Caustic-Side Solvent Extraction Full-Scale Test - 8431

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ABSTRACT

A Full-Scale Test (FST) program was performed by Parsons and its team members General Atomics and Energy Solutions to assess the performance of full-scale centrifugal contactors specified for the Department of Energy Salt Waste Processing Facility (SWPF). The SWPF, to be located at the Savannah River Site (SRS) in Aiken, South Carolina, will remove highly radioactive waste constituents, principally actinides, strontium (Sr), and cesium (Cs) radionuclides, from salt waste solutions currently stored in SRS high-level waste tanks. Caustic-side Solvent Extraction (CSSX) removes Cs from waste feed that has been treated upstream to remove actinides and Sr. CSSX uses a custom solvent to extract Cs from the salt solution in a series of single stage centrifugal contactors. The test system comprised (a) eleven 25.4 cm (10") full-scale contactors (versus 36 in SWPF) for the extraction, scrub, strip, and wash stages; (b) two solvent recovery coalescers; and (c) the associated hardware and control system, packaged in four skid mounted modules. This paper describes the results of tests performed to define both hydraulic performance parameters (maximum hydraulic capacity and phase carryover) and solvent extraction performance parameters (Cs mass transfer efficiencies) using simulated SWPF waste and actual CSSX solvent. The test results confirmed key design features of the CSSX process and, as a consequence, the use of CSSX in the SWPF.

INTRODUCTION

The CSSX system at SWPF will be a continuous flow process utilizing 36 contactor stages for extraction, scrubbing, stripping, and washing of aqueous and organic streams. Cesium (Cs) is captured by contacting the Cs-bearing aqueous salt waste with a solvent (organic phase) in the extraction stage contactors at an organic to aqueous ratio (O:A) of 5:16. A simulated salt waste was used for the FST consisting primarily of sodium hydroxide, sodium nitrate, aluminum nitrate and sodium nitrite [1]. The solvent used in the CSSX process is primarily Isopar®L, with a specialty extractant (BOBCalixC6) a modifier (Cs-7SB), and a suppressant (tri-n-octylamine) [2]. The Cs-laden solvent exiting the extraction stage is scrubbed to remove Na and K impurities. The Cs is then stripped from the solvent. In the scrub and strip contactors, the O:A is 1:5. Both the scrub and strip phases utilize dilute nitric acid solutions. The stripped solvent is then washed with dilute caustic to remove any extracted impurities and solvent degradation products before it is recycled to the extraction system.

Each contactor stage mixes the aqueous and organic phases together, then separates them and discharges them separately. It is essential that each contactor stage both adequately mix and separate the solutions to ensure that the product streams meet waste acceptance criteria at downstream waste immobilization facilities (Defense Waste Processing Facility and Saltstone Production Facility). The separation of phases in centrifugal contactors is not absolute. A small amount of the aqueous phase remains entrained in the organic effluent, and a small amount of organic remains entrained in the aqueous effluent. Limiting the quantities of other-phase carryover is necessary to limit back-mixing effects on mass transfer performance and to insure that the chemistry in each section functions as intended.

Two primary objectives of the CSSX FST were to characterize other-phase carryover in contactor effluents and to determine the Cs mass transfer stage efficiencies. Aqueous and organic carryover in both, strip and extraction contactor effluents, were characterized as a functions of flow throughput, rotor speeds, weir selection and bottom plate type. Extraction and strip stage efficiencies were determined as functions of throughput and bottom plate type.

Operating conditions during the hydraulic and mass transfer testing are listed in Table I.

Table I. FST Operating Conditions

Parameter	Specification
Extraction Temperature	23°C ± 3°C
Strip Temperature	33°C ± 3°C
Extraction O:A	5:16
Scrub, Strip & Wash O:A	5:1
Ventilation Pressure	- 1 kPa

EQUIPMENT DESCRIPTION

The FST system was comprised of four extraction contactors, two scrub contactors, four strip contactors, one wash contactor, two coalescers, and fluid supply systems including pumps, tanks, heat exchangers, chiller, instrumentation and controls, and interconnecting piping and wiring to support continuous operation. The reduced number of extraction and stripping stages (as compared to the SWPF CSSX process) reduced the overall DF. The lower DF allowed measurements of system performance to be made using non-radioactive Cs, reducing the complexity and cost of the test program without compromising the test objectives. Figure 1 presents an overview of the CSSX FST system.

MEASUREMENT TECHNIQUES

Cs was measured in aqueous solutions via Enronmental Protection Agency method SW846 6020 by Inductively Coupled Plasma Mass Spectroscopy (ICPMS). The stage efficiencies determined during this test program are independent of Cs distribution coefficients (D_{Cs}) because the actual Cs equilibrium concentrations were measured for each stage. The efficiencies for each contactor stage were calculated based on the following equation:

$$E_i = \frac{(X_{i+1} - X_i)}{(X_{i+1} - X_{i,eq})} \qquad \text{(Eq. 1)}$$

Where Xi+1 is the concentration of the species in the aqueous phase entering stage i, Xi is the concentration of the species in the aqueous phase exiting stage i, and Xi,eq is the concentration of the species in the aqueous phase at equilibrium. Thus, the mass-transfer efficiency for stage i (Ei) is a measure of how close the Cs mass transfer in the system approaches equilibrium conditions established in the laboratory. The equilibrium concentrations (Xi,eq) were determined by contacting an organic and aqueous outlet sample from each stage in separatory funnels at the flowsheet temperatures and O:A's. The samples were first placed on an agitation table inside of an incubator and allowed to thoroughly mix at temperature. Next, the samples were allowed to settle and the aqueous phase was decanted. The decant was performed inside the incubator to ensure that the separation occurred at the process temperature.

Organic samples were drawn into tubes and centrifuged in the laboratory. Aqueous carryover in the organic samples was measured by visually comparing the centrifuged samples to a set of specially prepared standards.

