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**CAUSTIC-SIDE SOLVENT EXTRACTION FLOWSHEET FOR
OPTIMIZED SOLVENT**

by

R. A. Leonard

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ABSTRACT

Using cesium distribution ratio data from Oak Ridge National Laboratory (ORNL) on the candidates for the optimized solvent, calculations were made to determine how each solvent would perform in the caustic-side solvent extraction (CSSX) process. This report describes the effect that each solvent would have on the CSSX flowsheet for both the current solvent flow rate and the optimum solvent flow rate.

I. INTRODUCTION

The Savannah River Site (SRS) has 34 million gallons of high-level waste in 48 tanks that need to be decontaminated [LEVENSON-2000]. As a part of this process, the cesium will be removed from waste solutions containing both supernatant liquid and dissolved salt cake, then vitrified for disposal. After the cesium is removed, the decontaminated solution will be immobilized in low-level grout.

Work performed at Argonne National Laboratory (ANL) in FY2000 and FY2001 showed that cesium can be extracted from caustic aqueous solutions representative of the high-level waste at SRS using solvent extraction carried out in centrifugal contactors [LEONARD-2000, -2001]. In those proof-of-concept tests, the CSSX process achieved both key process goals: (1) the cesium was removed from the waste with decontamination factors greater than 40,000 and (2) the recovered cesium was concentrated by a factor of 15 in dilute nitric acid. In the final test, the solvent was recycled 42 times as the process was run around the clock for three days. In addition, a CSSX flowsheet test was run with real waste at the Savannah River Technical Center (SRTC). This two-day test also achieved both key goals [CAMPBELL-2001]. Based on the success of these tests, solvent extraction was chosen as the preferred technology to use in the SRS waste facility for the removal of cesium.

The baseline CSSX solvent for these flowsheet tests was the improved solvent developed at Oak Ridge National Laboratory (ORNL) in FY1999 [BONNESEN-2000]. It has four

components: (1) an extractant—a calixarene crown, calix[4]arene-bis(*tert*-octylbenzo-crown-6) designated BOBCalixC6 or Calix; (2) a modifier—an alkyl aryl polyether, 1-(2,2,3,3,-tetrafluoropropoxy)-3-(4-*sec*-butylphenoxy)-2-propanol, also called Cs-7SB; (3) a suppressant—an alkyl amine, trioctylamine (TOA), which suppresses impurity effects to ensure that the Cs can be stripped from the solvent; and (4) a diluent—a mixture of branched hydrocarbons, Isopar[®]L. The composition of the baseline CSSX solvent is 0.01 M BOBCalixC6, 0.50 M Cs-7SB, and 0.001 M TOA in Isopar[®]L.

Work done at ORNL in FY2001 indicates that, with respect to the BOBCalixC6 solubility, the baseline solvent is somewhat supersaturated [MOYER-2001]. This supersaturation of the solvent has not been a problem since no precipitation was observed in any of the BOBCalixC6 in the CSSX tests over the period of a year. To ensure that this is not a problem in the future, work is being done at ORNL to improve the solvent composition so that the BOBCalixC6 is no longer supersaturated. These improvements involve reducing the BOBCalixC6 concentration in the solvent while increasing the Cs-7SB concentration. In addition, the TOA concentration was increased in some solvent compositions as this gives additional protection from the effect of process impurities. The properties of these new solvents, including the Cs distribution ratios, have been measured at ORNL. Using the distribution ratios, the effect on the CSSX flowsheet was calculated at ANL and the results are reported here.

II. FLOWSHEET CALCULATIONS

The Cs distribution ratios (D_{Cs} or D) for the various CSSX solvent compositions (see Table 1) were measured at ORNL [DELMAU-2001]. These batch distribution ratios were obtained at 25°C with an O/A volume ratio of 1/3 for the extraction test and 5/1 for the two scrub tests and the four strip tests. Note that an identifying code for each solvent composition is also included in Table 1. The composition of the aqueous feed for the extraction test (Ex) was the SRS waste feed simulant given by [PETERSON-2000]. In this feed, the concentration of total Cs, both radioactive and non-radioactive, is 1.4E-04 M. The aqueous feed for the scrub tests (Sc1 and Sc2) was 0.05 M HNO₃. The aqueous feed for the strip tests (St1, St2, St3, and St4) was 0.001 M HNO₃. Each D value is an average of duplicate or, in some cases, triplicate tests.

