ANL/CSE-13/11 Rev

Centrifugal Contactor Operations for UREX Process Flowsheet: An Update

Chemical Sciences and Engineering Division

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Centrifugal Contactor Operations for UREX Process Flowsheet: An Update

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1 OVERVIEW

The uranium extraction (UREX) process separates uranium, technetium, and a fraction of the iodine from the other components of the irradiated fuel in nitric acid solution. In May 2012, the time, material, and footprint requirements for treatment of 260 L batches of a solution containing 130 g-U/L were evaluated for two commercial annular centrifugal contactors from CINC Industries [1]. These calculated values were based on the expected volume and concentration of fuel arising from treatment of a single target solution vessel (TSV). The general conclusions of that report were that a CINC V-2 contactor would occupy a footprint of 3.2 m^2 (0.25 m x 15 m) if each stage required twice the nominal footprint of an individual stage, and approximately 1,131 minutes or nearly 19 hours is required to process all of the feed solution. A CINC V-5 would require approximately 9.9 m^2 (0.4 m x 25 m) of floor space but would require only 182 minutes or ~ 3 hours to process the spent target solution. Subsequent comparison with the Modular Caustic Side Solvent Extraction Unit (MCU) at Savannah River Site (SRS) in October 2013 [2] suggested that a more compact arrangement is feasible, and the linear dimension for the CINC V-5 may be reduced to about 8 m; a comparable reduction for the CINC V-2 yields a length of 5 m. That report also described an intermediate-scale (10 cm) contactor design developed by Argonne in the early 1980s that would better align with the SHINE operations as they stood in May 2012. In this report, we revisit the previous evaluation of contactor operations after discussions with CINC Industries and analysis of the SHINE process flow diagrams for the cleanup of the TSV, which were not available at the time of the first assessment.

In addition to the two standard CINC contactor designs, we included an intermediate-scale contactor that has characteristics derived from the available CINC design specifications and the ROTOR model calculations for the two commercial units. CINC has stated that contactors can be fabricated at non-standard dimensions and mounted as required to meet user needs, so TSV processing need not be limited by the contactor obtainability. In this report, the three different sizes evaluated are the standard CINC V-2 and V-5 units and an intermediate unit designated the "V-3.5" (also referred to herein as "V-035"), which is based on the other two designs. The V-2 has a 2-in. (5-cm) dia rotor with a reported maximum total organic-plus-aqueous throughput of 1.9 L/min; this unit was evaluated for a maximum throughput of 1 L/min. The V-5 has a 5-in. (~12.5-cm) dia rotor with a reported maximum throughput of 19 L/min; it was evaluated for a throughput of 15 L/min. The V-3.5 unit has a 3.5-in. (9-cm) dia rotor with an estimated maximum throughput of ~6.6 L/min. This unit was evaluated for a maximum operational throughput of 5 L/min. The lower-than-maximum throughputs selected for evaluation are based on calculations using the Argonne ROTOR model, which defines the operational envelope for a given ratio of organic-to-aqueous (O:A) flow rate based on contactor parameters (weir dimensions, rotor diameter, etc.), the physical properties of the two immiscible fluids, and the allowable other-phase carryover. As the UREX process designed for this application [3, 4] has O:A ratios ranging from 0.8 to 5.6, adjustments to the weirs in different sections of the contactor bank can broaden the operational envelope for satisfactory separations, but throughput will remain short of the equipment maximum due to the physical characteristics of the two phases.

2 CALCULATIONS

The reference UREX flowsheet consists of an extraction, two scrubs, and a strip section with a total of 30 stages. All of the calculations described here assume that all 30 stages are housed in a single shielded facility with additional space allocated for pumps and tanks as required. Three additional diluent-wash contactor stages are used to clean the uranyl nitrate product of residual tributyl phosphate prior to denitration. Each of the wash stages operates on an independent diluent recycle loop and, therefore, requires an independent pump and recycle diluent tank. Four additional solvent-wash stages are also assumed, but would be used only periodically because of the low burn-up of the target solution. Both of these cleanup systems are excluded in the calculations because they can be located outside the shielded cell facility, based on the expected low activities in the spent solvent and the uranyl nitrate product.