Organic carry-over was determined by measurement of Cs-7SB in strip and extraction contactor aqueous effluents. The organic content in the aqueous samples was extracted with methylene chloride then concentrated to a smaller volume. The methylene chloride extract was then analyzed on an High Performance Liquid Chromatography/ UltraViolet/ Visible (HPLC/ UV/ VIS) system. The HPLC separates the individual components on a liquid (C18) column. The effluent flows through a UV/VIS detector to monitor for quantification of Cs-7SB.

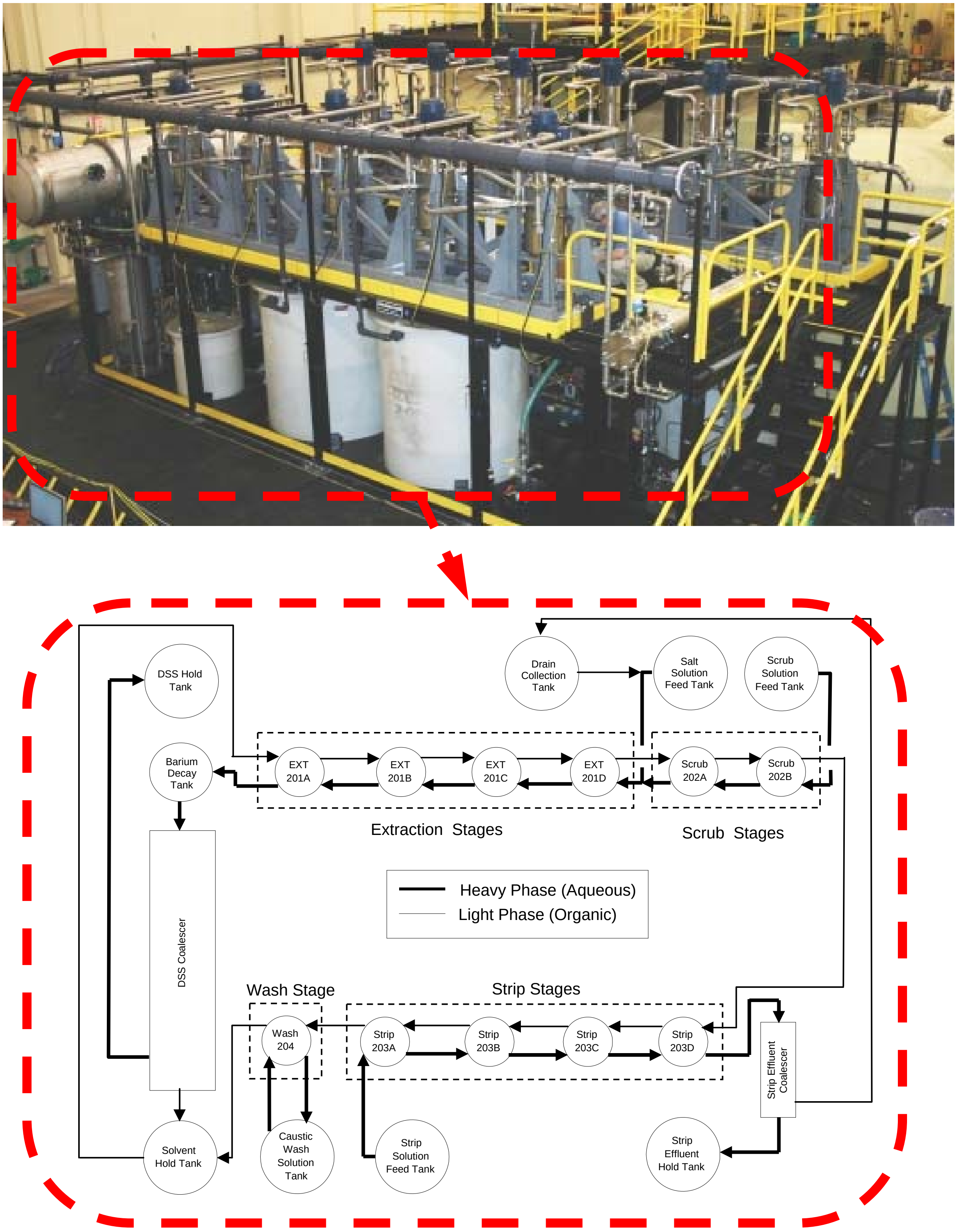

Fig. 1. FST overview with process flow detail

HYDRAULIC TESTING RESULTS

This test series was focused specifically on hydraulic performance irrespective of Cs mass transfer. Feeds used had similar physical properties (density, viscosity, surface tension) to actual waste in order to emulate the hydraulic performance in SWPF. The methodology employed during this testing series was to evaluate the hydraulic performance trends (as a function of bottom plate type, weir size, throughput, and rotor speed) within an operational envelope that provided acceptable carryover.

The limits of acceptable carryover were not explicitly defined for this test. Based on the mass balance model predictions, mass transfer performance in CSSX is not appreciably impacted until organic carryover is in excess of 1%. SWPF will include stilling tanks and coalescers on the aqueous effluents from strip and extraction to limit organic carryover to downstream processes. Thus, overall CSSX performance and effluent quality is not expected to be significantly impacted by organic carryover that is less than 1% in aqueous contactor effluents. An aqueous carryover limit from extraction was established at 0.2%. The metric of 0.2% aqueous carryover from EXT-201D was intended to eliminate neutralization of the scrub solution in EXT-202A which can cause the formation of precipitants and/or limit the solvent scrubbing effectiveness. Thus, the acceptable carryover envelope was defined as (1) organic carryover less than 1%, (2) aqueous carryover in the extraction organic effluent less than 0.2%, (3) no gross aqueous carryover from any contactor.

Off-skid simulant feed, effluent and waste tanks were not used during hydraulic testing. Scrub and waste simulant feeds were re-circulated directly from on-skid tanks. Normally, the scrub effluent is combined with the waste simulant and fed through extraction. A premixed scrub-diluted waste-simulant was utilized to ensure that the feed through extraction had prototypic physical properties. The scrub-diluted simulant waste was re-circulated to and from the Decontaminated Salt Solution Hold Tank. Strip solution was also re-circulated since mass transfer was not being evaluated.