Initial calculations using the distribution ratios given in Table 1 were done in two steps. First, the ratio, $D(\text{Ex})/D(\text{St4})$, was calculated for each solvent. This ratio is used as a tool to do an initial evaluation of solvent robustness with a higher ratio indicating a more robust solvent. As can be seen in Table 2, the two solvents containing 10 mM TOA should be the least robust based on the ratio $D(\text{Ex})/D(\text{St4})$. Second, the data were processed to get the values needed in the

Spreadsheet Algorithm for Stagewise Solvent Extraction (SASSE) worksheet [LEONARD-1994]. This preliminary data processing develops concepts, assumptions, and equations that will allow calculation of how D changes from stage to stage within a section. In the extraction and scrub sections, D is affected by solvent loading. In the strip section, D is affected by nitrate concentration.

Table 1. Cesium Distribution Ratios for Various CSSX Solvents^a

Solvent Code	Calix (x), mM	Cs-7SB (y), M	TOA (z), mM	D(Ex)	D(Sc1)	D(Sc2)	D(St1)	D(St2)	D(St3)	D(St4)
01	10	0.50	1	17.2	1.52	1.52	0.114	0.070	0.055	0.051
02	10	0.65	1	19.6	1.75	1.79	0.136	0.084	0.066	0.057
03	10	0.75	1	20.7	1.91	1.91	0.152	0.092	0.072	0.062
04	8	0.65	1	15.4	1.38	1.44	0.109	0.066	0.053	0.045
05	8	0.75	1	16.1	1.52	1.54	0.120	0.075	0.056	0.050
06	8	0.85	1	17.2	1.68	1.66	0.134	0.077	0.062	0.053
07	8	1.00	1	17.7	1.87	1.78	0.145	0.086	0.069	0.060
08	6	0.75	1	12.2	1.12	1.16	0.089	0.051	0.042	0.036
09	6	0.85	1	12.3	1.23	1.25	0.095	0.055	0.044	0.040
10	6	1.00	1	13.6	1.39	1.39	0.112	0.065	0.051	0.046
11	8	0.65	3	14.9	1.08	1.39	0.116	0.081	0.069	0.056
12	8	0.65	10	14.7	1.00	0.76	0.134	0.104	0.090	0.076
13	8	0.75	3	15.5	1.26	1.49	0.124	0.083	0.075	0.059
14	8	0.75	10	15.2	1.20	0.70	0.137	0.101	0.091	0.078

^aThese Cs distribution ratios were measured at ORNL [DELMAU-2001].

Table 2. Initial Calculated Results Using the D Values

Solvent Code	D(Ex)/D(St4)	D_{0,Ex}	D_{0,Sc}	D_{0,St1}	D_{0,St2}	D_{0,St3}	D_{0,St4}	D_{0,St.Avg2-4}
01	337	17.8	1.57	0.060	0.051	0.050	0.050	0.050
02	344	20.3	1.83	0.073	0.059	0.058	0.055	0.058
03	334	21.5	1.97	0.082	0.064	0.063	0.060	0.062
04	342	16.1	1.46	0.058	0.049	0.048	0.044	0.047
05	322	16.8	1.59	0.064	0.054	0.050	0.049	0.051
06	325	18.0	1.74	0.072	0.054	0.055	0.051	0.054
07	295	18.5	1.90	0.079	0.060	0.061	0.058	0.060
08	339	12.9	1.19	0.048	0.039	0.040	0.036	0.038
09	308	13.0	1.30	0.051	0.042	0.041	0.039	0.041
10	296	14.4	1.46	0.061	0.048	0.047	0.045	0.047
11	266	15.6	1.28	0.064	0.061	0.063	0.055	0.059
12	193	15.4	0.91	0.079	0.080	0.081	0.073	0.078
13	263	16.2	1.43	0.068	0.061	0.068	0.057	0.062
14	195	15.9	0.98	0.081	0.077	0.082	0.075	0.078

For the extraction and scrub batch tests, it is assumed that D is proportional to the concentration of free BOBCalixC6 in the organic phase—that is, the concentration of BOBCalixC6 that has not extracted any Cs. In addition, it is assumed that one molecule of BOBCalixC6 extracts only one Cs ion. The value of D when no Cs is loaded on the solvent is D₀. D₀ is obtained from the measured D for batch test i, D_i, by using

$$D_0 = D_i \cdot \left(\frac{y_{Calix}}{y_{Calix} - y_{Cs,i}} \right), \quad (1)$$

where y_{Calix} is the total concentration of BOBCalixC6 in the organic phase in \underline{M} and $y_{Cs,i}$ is the concentration of Cs in the organic phase from test i in \underline{M} . The value of $y_{Cs,i}$ is obtained by using the definition of D for batch test i, D_i, as follows:

