

# **Evaluation of Mass Transfer Performance for Caustic-Side Solvent Extraction of Cesium in a Conventional 5-cm Centrifugal Contactor**

**January 2002**

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Nuclear Science and Technology Division

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## **ABSTRACT**

Tests have been conducted to determine if satisfactory mass transfer performance is achieved using a fully pumping 5-cm centrifugal contactor under conditions present in the Caustic-Side Solvent Extraction (CSSX) process. Tests utilized a commercially available contactor that had been modified by installation of a rotor housing bottom that had straight radial vanes on the process side. As received from the vendor, the housing bottom was equipped with curved (impeller-type) vanes that were intended to promote phase separation by minimizing mixing of influent solutions.

Stage efficiencies exceeding 85% were obtained under conditions, present in the extraction section of the CSSX flowsheet. Under CSSX stripping conditions the stage efficiency exceeded 90%. In both cases, the efficiencies obtained exceed the minimum requirement for acceptable transfer of cesium in the CSSX process.





## 1. INTRODUCTION

This report describes the third in a series of three tests designed to evaluate the mass transfer performance of a 5-cm centrifugal contactor for cesium transfer in a Caustic-Side Solvent Extraction (CSSX) process. The objective was to determine if acceptable cesium transfer efficiencies could be obtained in a 5-cm centrifugal contactor that was operated in the fully pumping mode.

Results of two previous tests indicated that certain elements of centrifugal contactor configuration have significant impacts on mass transfer efficiency. In the first test, commercially available contactors of the manufacturer's standard design were used in single-stage and multistage mass transfer tests. The contactors, which were operated as received from the vendor, are similar to devices that have been designed and operated at the U.S. Department of Energy's (DOE's) Argonne National Laboratory (ANL), Oak Ridge National Laboratory (ORNL), and Savannah River Site (SRS). Unlike previous designs, the commercial contactors utilize a rotor bottom plate that has curved impeller-type vanes on the process side of the housing.

All contactor designs generated at DOE facilities incorporate straight vanes on the rotor bottom plate. Placement of vanes in the region below the contactor rotor in close proximity to the rotor is needed to mitigate vortex formation that would prevent flow into the rotor. Use of curved vanes by the vendor is intended to enhance phase separation by minimizing mixing and reflects the target application of the vendor—the separation of petroleum fractions from water. Results of throughput tests using the as-received contactors indicated that the curved-vane design resulted in poor retention of liquid in the region of the contactor between the rotor and stationary housing. Mixing of influent solutions in this region is essential for effective solute transfer in solvent extraction operations. Mass transfer test results using the as-received units reaffirmed this concept: stage efficiencies obtained using the as-received contactor were low, particularly under stripping conditions.

To improve phase mixing, a commercial contactor was modified by replacing the housing bottom plate with a plate having straight vanes. This modification increases both turbulence and pressure drop in the rotor housing and results in increased retention of

solutions in the contactor's mixing zone. Additional retention of liquid in the mixing zone was obtained by increasing the size of the opening in the bottom of the rotor. Based on results of hydraulic calculations, the rotor bottom opening was enlarged sufficiently that the contactor could not pump solution to the outlet ports without becoming partially flooded. The resultant increase in liquid retention in the contactor mixing zone promotes solute transfer by increasing the interfacial area available for solute transfer between phases.

Single-stage mass transfer tests performed using the modified contactor produced efficiencies under extraction and stripping conditions that exceeded the 80% acceptance criteria.

In the test reported herein, a commercial contactor was used with an unmodified (fully pumping) rotor in combination with a straight-vane housing bottom. To allow direct comparison of results, flow and rotor speed conditions applied in the current tests were identical to those applied in the previous test program.<sup>1</sup>

## **2. EXPERIMENTAL PROGRAM**

The experimental program had two components: (1) verification of acceptable contactor operation at flow conditions applicable to the mass transfer test effort and (2) the mass transfer test itself.

### **2.1 EQUIPMENT**

#### **2.1.1 Centrifugal Contactor**

The 5-cm centrifugal contactor used in the subject test program was obtained from CINC, Inc. (Carson City, NV), as a standard-design item (model V-2). The unit, one of four obtained in July 2000, was used in previous multistage mass transfer testing but had not been used in single-stage testing. The version of the V-2 that was used in the subject test differs from the unit used in previous single-stage evaluations in that the inside diameter of the housing is increased from the top of the housing to the bottom lip of the light-phase collection ring. The outside diameter of the rotor is also increased from a point below the light-phase discharge ports to the aqueous discharge, so that the rotor extends over the lip of the light-phase collection ring. The version of the V-2 contactor

(procured in the late 1990s) that was used in previous single-stage tests was equipped with a rotor that had a uniform outside diameter over its entire length. Accordingly, the inner bore of the contactor housing was also uniform from top to bottom. It is assumed that the modification in the newer contactor design was intended to minimize spillage from the light-phase discharge ports into the contactor's mixing zone. The diameter offset also allows for placement of a screwed connection in the rotor body just below the organic weir so that the lower rotor section can be separated from the section containing the heavy- and light-phase weirs. This permits the user to access the lower portion of the rotor for inspection and cleaning purposes. The rotor diameter offset is achieved by increasing the thickness of the rotor wall and does not affect the hydraulics inside the rotor.