Flow limitations due to foaming at the scrub contactor organic vent line prevented the system from achieving 100% flow during hydraulic testing. As a result, 85% flow was the highest attainable throughput during this phase of testing. Fluid flows at 100% and 85% of maximum flow are shown in Table II. The throughput at 100% flow equates to slightly more than 35.6×10^6 L/yr (9.4 Mgal/yr) of waste processed in SWPF which is anticipated to be the peak plant throughput.

Table II. Throughput at 100% and 85% of Maximum

Fluid	100% Flow (Lpm)	85% Flow (Lpm)
Simulant Waste Feed	81.12	68.95
Solvent Feed	27.04	22.98
Scrub Feed	5.41	4.6
Strip Feed	5.41	4.6
Wash Feed	5.41	4.6

Process fluids were monitored for property changes throughout hydraulic testing. Of the properties monitored, the scrub-diluted simulant, solvent, and strip densities were monitored most closely. The pH levels of the process fluids were observed but not frequently adjusted during the course hydraulic testing. The process fluids included scrub-diluted waste simulant, solvent, strip, scrub, and wash solutions. For each set of conditions tested, the system was allowed to reach steady-state as indicated by stable temperatures, flow rates and pressures (Table I). Samples were drawn after a minimum of twenty minutes of steady-state operation.

The results from the analytical method used to measure Cs-7SB report the total concentration, which includes both the partitioning (organic concentration miscible in the aqueous phase) concentration and the carryover (organic concentration immiscible in the aqueous phase) concentration. A study was performed to determine the Cs-7SB partitioning concentrations in both strip and scrub-diluted waste simulant solutions. Aqueous samples were drawn from the strip and extraction effluents and allowed to settle overnight in separatory funnels at room temperature. The lower, aqueous, portion of these samples was decanted and analyzed. The mean Cs-7SB partitioning concentrations were 5.5 mg/L and 33.1 mg/L in extraction and strip samples, respectively. It is likely that all samples taken during hydraulic testing were completely saturated with solvent due to the relatively small volumes of strip and scrub-diluted simulant being re-circulated for this test series.

The concentrations of immiscible (carryover) Cs-7SB in the aqueous samples were calculated by subtracting the mean partitioning concentrations from the laboratory measured concentrations. The immiscible Cs-7SB concentrations were used to calculate the equivalent Isopar®L carryover, which is the basis for the SWPF Waste Acceptance Criteria (WAC) limit. The Isopar®L carryover results have been reported in units of mg/L and mg/kg in simulant and strip solutions, respectively.

Due to large number of subtests involved in this testing series it was advantageous to use as few contactors as possible to conduct the testing. Minimizing the number of contactors that required weir changes substantially reduced the testing scope. Individual contactor tests were not feasible due to the system design (welded hard pipe etc). These tests were conducted by changing weirs in only two adjacent contactors in the extraction and stripping sections even though the feeds were processed through all eleven contactor stages. Weir changes were made in contactors A and B, while nominally sized weirs were left in C and D. Aqueous samples were drawn from the effluent of contactor A and organic samples were drawn from the effluent of contactor B. In this way, both the organic stream and the aqueous stream passed through two contactors with identical weirs prior to being sampled.

All samples were taken in duplicate. Data sets collected using weirs that resulted in excessive gross organic and/or aqueous carryover were not included in the data analyses. Eight aqueous carryover data points were excluded from the data set obtained with weirs that gave acceptable performance. These were omitted because aqueous carryover in duplicate samples differed substantially (by more than 0.2 volume percent aqueous carryover in organic). The difference between duplicates was likely due to inadequate purging during sampling.

The large number of variables involved in hydraulic testing required the variables be separated, to the extent possible, in order to evaluate the results. Extraction and strip results were considered independent and were evaluated separately. Aqueous-carryover in organic samples and organic-carryover in aqueous samples were the two dependent measured variables. Each of the two dependents had four independently controlled variables: bottom plate type, heavy-phase weir inner-diameter, flow rate, and contactor rotor speed. The number of independent variables was reduced to three by evaluating curved-vane results separately from straight-vane results. The curved and straight-vane bottom plates were compared in separate analyses as a function of aqueous and organic carryover, for both extraction and strip sections. The end result was 28 separate analyses with a maximum of three independent variables interacting simultaneously. Table III presents the break down of the variables and the number separate analyses performed to evaluate the hydraulic testing data set.

Due to the complexity of the hydraulic testing data set, the discussion of the results relies heavily on rigorous statistical methods. Multiple regression least squares analyses were used to generate leverage plots for a single dependent variable (e.g. aqueous carryover in the strip section with straight-vanes) as a function of multiple independent variables (weir size, flow rate, and rotor speed).

Table III. Hydraulic Testing Multiple Variable Analyses Summary

<table>
<tr><th colspan="2">Independent Discrete Effects</th><th>Independent Variables</th><th>Dependent Variables</th></tr>
<tr><td rowspan="14">Extraction Section</td><td rowspan="6">Straight-Vane</td><td rowspan="3">Aqueous CO</td><td>Weir Size</td></tr>
<tr><td>Flow Rate</td></tr>
<tr><td>Rotor Speed</td></tr>
<tr><td rowspan="3">Organic CO</td><td>Weir Size</td></tr>
<tr><td>Flow Rate</td></tr>
<tr><td>Rotor Speed</td></tr>
<tr><td rowspan="6">Curved-Vane</td><td rowspan="3">Aqueous CO</td><td>Weir Size</td></tr>
<tr><td>Flow Rate</td></tr>
<tr><td>Rotor Speed</td></tr>
<tr><td rowspan="3">Organic CO</td><td>Weir Size</td></tr>
<tr><td>Flow Rate</td></tr>
<tr><td>Rotor Speed</td></tr>
<tr><td>N/A</td><td>Aqueous CO</td><td>Bottom Plate</td></tr>
<tr><td>N/A</td><td>Organic CO</td><td>Bottom Plate</td></tr>
<tr><td rowspan="14">Strip Section</td><td rowspan="6">Straight-Vane</td><td rowspan="3">Aqueous CO</td><td>Weir Size</td></tr>
<tr><td>Flow Rate</td></tr>
<tr><td>Rotor Speed</td></tr>
<tr><td rowspan="3">Organic CO</td><td>Weir Size</td></tr>
<tr><td>Flow Rate</td></tr>
<tr><td>Rotor Speed</td></tr>
<tr><td rowspan="6">Curved-Vane</td><td rowspan="3">Aqueous CO</td><td>Weir Size</td></tr>
<tr><td>Flow Rate</td></tr>
<tr><td>Rotor Speed</td></tr>
<tr><td rowspan="3">Organic CO</td><td>Weir Size</td></tr>
<tr><td>Flow Rate</td></tr>
<tr><td>Rotor Speed</td></tr>
<tr><td>N/A</td><td>Aqueous CO</td><td>Bottom Plate</td></tr>
<tr><td>N/A</td><td>Organic CO</td><td>Bottom Plate</td></tr>
</table>