2.1 CENTRIFUGAL CONTACTOR FOOTPRINT

Table 1 provides key layout parameters for the three contactor systems from CINC Industries. The 30-stage cases assume that the spacing allotted for the inter-stage lines required for multiple stages in series will not increase the lateral dimension of the contactor beyond that needed for the support stands supplied with the individual units by CINC Industries if the interconnect tubing is aligned perpendicular to the stages [1]. This is the arrangement adopted for the MCU at SRS [2] as the stages to be situated are much closer together than the arrangement with inter-stage lines between the contactor bodies, reducing the lateral dimension (length) but increasing the front-to-back width. The perpendicular arrangement also allows for easier access to the inter-stage lines if both the front and back of the shielded cell can be accessed remotely. The values reported below for the V-2 and the V-5 are based on the specifications for those units listed on the CINC website as of April 2014 [5]. Based on the specifications, a single V-5 bank will require approximately 2.8 m^2 of floor space (0.3 m x 9.2 m), while the V-3.5 would require approximately 2.0 m^2 (0.26 m x 7.7 m). Even if the very compact custom arrangement of the MCU is used, the relative scales should basically hold. The contactor can be segregated at various points to break the straight line into sections if a smaller lateral dimension is required to accommodate the shielded cell space. For example, there is a natural break between the second scrub and strip section, where the contactor could be divided into an 18-stage and 12-stage unit. This approach was taken in SHINE's UREX cell schematic shown in Figure 1. By breaking up the sections in this way, the two segments can be arranged in separate rows or even tiered.

Note that the width of the base of the contactor support was listed as 40.6 cm in the specifications page for the V-5 on the CINC Industries webpage in May 2012. Either a narrower support structure is now used for the standard model, or the previous value was erroneous since the current specifications list a base width of 30.5 cm. The 40.6 cm value results in a 12.2-m long contactor for closely packed stages rather than the 9.2 m length given above and Table 1. Assuming an inter-stage spacing equivalent to the width of the base (effectively doubling the length of a 30-stage bank) results in a linear width of 18.4 m for 30 stages for a 30.5 cm base rather than the 25 m listed in our earlier report [1] for the 40.6 cm base. The narrower base

TABLE 1 Dimensional Specifications for CINC V-2 and V-5 Contactors [5] and Estimates for a V-3.5 Contactor

	V-2		V-5		V-035	
Characteristic	1-stage	30 stages*	1-stage	30 stages*	1-stage	30 stages*
Width (m)	0.23	6.9	0.305	9.2	0.26	7.7
Footprint (m^2)	0.05	1.6	0.09	2.8	0.07	2.0
Height (m)	0.610		1.070		0.828	
Weight (kg)	12.8	384.0	77.1	2313.0	39.6	1187.6
Max Throughput (L/min)	1.900		18.900		6.650	
Rotor Speed (rpm)	2000-6000		800-4000		800-4000	
Hold-up (L)	0.250	7.5	3.250	97.5	1.400	42.0

*The thirty-stage case includes an estimate of added space required for inter-stage lines.

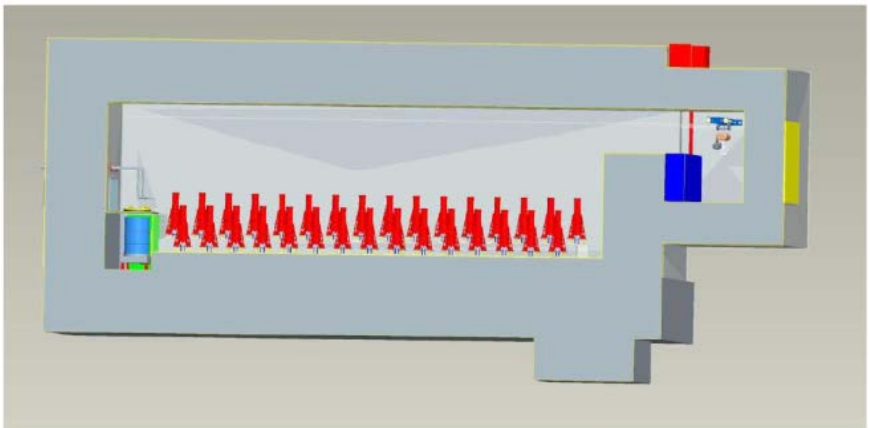

FIGURE 1 Schematic of Contactors in Shielded Cell Facility

dimension is also more in line with the ~26 cm width per stage estimate for the MCU contactor [2]. The specified V-2 base width is unchanged at 23 cm.