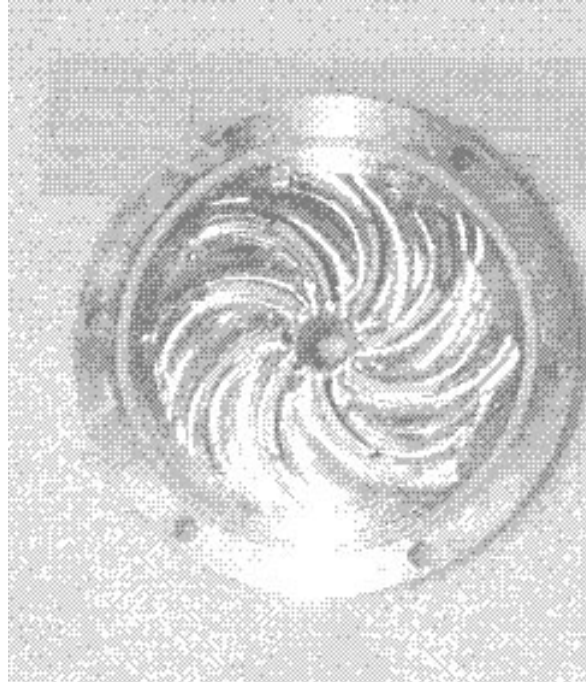
The contactor was powered by a 110-vac single-phase motor, which was controlled by a variable-frequency drive. The maximum frequency of the drive controller was 100 Hz, which corresponds to a nominal rotor speed of 6000 rpm. All wetted contactor components had been fabricated from 316L stainless steel, TFE Teflon™, or TFE-encapsulated Viton™.

As described in the Introduction, the as-received contactor was shipped with the housing bottom plate shown in Fig. 1. The direction of the vane curvature matches the direction of rotation of the contactor to reduce turbulence as fluid moves from the annulus between the rotor and housing into the region below the rotor. Figure 2 is a photograph of the modified housing bottom used in the subject test.

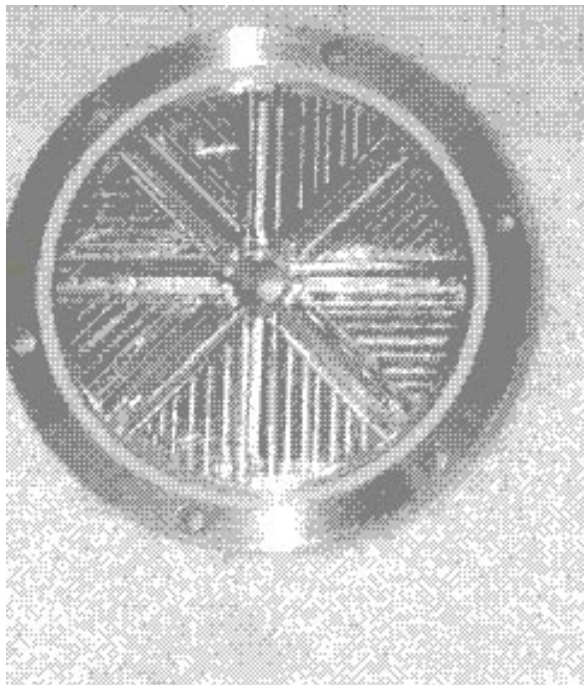
Prior to its use in the reported test, the contactor was dismantled and was cleaned with dilute acid and demineralized water.

### **2.1.2 Pumps**

All solution transfers were performed using piston-type metering pumps (Fluid Metering Inc., Model QV-2) with a maximum nominal throughput of 1296 mL/min. The pump heads were equipped with 300 series stainless steel sleeves and ceramic pistons.



**Fig. 1. Photograph of contactor housing bottom as received from vendor.**



**Fig. 2. Photograph of contactor housing bottom after modification.**

### 2.1.3 Vessels and Tubing

The vessels used to supply and receive solutions (15-L capacity high-density polyethylene tanks) were procured from VWR Scientific Products (Oak Ridge, TN, catalog no. 60464-043).

All tubing used during testing was PFA Teflon™; all tubing fittings were constructed from 304 L stainless steel.

### 2.1.4 Thermometers

The temperature of solution drained from the contactor mixing zone during mass transfer tests was determined using digital thermometers that were procured from the Cole-Parmer Instrument Co. (Vernon Hills, IL, catalog no. P-90003-00). The thermometers used are certified to have resolution to within 0.1°C up to 199.9°C.

## 2.2 CHEMICALS

The CSSX solvent used in the subject test was the baseline formulation as described in Table 1. The solvent had been used in previous contactor throughput and mass transfer testing<sup>1</sup> and was used most recently in tests to determine the ability of the contactor to process sludge-containing waste supernatant.<sup>2</sup> Prior to its use in the reported test program, the solvent was clarified by gravity settling to remove solids remaining from the preceding test. The solvent was then scrubbed by contact with 0.05 *M* HNO<sub>3</sub>, stripped with 0.001 *M* HNO<sub>3</sub>, and washed with 0.01 *M* NaOH. Scrubbing, stripping, and washing operations were performed using the same contactor that was used in the mass transfer test reported here.

Samples of the solvent were collected and processed through a series of cesium extractions, acid scrubs, and dilute acid strips by personnel from the ORNL Chemical and Analytical Sciences Division (currently the Chemical Sciences Division) to verify the effectiveness of the wash procedure.<sup>3,4</sup> Because of schedule constraints, the reported mass transfer test was performed before results of the solvent analysis were received. Under all conditions, the solvent analysis found cesium distribution coefficients that were significantly higher than those obtained

**Table 1. Composition of CSSX solvent**

Component	Concentration
Calix(4)arene-bis-( <i>tert</i> -octylbenzo-crown-6) (aka BOBCalixC6)	0.01 <i>M</i>
(2,2,3,3-Tetrafluoropropoxy)-3-(4- <i>sec</i> -butylphenoxy)-2- propanol (aka Cs-7SB)	0.50 <i>M</i>
Trioctylamine	0.001 <i>M</i>
Isopar® L	Balance

using pristine solvent. Distribution results for the solvent used in the reported test and for pristine solvent are listed in Table 2.