Extraction Hydraulic Testing Results

The tests were conducted using contactor speeds from 1200 rpm to 2700 rpm, fluid throughput from 44% to 85% of maximum, weir sizes from 12.95 cm (5.1”) to 13.97 cm (5.5”) with curved-vane bottom plates, and 13.46 cm (5.3”) to 13.97 (5.5”) with straight-vane bottom plates.

The carryover trends observed during extraction testing with curved-vane bottom plates are:

- There was a significant trend of increasing aqueous carryover with increasing weir size. There is an insignificant difference between the organic carryover with differing weir sizes.
- There was a significant trend of increasing aqueous and organic carryover with increasing flow.
- There was a significant trend of decreasing aqueous carryover with increased contactor rotor speeds for aqueous carryover. Rotor speed did not have a significant effect on organic carryover.

The carryover trends observed during extraction testing with straight-vane bottom plates are:

- There was no significant difference in aqueous or organic carryover between the weirs tested (13.46 cm, 13.72 cm, and 13.97 cm);
- There was a significant trend of increasing aqueous and organic carryover with increasing flow;

- Aqueous and organic carryover showed a significant decreasing trend with increasing rotor speeds.

Comparing hydraulic performance between curved and straight-vane bottom plates in extraction:

- Curved-vane bottom plates exhibited significantly less aqueous and organic carryover compared to straight-vane bottom plates over the range of independent variables tested.

Table IV summarizes the extraction aqueous and organic carryover trends as functions of weir size, flow rate, rotor speed, and bottom plate type.

Table IV. Summary of Extraction Hydraulic Performance Trends

Independently Controlled Variables		Dependent Variables	Independent Variables	Significant or Not Significant at 95% Confidence?	Trend[a]
Extraction	Curved-Vane	Aqueous CO	Weir Size	Significant	Increase
			Flow Rate	Significant	Increase
			Rotor Speed	Significant	Decrease
		Organic CO	Weir Size	Not Significant	N/A
			Flow Rate	Significant	Increase
			Rotor Speed	Not Significant	N/A
	Straight-Vane	Aqueous CO	Weir Size	Not Significant	N/A
			Flow Rate	Significant	Increase
			Rotor Speed	Significant	Decrease
		Organic CO	Weir Size	Not Significant	N/A
			Flow Rate	Significant	Increase
			Rotor Speed	Significant	Decrease
	Bottom Plate Comparison	Aqueous CO	Bottom Plate	Significant	CV Lower
		Organic CO	Bottom Plate	Significant	CV Lower

[a]Trend refers to the effect on the dependent variable when the independent variable is increased.

There was a significant trend of increasing carryover (aqueous and organic) with increasing extraction throughput over the full independent variable ranges tested. Increasing rotor speed tended to decrease carryover for all cases except for organic carryover with curved-vane bottom plates. Weir size only had a significant effect on aqueous carryover with curved-vane bottom plates over the test range.

Table V shows a summary of the mean carryover concentrations during extraction hydraulic testing. The mean aqueous and organic carryover is presented in two forms. The first is the mean carryover, for both straight and curved-vane bottom plates, over the entire test range (all weirs, speeds and flows). Secondly, the means are shown for a constrained test range. The range constraints correspond to the high flow regime and to the weir size and rotor speed (independent variables) trends summarized in Table IV. The high flow regime was selected because SWPF is expected to primarily operate at the highest flows that allow acceptable mass transfer and hydraulic performance. The ranges of weir sizes and rotor speeds were constrained to conditions that tended to improve hydraulic performance. If an independent variable did not have a significant effect on carryover, then the mean was calculated over the entire range of that variable.

Table V. Extraction Hydraulic Testing Carryover Summary

Bottom Plate Type	Flow(s) {% of max.}	Rotor Speed(s) {rpm}	Weir Size(s) {cm}	Mean Aq. Carryover {Vol%}	Mean Isopar®L Carryover {mg/L}	Process Conditions
Curved-Vane	44 - 85	1200 - 2700	12.95, 13.46, 13.97	0.18	693	All conditions
	≥ 79	≥ 2100	5.1	0.11	-	High flows and high rotor speeds
	≥ 79	All	All	-	963	High flows
Straight-Vane	44 - 85	1200 - 2700	13.46, 13.72, 13.97	0.36	1009	All conditions
	≥ 79	≥ 2100	All	0.16	-	High flows and high rotor speeds
	≥ 79	≥ 2100	All	-	1285	High flows and high rotor speeds

Figure 2 shows graphical representations of the aqueous and organic carryover results, respectively. Each flowchart is a data tree, where each branch represents a split in the data set. The splits correspond to the constraints used to determine the mean carryover concentrations in Table V. Each data box contains the number of data points, mean carryover, and the standard deviation for the constrained data set.

Strip Hydraulic Testing Results

Strip hydraulic testing was conducted with rotor speeds ranging from 1200 rpm to 2700 rpm, throughputs ranging between 44% and 85% of maximum flow, 11.43 cm (4.5”) and 11.94 cm (4.7”) weir sizes with curved-vane bottom plates, and 11.43 cm (4.5”) to 12.45 cm (4.9”) with straight-vane bottom plates.

