmium containing lithium. The conditions and data for this test series are summarized in Table IV. Two tests were conducted. The metal feed in both runs contained 0.008 wt% lithium and 0.05 wt% cerium. The eutectic salt feed contained 0.093 wt% CeCl₃ and 0.055 wt% CdCl₂. The small amounts of cerium in the metal and CdCl₂ in the salt were left over from the first test series. The excess lithium in the metal feed had essentially extracted all of the cerium and cadmium from the salt at equilibrium. The cerium material balances, with one exception, were within 100 ± 10%.

Table IV. Summary for Li-CeCl₃ Tests

Run Number	Run 3				Run 4				
	Run 3A		Run 3B		Run 4A		Run 4B		
Contactor Speed, rpm	2650	2400	2100	2680	2400	2640	2400	2100	2660
Metal Feed Rate, mL/min	506	506	506	448	448	530	530	530	530
Salt Feed Rate, mL/min	345	345	345	345	345	480	480	480	379
Total Feed Rate, mL/min	851	851	851	793	793	1010	1010	1010	909
Metal-to-Salt Volume Ratio	1.47	1.47	1.47	1.30	1.30	1.10	1.10	1.10	1.40
Metal Feed, mol/min									
Ce, measured	0.0172	0.0161	0.0161	0.0147	0.0147	0.0207	0.0207	0.0207	0.0207
Li, measured	0.0569	0.0569	0.0569	0.0504	0.0504	0.0596	0.0596	0.0596	0.0596
Salt Feed, mol/min									
Cd, measured	0.0172	0.01520	0.0142	0.01470	0.01470	0.01200	0.01200	0.01200	0.00946
Ce, measured	0.0041	0.00366	0.0035	0.00354	0.00354	0.00509	0.00509	0.00509	0.00402
Metal Effluent, mol/min									
Ce, measured	0.0194	0.0186	0.0158	0.0160	0.00998	0.0236	0.0207	0.0207	0.0207
Ce, calculated	0.0213	0.0197	0.0196	0.0182	0.01820	0.0257	0.0257	0.0257	0.0247
Li, measured	0.0171	0.0228	0.0341	0.0100	0.0100	0.0119	0.0238	0.0298	0.0119
Li, calculated	0.0095	0.0145	0.0169	0.00969	0.00969	0.0190	0.0190	0.0190	0.0265
Salt Effluent, mol/min									
Cd, measured	0.00446	0.00405	0.00669	0.00314	0.00456	0.00352	0.00493	0.00634	0.00278
Cd, calculated	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Ce, measured	0.00085	0.00118	0.00370	0.00240	0.00455	0.00339	0.00453	0.00622	0.00223
Ce, calculated	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Cerium Material Balance, %	95	100	100	101	80	105	98	104	93
Stage Efficiency, %									
Metal Phase	84	81	57	99	99	117	88	73	144
Salt Phase	75	72	41	70	50	60	45	27	63

The extraction efficiencies versus contactor speeds are shown in Fig. 5 with $\pm 20\%$ error bars. The arithmetic means of the extraction efficiencies at contactor speeds of 2100, 2400, and 2680 rpm are 50, 73, and 89%, respectively. These data were more scattered than the results from the Ce-CdCl₂ extraction because of the presence of cerium in the metal feed and CdCl₂ in the salt feed. Yet, the majority of the data still fell within 20% of the mean extraction efficiencies. As the contactor speeds increased, the extraction efficiencies also increased, as shown in Fig. 6. Throughout this test series, the pyrocontactor ran smoothly, mixing and separating liquid metal and salt at 500°C without any operating problem. Excellent extraction efficiencies were again obtained at contactor speeds near 2700 rpm.