In addition to determining distribution coefficients for the solvent, density and dispersion number determinations were made as part of an evaluation of alternative solvent formulations. Data obtained are presented in Table 3. Comparison of the values listed with those for pristine baseline CSSX indicates that the solvent characteristics had changed significantly. It is suspected that the change in solvent physical properties was due to evaporation of Isopar L® diluent from the blend. Loss of diluent and the resulting concentration of the remaining solvent components are likely causes for increased density, increased distribution coefficients, and impaired phase separation.

Scrub (0.05 *M* HNO<sub>3</sub>) and strip (0.001 *M* HNO<sub>3</sub>) aqueous solutions were prepared using 0.10 *N* HNO<sub>3</sub> procured from J. T. Baker Co. and diluted with water that had been deionized using a Barnstead Nanopure® filtration system. Sodium hydroxide solutions used to wash the solvent were prepared using a standard 0.1 *N* sodium hydroxide solution (ACS reagent grade, procured from J. T. Baker Co.).

SRS waste supernatant simulant was prepared according to SRS procedure WSRC-RP-2000-00361, Rev. 0, and had the composition listed in that document for “average” SRS supernatant simulant.<sup>5</sup> The nominal cesium concentration in all supernatant simulant that was used in testing was 0.00014 *M*.

**Table 2. Results from solvent analysis by cesium distribution determination**

Condition	D <sub>Cs</sub> for pristine solvent	Mean D <sub>Cs</sub> for solvent used in 3 <sup>rd</sup> mass transfer test	Range of D <sub>Cs</sub> values for the test solvent
Extraction	18.7	48.7	19.4
1st Scrub	1.66	7.24	0.08
2nd Scrub	Not available	4.75	0.19
1st Strip	0.141	0.535	0.036
2nd Strip	0.083	0.302	0.008
3rd Strip	0.062	0.261	0.020
4th Strip	0.056	0.218	0.003

**Table 3. Properties of CSSX solvent used in mass transfer testing**

Property	Pristine solvent	Solvent after recovery from 3rd mass transfer test
Density	0.81–0.82 at 25°C	0.9256 at 25°C
Extraction dispersion number	$1.49 \times 10^{-3}$	$5.93 \times 10^{-4}$
Scrubbing dispersion number	$9.4 \times 10^{-4}$	$7.78 \times 10^{-5}$
Stripping dispersion number	$1.15 \times 10^{-3}$	$9.09 \times 10^{-4}$

## 2.3 EXPERIMENTAL PROCEDURES

### 2.3.1 Contactor Throughput Verification

Evaluations of contactor throughput and phase separation capability were conducted to verify that acceptable performance was obtained at conditions applicable to the subsequent mass transfer test. In these tests, the contactor was operated under extraction and stripping conditions (i.e., organic-to-aqueous flow ratios and stream compositions) over a range of flow rates and rotor speeds. For each test condition,

samples of the aqueous and organic effluents were collected and examined for contamination with the opposing phase.

Contactors hydraulic verification tests were performed with the contactor configured for constant recycle, as shown in Fig. 3. For any particular test condition, rotor operation was initiated at 4000 rpm. As soon as the target speed was reached, flow of aqueous solution was started and continued without solvent flow until solution was observed in the aqueous discharge line. At that point, the flow of organic solution was initiated.

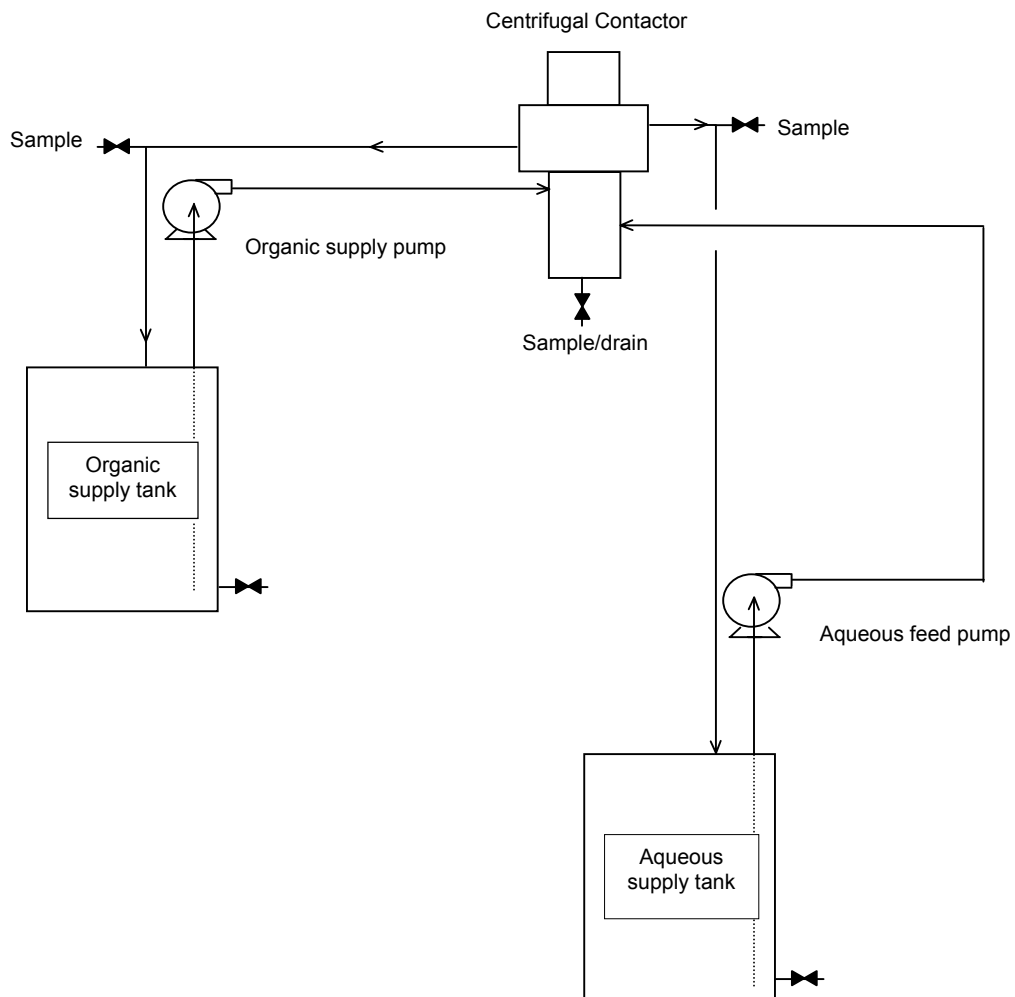
After operation of the test loop for at least 3 min, samples of both effluent streams were collected and examined visually for cross-phase contamination. If no contamination was observed, the contactor rotor speed was reduced incrementally and sampling was repeated. This process was continued until either of the effluent streams exhibited contamination with the opposing phase. The first observation of contamination of organic samples was the appearance of very small aqueous droplets in the bottom of the sample collection beaker. The first indication of organic carryover in the aqueous effluent was the appearance of an organic film floating on top of the aqueous sample.