The carryover trends observed during strip testing with curved-vane bottom plates are:

- Aqueous carryover was significantly less with 11.94 cm weirs as compared to 11.43 cm weirs. There is an insignificant difference between the organic carryover with differing weir sizes.
- There was a significant trend of increasing aqueous carryover with increasing throughput. Flow did not have a significant effect on organic carryover
- There was a significant trend of decreasing aqueous carryover with increasing contactor rotor speeds. Organic carryover showed a significant increasing trend with increasing rotor speeds.

The carryover trends observed during strip testing with straight-vane bottom plates are:

- Aqueous carryover with 4.9” weirs was significantly less compared to the 11.43 cm and 11.94 cm weirs. Weir size did not have a significant effect on organic carryover.
- There was a significant trend of increasing aqueous carryover with increasing throughput. Flow rate did not have a significant effect on organic carryover.
- There was a significant trend of decreasing aqueous carryover with increasing contactor rotor speeds. Organic carryover showed a significant increasing trend with increasing rotor speeds.

Comparing hydraulic performance between curved and straight-vane bottom plates in strip:

- There was no significant difference in hydraulic performance (aqueous and organic carryover) between curved or straight-vane bottom plates in strip.

Table VI summarizes the strip aqueous and organic carryover trends as functions of weir size, flow rate, rotor speed, and bottom plate type.

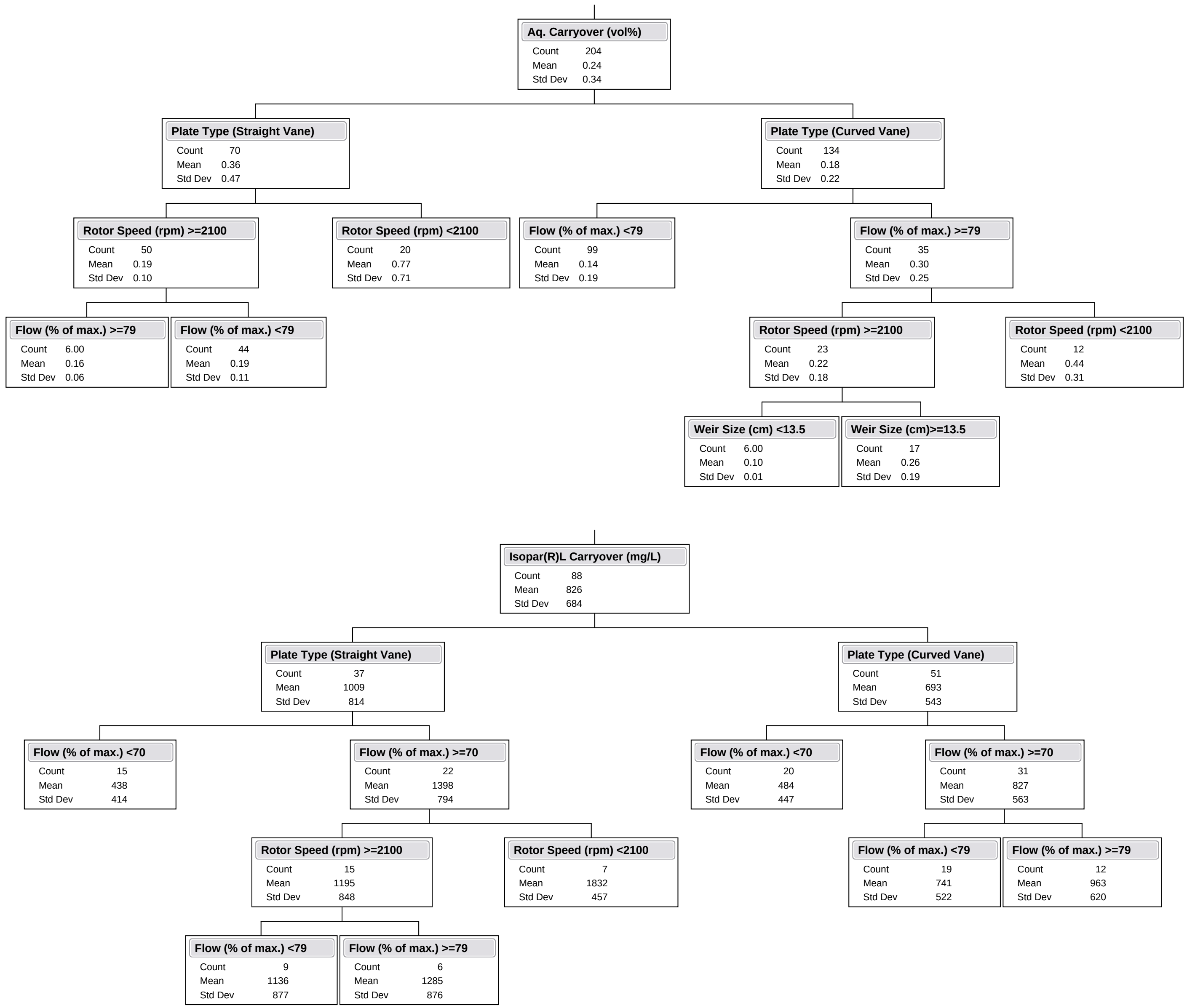

Fig. 2. Aqueous and Isopar®L Carryover Data Trees for Extraction Hydraulic Testing

Table VI. Summary of Strip Hydraulic Performance Trends

Independently Controlled Variables		Dependent Variables	Independent Variables	Significant or Not Significant at 95% Confidence?	Trend[a]
Strip Section	Curved-Vane	Aqueous CO	Weir Size	Significant	11.94 cm < 11.43 cm
			Flow Rate	Significant	Increase
			Rotor Speed	Significant	Decrease
		Organic CO	Weir Size	Not Significant	N/A
			Flow Rate	Not Significant	N/A
			Rotor Speed	Significant	Increase
	Straight-Vane	Aqueous CO	Weir Size	Significant	12.45 cm < 11.94 cm & 11.43 cm
			Flow Rate	Significant	Increase
			Rotor Speed	Significant	Decrease
		Organic CO	Weir Size	Not Significant	N/A
			Flow Rate	Not Significant	N/A
			Rotor Speed	Significant	Increase
	Bottom Plate Comparison	Aqueous CO	Bottom Plate	Not Significant	N/A
		Organic CO	Bottom Plate	Not Significant	N/A

[a]Trend refers to the effect on the dependent variable when the independent variable is increased.