From an examination of the PFDs provided in a SHINE report [6], the volume of solution to be processed using UREX would not be 260 L, the nominal TSV volume assumed in the 2012 report. Rather, it is approximately 2.3 times larger based on the relative mass flows of stream 611B, which is the total uranyl nitrate solution to UREX (1741.4 lb/batch), and stream 508B, which is the spent TSV solution and the sulfuric acid wash (749.4 lb/batch). Also, the reference UREX flowsheet was developed for a 0.55M uranium feed, which is similar to, but slightly higher than, the uranium concentration in stream 611B, which is closer to 0.45M (9.06 mass% uranium). To maintain comparative consistency with the 2012 report, the flowsheet is left unchanged. However, calculations are done for both 260 L and 625 L of uranyl nitrate feed. We calculated the latter volume from a simple ratio of mass flows reported for streams 508B and 611B in the Uranyl Nitrate Preparation PFD, accounting for the small difference in densities.

2.2 UREX PROCESSING TIME REQUIREMENTS

The initial operational sequence for the UREX centrifugal contactor consists of four basic steps:

1. Cold startup: All the feeds are introduced to pre-fill the contactor before introduction of spent uranium nitrate solution.

2. Processing of uranium feed: Once the contactor is filled with both aqueous and solvent phases, uranium feed is introduced, after which residual fission products first exit in raffinate, and purified uranium is first recovered as product.

3. Residual uranium rinse: Cold feeds reintroduced to flush out remaining uranium to product.

4. Residual acetohydroxamic acid (AHA) rinse: Cold feeds continue with AHA removed from scrub.

After all of the irradiated fuel is introduced, a substantial fraction of the uranium remains in the contactor. As a result, a cold 1 M nitric acid feed must be introduced to recover this uranium. If uranium solution processing is on a short schedule (e.g., weekly), steps 1 and 4 can likely be combined, or perhaps omitted if the contactor is routinely left filled with uranium rinse solution. Both steps 1 and 4 should be retained if downtime is long, on the order of a month.

Table 2 provides time required for each process step and the cumulative processing time for a single 260 L TSV batch for a V-2 contactor operating at a maximum flow rate of 1 L/min, a V-5 contactor at 15 L/min, and a V-3.5 contactor at 5 L/min. The startup and rinse/shutdown times are comparable for all three cases. Once the hot feed is exhausted, cold acid feed is introduced to initiate the uranium cleanout, while all other flows are maintained. This cleanout moves essentially all of the uranium in the extraction and scrub sections out into the uranium strip product after the hot feed is exhausted. Nearly all of the residual uranium remaining after processing will be collected in the initial pass, but a flush of the entire system is assumed in order to recover residual uranium due to other-phase carryover. The fuel processing time for the V-3.5 will be a factor of two larger than that for the V-5, and a factor of four smaller than that for the V-2. The overall processing time, including rinses, is larger than that of the V-5 by nearly a factor of two. For this case, the V-3.5 requires 5.7 hours to process all of the feed rather than the 3 hours for the V-5 and 19 hours for the V-2.

The estimated processing times for a 625 L batch of feed are shown in Table 3. The cold startup and rinses are identical to the 260 L case and thus account for a smaller fraction of the time required to process the uranium feed. Obviously, the larger feed volume translates into significantly longer processing times: 10 hours for the V-3.5, 4.5 hours for the V-5, and 41 hours for the V-2.

TABLE 2 Processing Time Requirements for V-2, V-5, and V-3.5 Contactors for 260 L of Feed

Process Step	Run Time (min) V-2 (1 L/min)	V-5 (15 L/min)	V-035 (5 L/min)	Cumulative Run Time (min) V-2 (1 L/min)	V-5 (15 L/min)	V-035 (5 L/min)
Cold Startup of all feeds	31	25	32	31	25	32
Initial hot feed to activity in raffinate	4	3	4	35	28	36
Initial hot feed to uranium in product	22	16	20	57	44	57
Process 260 L of hot feed	972	65	194	1029	109	251
Initial hot feed to exit last uranium in product	994	81	215	1051	125	271
Residual U rinse	44	32	41	1077	144	296
Residual AHA rinse*	54	37	47	1131	182	343

*Residual AHA rinse is only required for extended shutdown.