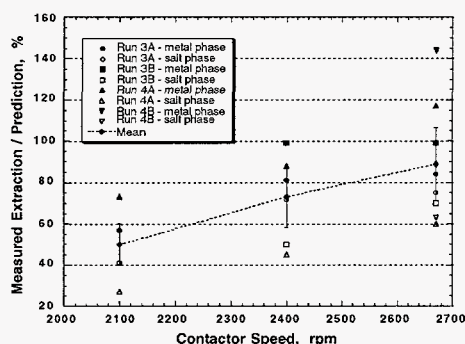


Fig. 5. Extraction Efficiency vs. Contactor Speed for Li-CeCl₃ Tests

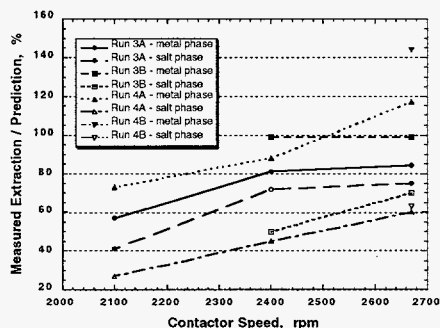


Fig. 6. Qualitative Trends of Extraction Efficiency vs. Contactor Speed for Li-CeCl₃ Tests

C. Cerium Contacted with Yttrium and Lanthanum Chlorides

The third test series used cerium to preferentially extract lanthanum from the eutectic salt containing lanthanum and yttrium chlorides. Two tests were conducted with the metal feed containing 0.28 wt% cerium and the eutectic salt feed containing 1.66 wt% LaCl₃ and 1.35 wt% YCl₃. The test conditions and results of the two runs are summarized in Table V. The extraction efficiencies versus contactor speeds are shown in Fig. 7. The arithmetic means of the extraction efficiencies at contactor speeds of 2100, 2400, and 2680 rpm were 41, 48, and 68%, respectively. These results are more scattered than those obtained in the first and second test series. In the previous tests, the only information needed to determine the extraction efficiency was the amount of cerium remaining in the extracted effluent, which should have been zero for a 100% efficient stage. To determine the extraction efficiency for the third test series, the amounts of cerium, lanthanum, and yttrium in the metal and salt phases and the separation factors of lanthanum and yttrium with respect to cerium are needed. Although more uncertainties were involved in determining the extraction efficiency for the Ce-LaCl₃-YCl₃ system, the majority of the extraction efficiency data still fell within the estimated experimental error of $\pm 20\%$. The material balances for the chemical species were mostly within $100 \pm 10\%$. The most encouraging result is that the concentration of yttrium in all metal effluent samples was less than 0.001 mol, which agrees very well with predictions. The extraction efficiencies increased with contactor speeds, as shown in Fig. 8.

Throughout this test series, the pyrocontactor ran smoothly, mixing and separating liquid metal and salt at 500°C without any operating problem. Excellent extraction efficiencies were obtained at contactor speeds near 2700 rpm.

TABLE V. Summary for Ce-LaCl₃-YCl₃ Tests

Run Number	Run 5				Run 6	
	Run 5A	Run 5B	Run 5C			
Contactors Speed, rpm	2680	2670	2400	2100	2680	2380
Metal Feed Rate, mL/min	428	433	282	282	380	380
Salt Feed Rate, mL/min	446	446	446	446	660	660
Total Feed Rate, mL/min	874	879	728	728	1040	1040
Metal-to-Salt Volume Ratio	0.96	0.97	0.63	0.63	0.58	0.58
Metal Feed, mol/min						
Ce, measured	0.0615	0.0628	0.0402	0.0404	0.0444	0.0445
La, measured	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Y, measured	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Salt Feed, mol/min						
Ce, measured	0.00237	0.00241	0.00257	0.00237	0.00932	0.01010
La, measured	0.0440	0.0453	0.0442	0.0440	0.0548	0.0526
Y, measured	0.0301	0.0301	0.0300	0.0301	0.0490	0.0493
Metal Effluent, mol/min						
Ce, measured	0.0458	0.0545	0.0298	0.0309	0.0296	0.0360
Ce, calculated	0.0393	0.0401	0.0222	0.0222	0.0241	0.0247
La, measured	0.0103	0.0131	0.0067	0.0044	0.0098	0.0064
La, calculated	0.0165	0.0170	0.0128	0.0127	0.0128	0.0125
Y, measured	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Y, calculated	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Salt Effluent, mol/min						
Ce, measured	0.0166	0.0150	0.0120	0.0089	0.0280	0.0218
Ce, calculated	0.0246	0.0252	0.0206	0.0206	0.0297	0.0299
La, measured	0.0321	0.0328	0.0375	0.0323	0.0470	0.0502
La, calculated	0.0275	0.0283	0.0314	0.0313	0.0420	0.0401
Y, measured	0.0272	0.0286	0.0299	0.0284	0.0429	0.0416
Y, calculated	0.0297	0.0297	0.0297	0.0298	0.0486	0.0491
Material Balance, %						
Ce	98	106	98	93	107	106
La	96	101	100	83	104	108
Y	90	95	100	94	88	85
Stage Efficiency, %						
Ce - Metal Phase	71	37	58	52	73	43
Ce - Salt Phase	64	55	52	36	92	59
LaCl ₃ - Metal Phase	62	77	52	35	77	51
LaCl ₃ - Salt Phase	72	74	53	92	61	19