Testing with curved and straight-vane bottom plates showed the same trends in aqueous and organic carryover as a function of weir size, flow rate and rotor speed. Larger weir sizes showed superior aqueous carryover performance. Increasing throughput significantly increased aqueous carryover. Increasing rotor speeds tended to reduce aqueous carryover. Rotor speed was the only independent variable that a significant effect on organic carryover with either bottom-plate type. Organic carryover tended to increase with increasing rotor speeds. Note that this is the opposite effect that rotor speed had on aqueous carryover.

Table VII shows a summary of the mean carryover concentrations during extraction hydraulic testing. The mean aqueous and organic carryover is presented in two forms. The first is the mean carryover over the entire test range (all weirs, speeds, flows, and bottom plate types). Secondly, the means are shown for a constrained test range. The range constraints correspond to the high flow regime and to the weir size and rotor speed (independent variables) trends summarized in Table VI.

The data is presented irrespective of bottom plate type because there was no significant performance difference between bottom-plate types. The high flow regime was selected because the plant is expected to primarily operate at the highest flows that allow acceptable mass transfer and hydraulic performance. If an independent variable did not have a significant effect on carryover then the mean was calculated over the entire range of that variable. Rotor speed was the only variable that had a significant effect on organic carryover. However, rotor speed had opposing effects on aqueous and organic carryover. Increasing it tended to decrease aqueous carryover while simultaneously increasing organic carryover and vice versa. Thus, rotor speed was constrained to an intermediate range (1500 – 2100 rpm) as a compromise between aqueous and organic carryover.

Table VII. Strip Hydraulic Testing Carryover Summary

Flow(s) {% of max.}	Rotor Speeds {rpm}	Weir Sizes {cm}	Mean Aq. Carryover {Vol%}	Mean Isopar®L Carryover {mg/kg}	Process Conditions
44 - 85	1200 - 2700	11.43, 11.94, 12.45	0.32	58	All conditions
≥ 79	1500 - 2100	≥ 4.7	0.38	-	High flows, large weirs, and intermediate rotor speeds
≥ 79	1500 - 2100	All	-	22	High flows and intermediate rotor speeds

Figure 3 shows graphical representations of the aqueous and organic carryover results. Each figure is a data tree, where each branch represents a split in the data set. The splits correspond to the constraints used to determine the mean carryover concentrations in Table VIII. Each data box contains the number of data points, mean carryover, and the standard deviation for the constrained data set.