If contamination was found in either or both effluent streams, the rotor speed was increased, the system was operated for at least 3 min, and sampling was repeated. Incremental rotor speed increases were made in this manner until the system was restored to acceptable phase separation performance. After a bounding combination of flow rate and contactor speed was established, the rotor speed was reset to 4000 rpm, flows were increased proportionally, and the determination procedure was repeated. Testing was continued until adequate phase separation could not be achieved at 4000 rpm, or until a pump capacity limit was reached.

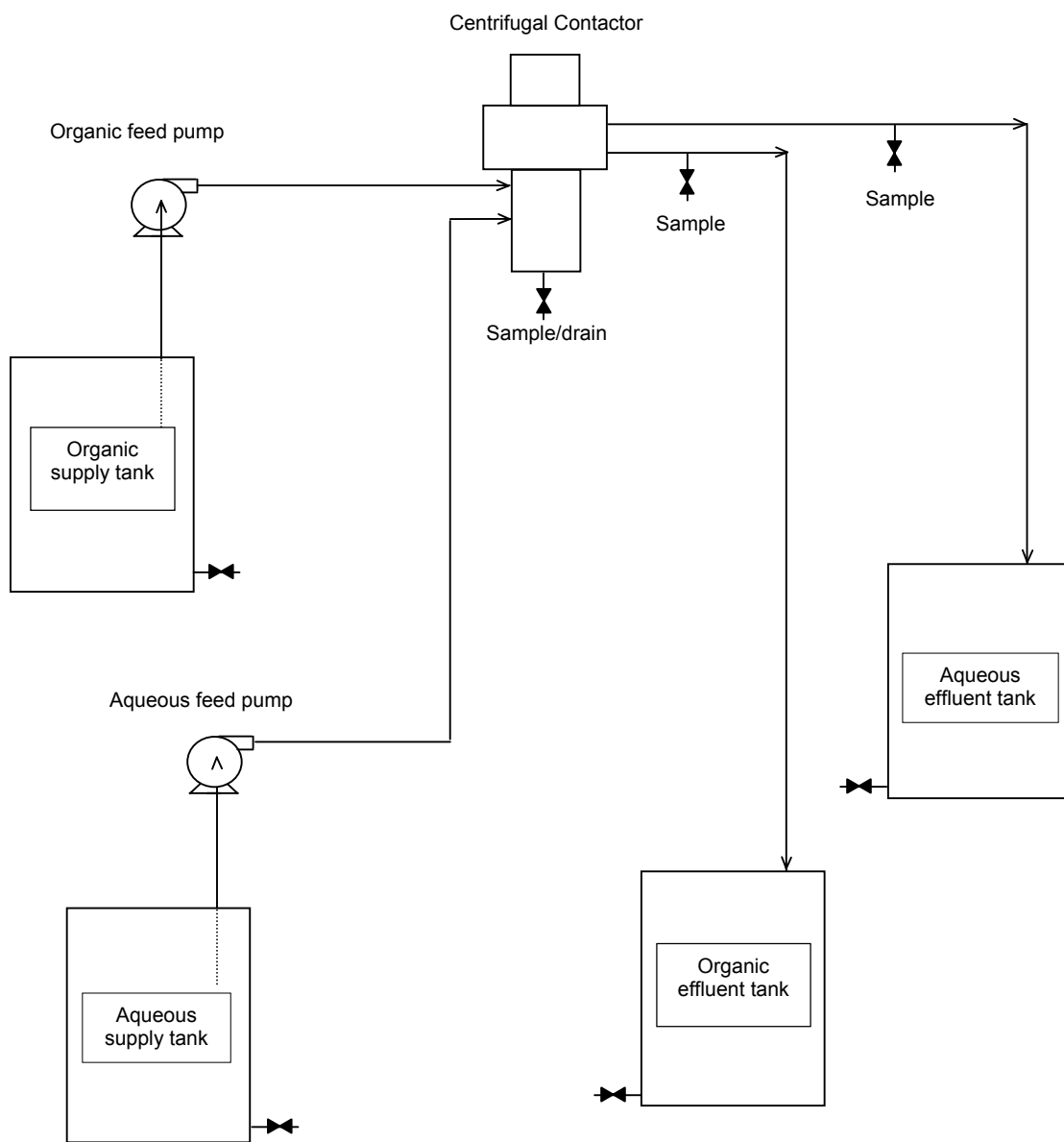
### **2.3.2 Mass Transfer Evaluations**

Mass transfer efficiency determinations were performed with the contactor and ancillary equipment configured for once-through processing of solutions as shown in Fig. 4. Testing was performed under conditions present in the extraction, scrubbing, and stripping sections of the CSSX cascade. All tests were performed at a rotor speed of





**Fig. 3. Equipment configuration for contactor hydraulic tests.**



**Fig. 4. Equipment configuration for mass transfer testing.**

3600 rpm. Single sets of flow conditions were applied in each phase of testing. Flow rates were established at values used in the previous mass transfer test<sup>1</sup> to facilitate direct comparison of results.

For each test condition, the contactor was empty when rotation was started. Aqueous feed was introduced at the test flow rate until solution was observed in the aqueous discharge line. At that point, the flow of organic was initiated at the designated rate and the experiment clock was started.

In extraction-mode testing, duplicate samples of the aqueous and organic effluents were collected after operating for a period equal to at least 4 contactor residence times and again after a minimum of 8 residence times. Based on a contactor operating volume of 250 mL, 4 residence times was equivalent to 1 min 35 s and 8 residence times was equal to 3 min 10 s under extraction test conditions. Due to the limited inventory of solvent available for testing, stripping test samples were collected after 3 and 4 contactor residence times (4 min 10 s and 5 min 34 s, respectively).

Following collection of the second set of samples for each set of test conditions and with the rotor still turning, approximately 50 mL of solution was drained from the contactor through a valve connected to the housing bottom. The temperature of this solution was measured immediately. Temperature measurements were not made following collection of the first sample under each condition to avoid upsetting the system prior to the second sampling.

Between test phases (i.e., before the scrubbing and stripping tests), the contactor was dismantled and was rinsed with demineralized water to prevent carryover of solutions from the preceding test phase.

To determine the efficiency of the contactor, aliquots were taken from each sample collected and equilibrated under the conditions (phase ratio and temperature) present during testing. Equilibrations were performed by placing the sample aliquots into sealed cuvettes that were submerged in a controlled-temperature water bath set at the temperature that was recorded during the second sampling. After equilibrating thermally for at least 15 min, the cuvette contents were agitated manually for two 20-s intervals with an intermediate 10-s hold period. The samples were returned to the water bath and