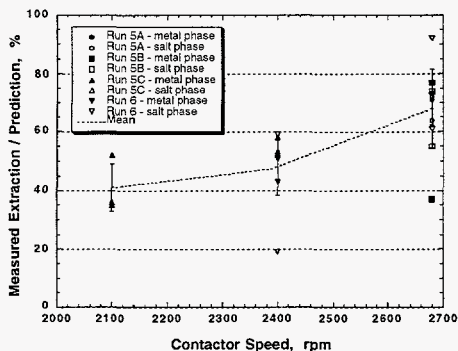


Fig. 7. Extraction Efficiency vs. Contactor Speed for Ce-LaCl₃-YCl₃ Tests

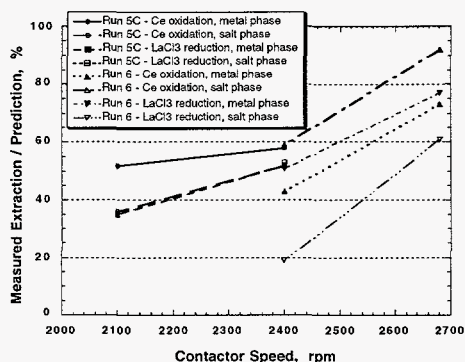


Fig. 8. Qualitative Trends of Extraction Efficiency vs. Contactor Speed for Ce-LaCl₃-YCl₃ Tests

V. CONCLUSIONS

(a) A single-stage pyrocontactor was successfully tested using chloride salts and cadmium alloys at 500°C with contactor speeds ranging from 2100 to 2700 rpm, total flow rates from 600 to 1040 mL/min, and metal-to-salt volume ratios from 0.57 to 1.66.

(b) The pyrocontactor ran smoothly for a total run time (accumulated) of about 100 h without any operating problem. The motor drive and its bearings were successfully isolated from the 500°C fluids. No vibration of the contactor was observed during operation.

(c) The total throughput of the contactor was high enough (up to 1040 mL/min tested) that 500 kg of waste salt can be processed in 12 h, or 1 metric ton of spent fuel with 3% burnup

can be treated. The effect of total throughput on the extraction efficiency was not obvious, even though a decrease in efficiency was expected when the total throughput increased.

(d) Since the extraction efficiency was high, we infer that the entrainment of salt in metal or metal in salt was low, although there were no direct experimental data.

(e) Results from the extraction tests (1) cerium in cadmium contacted with cadmium chloride in salt, (2) lithium in cadmium contacted with cerium chloride in salt, and (3) cerium in cadmium contacted with lanthanum chloride and yttrium chloride in salt indicate that the extraction efficiency increased as the contactor speed increased. The preferred operating speed for this pyrocontactor is near 2700 rpm, where the stage efficiency is at least 70%. Most of the extraction efficiency data for each contactor speed in the three series fell within $\pm 20\%$ of the arithmetic mean of the data. The material balance data are mostly within $100 \pm 10\%$.

VI. FUTURE WORK

Because of the success of the extraction tests with the single-stage pyrocontactor, a four-stage pyrocontactor unit was designed and fabricated. The four-stage unit is currently being tested to demonstrate its capability in providing continuous, countercurrent, multistage extraction.

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