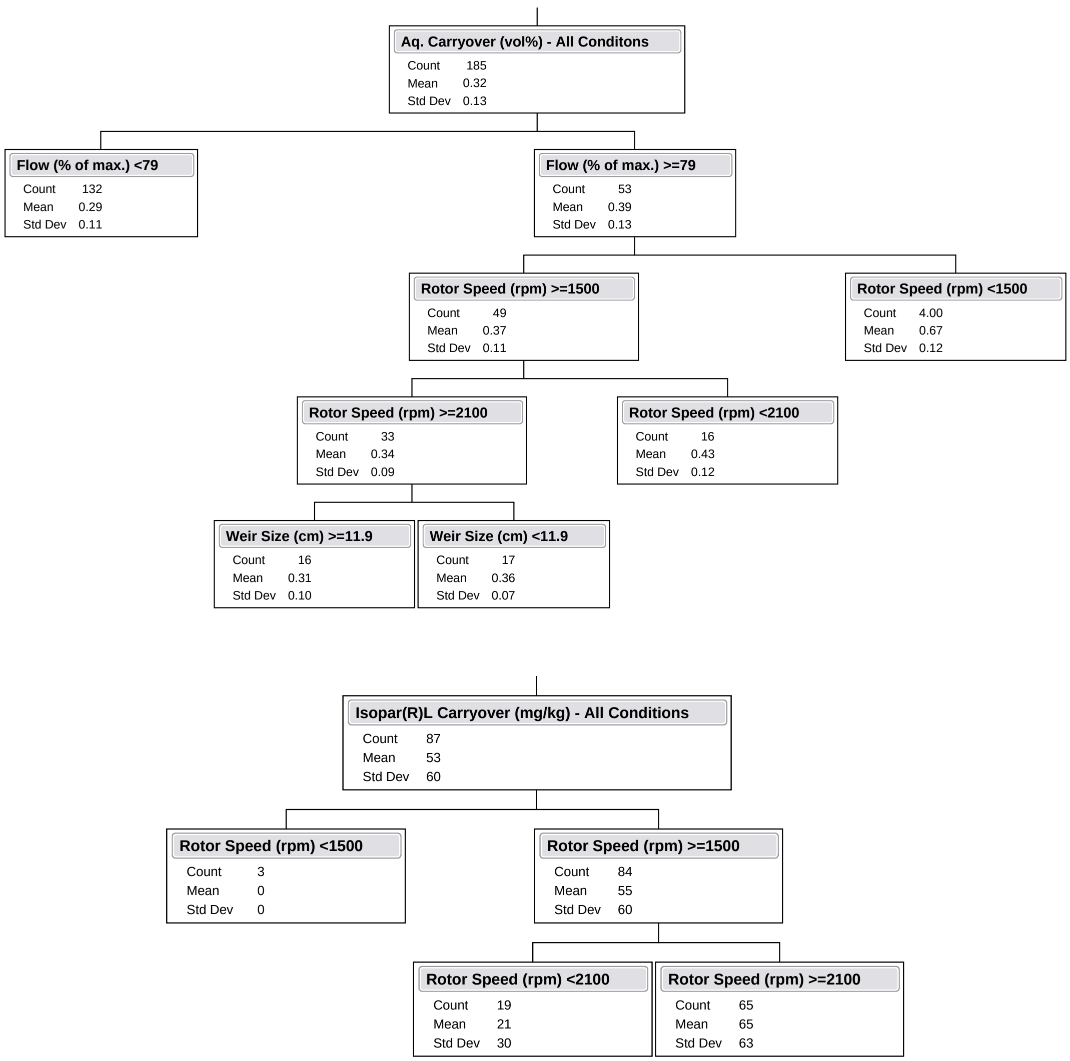

Fig. 3. Aqueous and Isopar®L carryover data trees for strip hydraulic testing

MASS TRANSFER TESTING RESULTS

The mass transfer test series characterized contactor stage efficiencies at 100%, 90%, 75% and 50% of maximum flow with both straight and curved-vane bottom plates. Operating conditions during the performance test series were intended to be prototypic of SWPF CSSX operations. The conditions specified for testing are shown in Table I. The original temperature tolerance specified was ±3°C of nominal in extraction and strip; actual temperature tolerances achieved were ±1°C in extraction and ±2°C in strip. For each test, the system was allowed to reach steady-state as indicated by stable temperatures, flow rates and pressure. Samples were drawn after a minimum of twenty minutes of steady-state operation.

Prior to the start of Performance testing the CSSX FST system total throughput was limited to 85% of maximum. The EXT-202B organic outlet piping was successfully modified prior to performance testing and 100% throughput was realized.

Isopar®L concentrations in the CSSX solvent were maintained by monitoring the solvent density as part of the normal start-up procedure. The solvent flow rate and density was measured using the inline Coriolis mass flow meter (LT-211). The solvent density was controlled to 0.851±0.01 g/cc throughout the test program by monitoring and adding Isopar®L as necessary.

The potassium concentrations in the waste simulant were measured for each of the Performance tests conducted. The average K concentration during Performance testing was 42 mM, which is considerably higher compared to the nominal concentration of 15mM. The high potassium concentrations in the simulant were expected to decrease the D_{Cs} because K and Cs are co-extracted by the CSSX solvent. K is extracted on the order of 100 times less strongly than Cs. However, K extraction can compete at a significant level by virtue of the nominally higher concentrations of K present in the waste, compared to that of Cs (~42mM K compared to ~0.44mM Cs). The high K concentrations present in the simulant should have decreased the CSSX-solvent D_{Cs} by approximately 30% [3].

The stage efficiencies determined during this test program are independent of D_{Cs} because the actual Cs equilibrium concentrations were measured for each stage. As previously mentioned, the K concentration in the simulant has a direct effect on D_{Cs}. However, the high K concentration in the simulant does not impact the contactor efficiency results presented herein because the stage efficiencies are independent of D_{Cs}.

The evaluation of mass transfer results relied heavily on statistical methods. In the case of stage efficiency calculations, where the measured values are combined and divided, the measurement errors propagate through the calculations to produce uncertainty in the final answer. For purposes of this discussion all Cs measurement errors are assumed to be random. The experimental uncertainties in the stage efficiencies, discussed herein, are assessed by statistical analysis based on repeated measurements. In most cases, stage efficiencies were calculated using the mean result of up to 3 replicated analyses and/or independent duplicate analyses for each sample point. There were five sample results that were excluded from the stage efficiency analysis. In each of these five cases there were independent duplicate equilibrium samples which had considerably different results. For conservatism, the equilibrium sample which resulted in the highest stage efficiency was excluded.

The stage efficiency results for the performance tests are provided in Table VIII. Stage efficiencies above 100% are theoretically impossible. However, measurement uncertainties in the stage efficiencies will result in some calculated results being higher and some lower than the actual stage efficiency. Averaging several measured values of efficiency, the high and low errors will tend to cancel each other out. For this reason several stage efficiencies above 100% have been retained (see extraction, straight vane, 90% flow, EXT-201A and -201D in Table VIII). There were 3 stage efficiency results above 120% which were excluded from subsequent analysis because they were considered unreasonably high. Note that the high efficiency results (shown shaded in Table VIII) all occurred in EXT-203A. It is likely that the high results are due to the presence of a systematic error. One plausible source systematic error could be that the equilibrium conditions used in the laboratory do not sufficiently emulate the conditions present in EXT-203A during these particular tests. The temperature, volume ratios, and/or the presence of a third phase could change the equilibrium distribution of Cs.

Table VIII. Performance Test Stage Efficiency Summary

		EXTRACTION					STRIP				
	FLOW	EXT-201A	EXT-201B	EXT-201C	EXT-201D	Mean	EXT-203A	EXT-203B	EXT-203C	EXT-203D	Mean
Straight Vane	50%	97.2%	95.2%	77.6%	79.7%	87.4%	124.8 %	78.4%	75.9%	79.0%	77.8%
	75%	73.8%	70.3%	84.8%	91.2%	80.1%	79.2%	69.9%	93.3%	99.2%	85.4%
	90%	105.3 %	93.4%	89.1%	100.6 %	97.1%	146.4 %	71.1%	92.3%	91.0%	84.8%
	100%	82.8%	89.5%	87.6%	93.0%	88.2%	64.1%	83.8%	96.4%	83.2%	81.9%
Curved Vane	50%	87.6%	89.2%	85.0%	95.2%	89.2%	44.5%	61.6%	51.9%	51.1%	52.3%
	75%	82.2%	84.4%	87.1%	93.5%	86.8%	62.7%	61.7%	73.3%	58.6%	64.1%
	90%	77.0%	78.6%	77.2%	93.7%	81.6%	84.1%	60.2%	48.7%	60.6%	63.4%
	100%	80.7%	88.6%	84.0%	85.8%	84.8%	124.9 %	81.0%	60.5%	96.5%	79.3%

The relation of the CSSX FST mass transfer performance to the SWPF CSSX mass transfer performance is depicted in Figure 4. The mean extraction efficiencies are plotted against the mean stripping efficiencies at each flow, for both straight and curved-vane bottom plates. The error bars depict the mean efficiency uncertainty as the standard deviation of the means. The solid curves show the mass balance model's prediction of the combinations of strip and extraction efficiencies that result in an overall Cs decontamination factor (DF) of 40,000. Each curve represents the prediction at differing CSSX-solvent extraction D_{Cs}. Data points on or above a D_{Cs} curve represent acceptable SWPF CSSX performance. At extraction D_{Cs} as high as 14, extraction efficiencies at or below 65% result in unacceptable overall CSSX performance regardless of stripping performance. Similarly, stripping efficiencies at or below 70% are expected to yield unacceptably low SWPF DFs regardless of extraction performance. The straight-vane efficiencies should support operation at or above target DF levels for D_{Cs} values as low as 10 for all flows tested. Curved-vane efficiencies supported operation at or above target DF levels only for 100% flow.