were allowed to separate under gravity. After at least a 10-min period of separation, the cuvettes were placed in a laboratory centrifuge to separate the phases completely. After centrifugation, the cuvettes were returned to the water bath for at least 5 min prior to collection of samples. All cesium analyses were performed using inductively coupled plasma–mass spectrophotometry.

Mass transfer efficiencies were calculated using the Murphree definition based on the aqueous-phase concentrations.

### **3. RESULTS**

#### **3.1 HYDRAULIC PERFORMANCE**

Testing was performed under extraction and stripping conditions. Testing under scrubbing conditions was not performed due to the similarity in scrubbing and stripping conditions and the nearly identical results that had been obtained under these conditions in previous tests.

Evaluations of hydraulic performance were limited to verification that the 5-cm contactor obtained from the vendor and configured with a straight-vane bottom plate provided acceptable phase separation at flow conditions to be applied in mass transfer tests. Results obtained early in extraction-mode hydraulic testing indicated that operation of the CINC contactor, as received, was limited to  $\leq 4000$  rpm due to transfer of organic feed solution into the aqueous feed inlet. This observation is described and an explanation for it is presented in the following section of this report.

##### **3.1.1 Extraction-Condition Throughput Performance**

Testing under extraction conditions was performed with an 0.950-in.-diam aqueous weir plate installed in the rotor. This is the same weir size that was used in hydraulic and mass transfer tests using a partially pumping rotor in combination with a straight-vane housing bottom. Unlike previous tests, visual observation of fluids in the contactor was not possible because the only available housing equipped with an observation window was the earlier version of the model V-2 contactor. The only rotor

compatible with this housing had been permanently converted to partial-pumping operation by enlargement of its lower-end opening.

Testing was started at flows approximating those to be used in the mass transfer test: 500 mL/min simulant plus scrub and 154 mL/min solvent. (Simulant and scrub solutions were combined in the proportion present in the extraction section of the baseline CSSX flowsheet.) Operation was verified at a rotor speed of 4000 rpm. Contamination of the aqueous phase appeared when the rotor speed was reduced to 3200 rpm. The contamination was eliminated when the rotor speed was increased to 3600 rpm and began to reappear as small droplets at 3500 rpm.

Flows were reduced proportionally to an aqueous rate of 400 mL/min and an organic rate of 123 mL/min. Effluent samples collected at rotor speeds ranging from 2000 to 3500 rpm were free of contamination. Testing at lower speeds was not performed since phase mixing at such speeds is not representative of normal contactor operation.