$$y_{Cs,i} = x_{Cs,i} D_i, \quad (2)$$

where $x_{Cs,i}$ is the concentration of Cs in the aqueous phase from batch test i in \underline{M} . The value of $x_{Cs,i}$ is obtained from the material balance for batch test i as

$$x_{Cs,i} = \left(\frac{x_{Cs,F,i}}{R_i} + y_{Cs,F,i} \right) \cdot \left(\frac{R_i}{1 + R_i D_i} \right), \quad (3)$$

where R_i is the O/A volume ratio for batch test i , $x_{Cs,F,i}$ is the concentration of Cs in the aqueous feed to batch test i in \underline{M} , and $y_{Cs,F,i}$ is the concentration of Cs in the organic feed to batch test i in \underline{M} . Equations 1-3 were applied to the D value from the extraction test to get D_0 for the extraction section, $D_{0,Ex}$. These values are listed in Table 2. The same three equations were applied to each batch scrub test to get D_0 for the scrub section, $D_{0,Sc}$. The average of the two D_0 values for the scrub section for each solvent are listed in Table 2.

For the strip batch tests, it was assumed that D_i is proportional to the concentration of nitrate ion in the aqueous phase. It was further assumed that this nitrate comes from only two sources, $CsNO_3$ in the organic feed to the strip tests and HNO_3 in the aqueous feed to the strip tests. The value of D when only HNO_3 from the strip feed is present is called $D_{0,St}$ and is obtained from D_i using

$$D_{0,St} = D_i \cdot \left(\frac{x_{Cs,F,i}}{x_{Cs,F,i} + x_{Cs,i}} \right). \quad (4)$$

The calculations from the batch tests for extraction and scrub give the concentration of Cs in the organic feed for the first batch strip test. Then Equations 2-4 are used to get D_0 for each batch strip test, $D_{0,Sti}$, where $i = 1, 2, 3, 4$. These values are listed in Table 2 along with the average of the last three $D_{0,Sti}$ values. The $D_{0,St1}$ value was omitted from the average as it is typically high. This is because our assumption about the amount of nitrate is not quite correct, especially for the first strip stage. There is some nitrate from $NaNO_3$, KNO_3 , and $Al(NO_3)_3$ in the first strip stage. The average is called $D_{0,St,Avg2-4}$. Because $D(St4)$ was typically as low or lower than $D_{0,St,Avg2-4}$, it was used in the SASSE calculations as the D_0 value for the strip section, $D_{0,St}$.

Using the processed D values, the flowsheet calculations were carried out using the SASSE worksheet in Microsoft Excel. The CSSX flowsheet is that shown in Figure 1. It has 15 extraction stages, 2 scrub stages, and 15 strip stages. The solvent is recycled. The temperature is $25^\circ C$; the Cs concentration in the DF feed is $1.4E-04 \underline{M}$; and the stage efficiency is assumed to be 80%. Equation 1 was rearranged and an iterative calculation was used to get the D_i values for the extraction and scrub sections with D_0 values of $D_{0,Ex}$ and $D_{0,St}$, respectively. The four $D(Sti)$ values were used as the D_i values for the first four stages of the strip section. All additional strip stages were assumed to have a D_i value of $D(St4)$. In these calculations, the ratio for the Cs concentration in the aqueous (DF) waste feed to that in the aqueous (DW) raffinate is divided by the desired ratio of 40,000. This number, called the robustness (N_{Rb}), gives a measure of how much room the process has for fluctuations in flow rate, concentration, temperature, and stage efficiency. For each solvent, N_{Rb} was calculated in two different ways. First, N_{Rb} was calculated

for a solvent flow rate (q_{DX}) of 6.6 gpm (gallons per minute), $N_{Rb, 6.6 \text{ gpm}}$. Second, the solvent flow rate was varied until the maximum N_{Rb} , $N_{Rb, \text{max}}$, was obtained. The results of these calculations are listed in Table 3. Note that the desired N_{Rb} , which is >3 [KLATT-2001], is met in all cases if the optimum flow rate is used.