The following trends were observed during mass transfer testing at a 95% level:

- Efficiency results with curved-vanes in extraction and straight-vanes in strip were flow independent from 50% to 100% of maximum throughput.
- Stripping efficiency tends to improve with increasing flow with curved-vanes.
- Extraction efficiency with straight-vanes was not significantly influenced by flow in the 50% to 100% throughput regime.
- There are no significant differences between extraction performances with either straight or curved-vane bottom plates.
- Stripping performance was significantly better with straight-vanes compared to curved-vanes.
- Utilizing straight-vanes in the stripping section, the overall SWPF CSSX performance is expected to meet or exceed the target DF of 40,000 with minimum extraction D_{Cs} of 10.

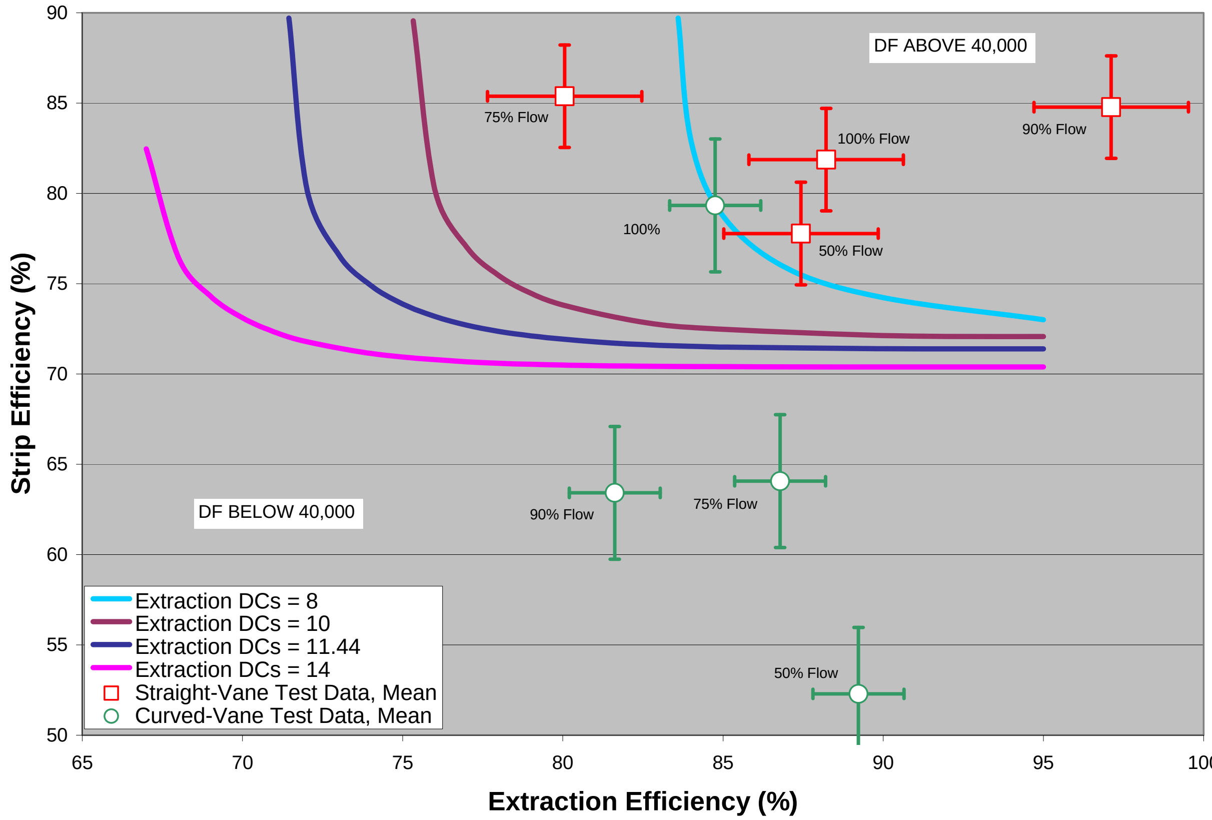

Fig. 4. CSSX FST mass transfer performance comparison to expected SWPF CSSX DF of 40,000

CONCLUSION

Total throughput was initially limited to 85% of maximum flow during FST. Minor system modifications performed prior to mass transfer testing series resulted in the realization of 100% throughput. The 100% flow equates to slightly more than 35.6 x 10^6 L/yr (9.4 Mgal/yr) of waste processed in SWPF which is anticipated to be the peak plant throughput.

To achieve the best hydraulic performance in extraction, it is recommended that the extraction contactors be operated at the highest reasonable speed possible (>2100 rpm). Vibration, hardware limitations, bearing life, and other factors should be considered prior to final selection of extraction contactor speeds in SWPF. In strip (also scrub and wash) aqueous carryover decreased and organic carryover increased as the rotor speeds increased. It is recommended that the strip, scrub, and wash contactors be operated at intermediate speeds (between 1500 and 2100 rpm) to achieve a performance compromise between aqueous and organic carryover.

Curved-vane bottom plates showed a significant hydraulic performance (aqueous and organic carryover) advantage over straight-vane bottom plates in extraction. There was no significant mass transfer performance advantage for either plate type in extraction. Thus, curved-vane bottom plates in extraction may be the better option for use in SWPF. There was no significant hydraulic performance difference between the plate types in strip. Straight-vanes provided significantly better mass transfer performance in strip compared to curved-vanes. Based solely on mass transfer performance, straight-vane bottom plates in the strip, scrub, and wash contactors are recommended for use in SWPF. Utilizing straight-vanes in the stripping section, the overall SWPF CSSX performance is expected to meet or exceed the target DF of 40,000 with minimum extraction D_{Cs} of 10.

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