The rotor speed was increased to 4200 rpm, and flows were increased to 600 mL/min aqueous and 185 mL/min organic. Initially, no effluent contamination was observed. After the rotor speed was decreased to 4000 rpm, a significant amount of aqueous contamination was found in the organic discharge. In an attempt to reestablish acceptable phase separation, the rotor speed was increased—first to 4500 rpm, then to 5400 rpm. The contamination problem became more severe with each increase in rotor speed.

Because of the accumulation of aqueous solution in the organic catch tank, the test loop was shut down and the solution in the tank was separated using separatory funnels. Testing was resumed with verification of results obtained at the initial conditions (500 mL/min simulant/scrub and 154 mL/min solvent at 4000 rpm). Subsequently, the flows were reset to an aqueous rate of 600 mL/min and an organic rate of 185 mL/min. No evidence of effluent contamination was found at a rotor speed of 4000 rpm, or when the speed was reduced to 3800 rpm. When the rotor speed was increased to 4800 rpm, a significant volume of aqueous solution was found in an organic effluent sample of approximately 50 mL. A reduction in organic effluent contamination was observed after reducing the rotor speed to 4000 rpm, and contamination was

eliminated by operation at 3800 rpm. Contamination reappeared when the rotor speed was increased to 4400 rpm.

Because contamination occurred at high rotor speeds and the contamination mode (aqueous in organic) was atypical of operation at low organic-to-aqueous flow ratios (which create dispersions with continuous aqueous phases), it was suspected that the dispersion was being carried over from the mixing zone into the organic collection ring. It was hypothesized that the orientation of the inlet feed lines (normal to the housing and rotor walls) permitted a portion of the entering flow streams to splash upward when entering the contactor mixing zone due to impingement on the rotor. At high rotor speeds, some of this material could obtain enough upward momentum to reach the lower (organic) collector ring. In order to test this hypothesis, aqueous and organic flows were reduced to rates that had been processed successfully at 3600 rpm (500 mL/min aqueous and 154 mL/min organic). After confirming acceptable hydraulic performance at these conditions, the rotor speed was increased to 4200 rpm. This adjustment should increase the degree of separation in the rotor (due to increased centrifugal force) and should also result in movement of the aqueous-organic interface outward, away from the organic weir. Once again, a large volume of aqueous solution was found in a sample of the organic effluent. The system was found to recover when the rotor speed was reduced to 4000 rpm.

After it was determined that satisfactory phase separation was problematic at rotor speeds exceeding 4200 rpm, no subsequent testing was performed at rotor speeds greater than 4000 rpm. Additional tests under extraction conditions found that the rotor performed acceptably at 4000 rpm with an aqueous flow rate of 1000 mL/min and an organic flow rate of 310 mL/min. Because this aqueous flow rate represented the maximum throughput of the pump being used and both flows were more than twice those to be applied in mass transfer testing, no additional testing was performed under extraction conditions. In addition to the presence of organic effluent contamination at higher rotor speeds, accumulation of aqueous solution in the solvent feed line was also observed. This observation was made during contactor operation at speeds above 4200 rpm when both organic and aqueous solutions were being delivered to the unit and when only aqueous solution was being fed. It was suspected that momentum imparted on

aqueous solution entering the contactor causes a fraction of the stream to travel around the wall of housing with minimal vertical drop and that centrifugal force causes this fluid to exit the housing when the organic inlet is reached. Organic solution was not seen in the aqueous feed stream, probably because the lighter solvent phase would continually be purged by the flow of heavier aqueous solution in the upward-sloping feed line.

Results of throughput observations under extraction conditions are presented in Table 4.

### 3.1.2 Stripping-Condition Throughput Performance

Prior to stripping-mode throughput tests, the contactor was rinsed with deionized water and the system was flushed with strip solution (0.001 M HNO<sub>3</sub>). Initial attempts at throughput determination were made with a 0.950-in.-diam aqueous weir plate in place. Large amounts of organic contamination were found in aqueous effluent samples that were collected over a range of throughputs and at speeds ranging from 3000 to 4000 rpm. Since this failure mode does not typically define an operating boundary under high organic-to-aqueous phase ratios, it was suspected that the interface inside the rotor was located below the aqueous underflow. To move the interface inward (away from the underflow channels), the aqueous weir plate was replaced with one having a diameter of 0.875 in. Subsequently, good phase separation was achieved at flow rates of 700 mL/min organic and 140 mL/min aqueous over a rotor speed range of 2800 to 4000 rpm. Since the mass transfer test condition was less than one-fourth this total throughput, no additional hydraulic testing was performed under stripping conditions.

**Table 4. Results of contactor hydraulic testing under extraction conditions**

Rotor speed (rpm)	Nominal throughput (mL/min)
<2000	525
3500	650
3800	1050
4000	1300