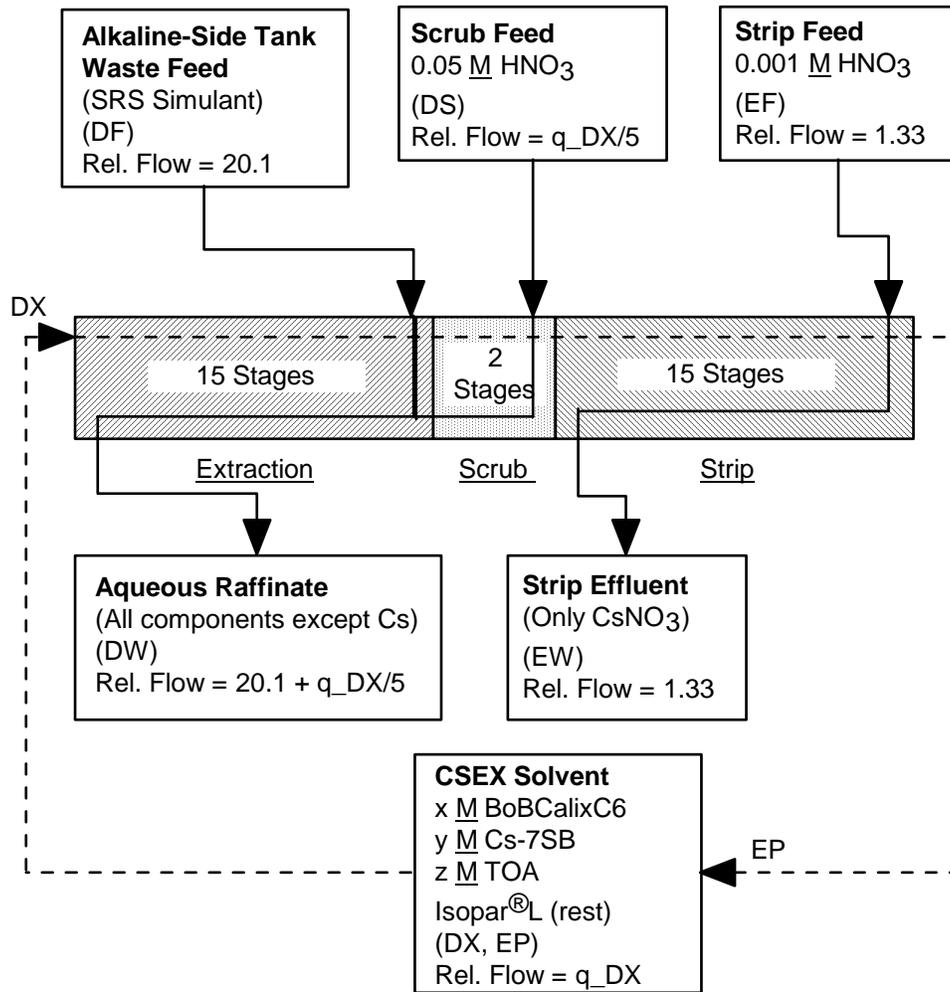


Fig. 1. CSSX Flowsheet Used for SASSE Calculations. The relative flow rates shown correspond to the baseline plant flow rates in gpm. Flow rates and concentrations not shown vary as stated in the text. The baseline flow rate for q_{DX} is 6.6 gpm. The baseline concentration for x is 0.010 M; y, 0.50 M; and z, 0.001 M.

Table 3. Calculated Robustness for Various CSSX Solvents

Solvent Code	Calix (x), mM	Cs-7SB (y), M	TOA (z), mM	$N_{Rb, 6.6 \text{ gpm}}$	$N_{Rb, \text{max}}$	Optimum Flow Rate (Flow Rate at $N_{Rb, \text{max}}$), gpm
01	10	0.50	1	21.9	25.3	6.1
02	10	0.65	1	11.86	26.4	5.3
03	10	0.75	1	6.56	23.6	4.9
04	8	0.65	1	25.0	25.3	6.7
05	8	0.75	1	19.33	20.7	6.2
06	8	0.85	1	15.71	21.2	5.8
07	8	1.00	1	7.28	15.37	5.4
08	6	0.75	1	8.76	24.3	8.5
09	6	0.85	1	9.06	17.84	8.0
10	6	1.00	1	14.32	15.63	7.0
11	8	0.65	3	8.82	10.13	6.1
12	8	0.65	10	1.14	3.03	5.3
13	8	0.75	3	6.90	9.74	5.8
14	8	0.75	10	1.01	3.23	5.1