## **3.2 MASS TRANSFER PERFORMANCE**

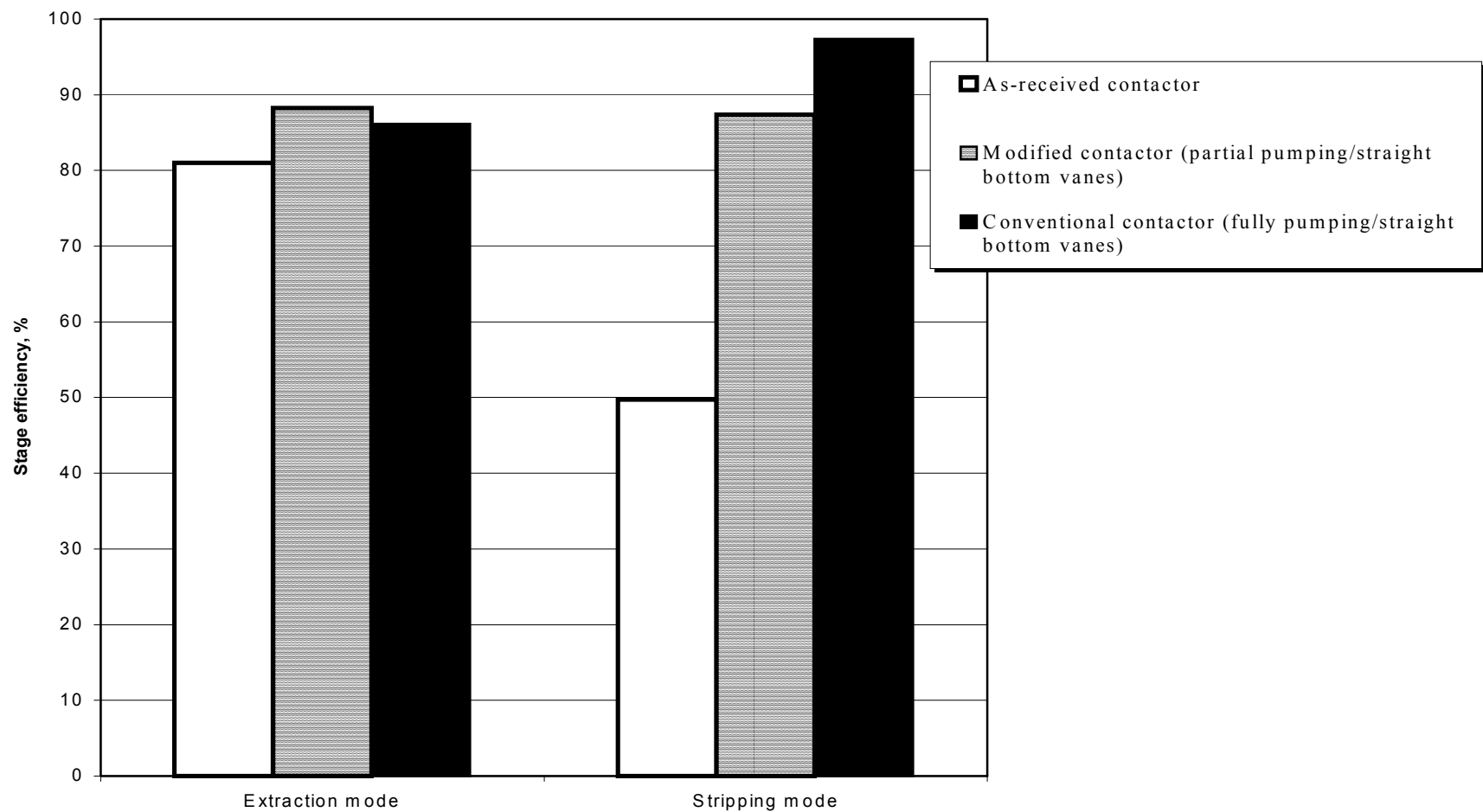
As described in the Introduction, observations made through a 1- by 2-in. port in the contactor housing during testing of an as-received contactor were of low liquid heights in the contactor mixing zone. Specifically, liquid levels were observed to reach only the lower edge of the observation window, which was located at an elevation just above the bottom of the rotor. Comparison of efficiency test results using this contactor and one that had been modified to improve mixing (Fig. 5) seems to confirm that mixing in the as-received contactor was insufficient to achieve mass transfer equilibrium. In the absence of visual observation, results of hydraulic testing described in the previous section indicate that mixing zone heights in the fully pumping 5-cm contactor using a straight-vane bottom housing are at desirable levels at speeds in the 3600- to 4000-rpm range. Consequently, stage efficiencies comparable with those determined from tests using a partially pumping rotor with a straight-vane housing bottom were expected.

### **3.2.1 Extraction-Mode Testing**

As in the previous mass transfer test,<sup>1</sup> contactor operation was initiated at 3600 rpm. Aqueous feed solution was introduced to the contactor at a rate of 484 mL/min. Solvent flow was initiated at a rate of 150 mL/min as soon as aqueous solution began exiting the contactor. Duplicate samples of both effluent streams were collected after 4 min of operation and again after 7 min of operation. Following collection of the second set of samples, the feed flows were stopped and the contactor was partially drained with the contactor rotor still turning. In this manner, the solution collected comes primarily from the contactor mixing zone. The temperature of the solution collected was measured and recorded. Test results are presented in Table 5.

Stage efficiencies calculated from test results indicate acceptable (>80%) mass transfer efficiency under the extraction condition. The lack of a significant difference between results from samples collected after 4 and 8 residence times indicates that steady state had been achieved at the time the first set of samples was collected. No cross-phase contamination was observed in either effluent during the extraction-mode test.





**Fig. 5. Comparison of stage efficiencies for as-received, modified, and conventional contactor configurations.**

**Table 5. Extraction and stripping mass transfer results**

Test condition <sup>a</sup>	Aqueous flow (mL/min)	Organic flow (mL/min)	Stage temperature (°C)	Organic effluent Cs conc. (ppm)	Aqueous effluent Cs conc. (ppm)	Equilibrated aqueous phase Cs conc. (ppm)	Aqueous effluent pH	Efficiency (%)	D <sub>Cs</sub> <sup>b</sup>
Extraction-1A	484	150	24.9	42.9	2.12	0.61	14	86.63	20.24
Extraction-1B	484	150	24.9	40.6	2.10	0.61	14	86.80	19.33
Extraction-2A	484	150	23.5	41.1	2.13	0.52	14	85.85	19.30
Extraction-2B	484	150	23.5	48.0	2.27	0.62	14	85.37	21.15
Stripping-1A	30	150	25.9	19.8	29.9	4.10	3	729.27 <sup>c</sup>	0.66
Stripping-1B	30	150	25.9	21.0	34.0	34.9	3	97.42	0.62
Stripping-2A <sup>d</sup>	30	150	25.3	36.9	21.4	1.87	10	1144.39	1.72
Stripping-2B <sup>d</sup>	30	150	25.3	36.3	25.7	16.1	8	159.63	1.41

<sup>a</sup>Identical numerical suffixes indicate duplicate samples collected to verify analytical precision.

<sup>b</sup>Calculated from concentrations of contactor effluent streams.

<sup>c</sup>Efficiency value is high due to suspect result from equilibrated effluents (see discussion in text).

<sup>d</sup>Samples were collected as the supply of extract (organic) was becoming depleted, resulting in unstable flow. Results from these samples are not valid for evaluating process performance.

Cesium distribution coefficients determined from test results are extremely high compared with standard values obtained in laboratory determinations using pristine baseline CSSX solvent. The reason for the abnormality is most likely a high extractant concentration, resulting from the evaporation of diluent from the solvent blend. Loss of diluent would also explain both the high density of the solvent and the low dispersion numbers that were determined in solvent formulation evaluations that were conducted after the mass transfer test.

### **3.3.2 Stripping-Mode Testing**

Prior to the stripping test, the extract generated in the extraction-mode test was scrubbed with 0.05 M HNO<sub>3</sub> using the contactor configuration that had been used in extraction testing. The scrubbing operation was performed with the contactor operating at 3600 rpm. The scrub solution flow rate was 30 mL/min, and the solvent flow rate was 150 mL/min.

Following scrubbing, the scrub solution in the aqueous feed tank was replaced with strip solution (0.001 M HNO<sub>3</sub>), and the vessel containing scrubbed extract was moved to the organic solution feed position. The contactor was dismantled and rinsed, and the contactor loop was flushed with strip solution.

After contactor operation was initiated at 3600 rpm, strip solution was started at a rate of 30 mL/min. Aqueous flow began to exit the contactor approximately 7 min after flow was initiated. Solvent flow was started at a rate of 150 mL/min. A set of effluent samples was collected approximately 4 min after starting organic-phase flow. Due to the amount of time required to obtain adequate aqueous sample volumes at the low aqueous flow rate, collection of the second set of samples began almost immediately after completing collection of the first set. As the second set was being collected, the flow of solvent became unstable because of depletion of the solvent inventory.

Test results are presented in Table 5. Values obtained from the second sample set (stripping-2A and -2B) are of dubious value due to loss of steady-state flow during sample collection. The instability of the system during collection of this sample set is

indicated by high and inconsistent pH values. The legitimacy of the pH results is supported by increased organic-phase cesium concentrations and high distribution coefficients, which reflect loss of stripping performance due to high counterion concentration.

Results from duplicate samples collected during the first collection period are consistent, with the exception of the aqueous-phase equilibrated sample results. It is suspected that the cesium result for the equilibrated stripping-1A organic sample is erroneous, based on results of material balance calculations. A material balance performed using analytical results from equilibration of the first aqueous and organic samples collected (stripping-1A) accounts for only 27% of the cesium present in the solutions that were equilibrated. A material balance using results from equilibration of the stripping-1B samples closes to within 11%.

#### **4. DISCUSSION AND CONCLUSIONS**

Acceptable phase separation was achieved under extraction and stripping conditions despite the use of a high-density solvent that displayed poor phase separation under CSSX flowsheet conditions (as indicated by low dispersion numbers). While the solvent condition necessitated adjustment of the aqueous weir size to obtain acceptable phase separation under stripping conditions, phase separation results should increase confidence in the ability of contactor-based extraction systems to respond to process upsets without loss of performance.

Mass transfer results obtained under the extraction condition indicate that the fully pumping 5-cm contactor, when operated with a straight-vane bottom housing, provides acceptable cesium transfer efficiency. Stage efficiencies obtained during extraction testing were consistently in the 85 to 87% range. Distribution coefficients were high, probably due to loss of diluent by evaporation. The solvent used in testing had been used repeatedly in various test procedures performed over a period of 13 months preceding the reported tests. The solvent had been stored in sealed containers

between uses but had been cycled through contactor test loops that were vented to a laboratory hood for a period of several hundred contactor residence times.

Results from the stripping-mode mass transfer test exhibit some inconsistencies, primarily in the cesium concentrations detected in equilibrated aqueous effluent samples. Inconsistencies in pH and cesium levels in duplicate samples collected near the end of the stripping test reflect loss of steady state due to depletion of the solvent inventory. It is believed that the other significant inconsistency, a low cesium indication in an equilibrated aqueous sample from the first set of stripping test samples, reflects an error in analysis. This conclusion is based on a mass balance analysis of the stripping test results. Based on sample results that are most consistent with material balance predictions and with distribution coefficients determined independently in a solvent quality analysis, acceptable mass transfer efficiency was obtained under stripping conditions using the fully pumping contactor with the straight-vane configuration.

Comparison of results with those reported previously for a fully pumping 5-cm contactor equipped with curved housing vanes indicates that the latter configuration adversely affects mass transfer. Comparison of the results presented in this report with those reported for a partially pumping rotor that was equipped with straight mixing vanes shows little difference in stage efficiencies.<sup>1</sup>

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