III. DISCUSSION

All the solvents tested meet the robustness acceptance criterion of 3 or greater [KLATT-2001], if the optimum flow rate for the solvent is used. The only solvent better than the baseline solvent 01 with respect to robustness is solvent 02. $N_{Rb, \text{max}}$ for solvent 04 is equal to that for solvent 01. Solvents 03 and 08 have a $N_{Rb, \text{max}}$ values that are close to that for solvent 01 (less than 10% less). Of the four solvents with increased TOA, solvent 11 has the highest $N_{Rb, \text{max}}$. Solvent density, along with the dispersion number, usually determines the maximum throughput that a centrifugal contactor of a given size can achieve. As solvent density increases, maximum throughput decreases. For the CSSX solvents, solvent density increases as the modifier, Cs-7SB, increases [BIRDWELL-2001]. Thus, the two solvents with 0.65 M Cs-7SB, 02 and 04, are especially attractive. Of these two solvents, solvent 04 is the more attractive one as it has less BOBCalixC6. This helps in two ways. First, solvent cost will be less. Second, the BOBCalixC6 will be further from a concentration where it could precipitate from the solvent or closer to being completely soluble. We need the results from the tests that are measuring BOBCalixC6 solubility in solvent before we can say which is the case.

To ensure good BOBCalixC6 solubility, the Cs-7SB concentration may need to be higher than 0.65 M. The reason for this is that increasing the concentration of Cs-7SB increases the solubility of BOBCalixC6. If a higher Cs-7SB concentration is needed, 0.75 M Cs-7SB should be considered next. Solvents 03, 05, 08, 13, and 14 all have this modifier concentration. Except for the two solvents with the higher TOA concentrations, all robustness values for the solvents with 0.75 M Cs-7SB are close to solvent 01, within a factor of 0.8 or higher.

It is important to note that the SASSE calculations are conservative since they assume a constant process temperature of 25°C and a stage efficiency of 80%. In an actual process, the extraction section would be kept cooler than the strip section so that process robustness is improved [BONNESEN-2000]. For example, if the extraction section were at 25°C, the scrub section at 29°C, and the strip section at 33°C, the N_{Rb} for a solvent flow rate of 6.6 gpm would increase from 21.9 to 80.7. The maximum N_{Rb} is 300 for this case and occurs at a solvent flow rate of 9.3 gpm. Thus, by controlling temperature, process robustness can be increased substantially.

While a stage efficiency of 80% was assumed for the centrifugal contactor, the actual efficiency can be 90% or greater. This is certainly true for larger contactors [LEONARD-1999], but it was also true for the small 2-cm contactor after it was modified as described in [LEONARD-2000]. If the stage efficiency were 90% in the SASSE calculations at 25°C, the N_{Rb} at a solvent flow rate of 6.6 gpm would increase from 21.9 to 599. The maximum N_{Rb} is 1003 and occurs at a solvent flow rate of 5.8 gpm. Thus, higher stage efficiency, which we can expect in the plant, will also increase process robustness.

It is desirable to have an easy way to estimate the robustness that can be expected for a given solvent. The use of the ratio $D(Ex)/D(St4)$ was reviewed here for its potential in this regard. As can be seen by comparing Tables 2 and 3, the use of this ratio in place of $N_{Rb, max}$ does give a guide as to the comparative robustness of two solvents. However, it is not a proportional effect. For example, the drop in robustness from the baseline solvent (solvent 01) to the two solvents with 10 mM TOA (solvents 02 and 04) is 4.7 times greater than predicted by the use of $D(Ex)/D(St4)$. Thus, while this ratio can be used as a guide, it should not be used in place of $N_{Rb, max}$.

Because all $N_{Rb, max}$ values in Table 3 meet the robustness criterion of 3 or greater, any of the 13 new CSSX solvents can be considered. Since the temperature and stage efficiency assumptions are conservative, any of these solvents would work well with respect to flowsheet robustness determined from the D_{Cs} values. Thus, the final solvent selection must be based on

other properties such as viscosity, dispersion number, third-phase formation, density, and BOBCalixC6 solubility.

IV. SUMMARY AND CONCLUSIONS

The D_{Cs} values for the new solvents to replace the current baseline CSSX solvent (0.010 M BOBCalixC6, 0.50 M Cs-7SB, 0.001 M TOA in Isopar[®]L) were compared using (1) the current baseline CSSX flowsheet and (2) the same flowsheet with a solvent flow rate optimized for the particular solvent. Except for the two solvents with 0.010 M TOA, all solvents met the robustness criterion at the baseline solvent flow rate of 6.6 gpm. When the optimized flow rate for each solvent is used, all solvents meet the robustness criterion of >3 [KLATT-2001]. Since the temperature and stage efficiency assumptions are conservative, any of these solvents would work well with respect to flowsheet robustness for the CSSX process. Thus, the final solvent selection will be based on other solvent properties including viscosity, dispersion number, third-phase formation, density, and BOBCalixC6 solubility.

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