TABLE 3 Processing Time Requirements for V-2, V-5, and V-3.5 Contactors for 625 L of Feed

Process Step	Run Time (min) V-2 (1 L/min)	V-5 (15 L/min)	V-035 (5 L/min)	Cumulative Run Time (min) V-2 (1 L/min)	V-5 (15 L/min)	V-035 (5 L/min)
Cold Startup of all feeds	31	25	32	31	25	32
Initial hot feed to activity in raffinate	4	3	4	35	28	36
Initial hot feed to uranium in product	22	16	20	57	44	57
Process 625 L of hot feed	2338	156	467	2394	200	524
Initial hot feed to exit last uranium in product	2359	172	488	2416	216	544
Residual U rinse	44	32	41	2442	235	569
Residual AHA rinse*	54	37	47	2496	273	616

*Residual AHA rinse is only required for extended shutdown.

2.3 IN-PROCESS HOLDUP AND EFFLUENT VOLUMES

The other major factors driving the contactor selection are related to fluid management, in-process holdup, and effluent volumes. Table 4 provides estimates of the in-process holdup for the four UREX sections and the overall fluid holdup for the three contactor types based on ROTOR code predictions. Holdup in a given section is ten to fifteen times larger for the V-5 compared with the V-2, and two to three times larger than the V-3.5. The ROTOR code predicts that the V-2 holdup will contain 2.9 L of organic phase and 5.7 L of aqueous phase, while the V-5 holdup will contain 39 L of organic and 73 L of aqueous phase. The ROTOR code results for these cases were used to estimate the holdup fractions for the V-3.5, yielding approximately 17 L of organic and 30 L of aqueous solution held up during a run. Because the composition of the phases will vary significantly across any section during uranium extraction, the U rinse initiated after all of the fuel feed has been introduced would serve to homogenize the solution that is held up as it forces all of the uranium to the strip product and the fission products to the raffinate.

Required input volumes for both the cold and the irradiated-fuel feed were calculated based on the UREX processing times in Table 2 and the volumetric feed flow rates in Table 5. These input volumes are shown in Table 6 for a 260 L feed and in Table 7 for a 625 L feed. For the solvent values, we assumed that a volume three times the total amount of solvent that is held

TABLE 4 Holdup per Section for V-2, V-5, and V-3.5 Contactors Based on the Reference UREX Flowsheet

	V-2 (1 L/min)		V-5 (15 L/min)		V-035 (5 L/min)	
Hold-up (L)	Organic	Aqueous	Organic	Aqueous	Organic	Aqueous
Extraction	0.7	1.4	8.7	17.5	3.9	7.3
Scrub 1	1.1	1.2	18.5	11.4	7.8	4.9
Scrub 2	0.1	0.1	2.2	1.2	0.9	0.5
Strip	0.8	2.2	9.5	29.8	4.4	12.4
Overall	2.9	5.7	39.0	72.8	17.3	30.4

TABLE 5 Process Flow Rates for V-2, V-5, and V-3.5 Contactors

Input Flow Rate (L/min)	V-2 (1 L/min)	V-5 (15 L/min)	V-035 (5 L/min)
Irradiated Fuel Feed	0.27	4.0	1.3
Solvent*	0.45	6.8	2.3
Scrub 1	0.01	0.2	0.1
Scrub 2	0.08	1.2	0.4
Strip	0.55	8.2	2.7
Raffinate	0.36	5.4	1.8
Product	0.55	8.2	2.7

*Solvent in recycle mode assumed to require about three times holdup.

TABLE 6 Process Feed Volumes Required for V-2, V-5, and V-3.5 for the UREX Flowsheet for 260 L of Feed

Process Feed (L required)	V-02 (1 L/min)	V-05 (15 L/min)	V-035 (5 L/min)
Irradiated Fuel Feed	260	260	260
Start-up/Rinse Feed	34	293	124
Solvent*	9	117	52
Scrub 1	15	24	18
Scrub 2	88	147	107
Strip	604	1003	732

*Solvent in recycle mode assumed to require about three times holdup.

TABLE 7 Process Feed Volumes Required for V-2, V-,5 and V-3.5 for the UREX Flowsheet for 625 L of Feed

Process Feed (L required)	V-02 (1 L/min)	V-05 (15 L/min)	V-035 (5 L/min)
Irradiated Fuel Feed	625	625	625
Start-up/Rinse Feed	34	293	124
Solvent*	9	117	52
Scrub 1	33	43	36
Scrub 2	198	256	217
Strip	1352	1752	1481

*Solvent in recycle mode assumed to require about three times holdup.

up in the contactor is circulated continually during processing, although the volume of solvent actually deployed will depend on the solvent wash schedule and the tankage allotment in the shielded cell facility.

Table 8 lists the total volume of effluent generated per processing step for the three operational cases for 260 L of feed, while Table 9 shows the values for the 625 L case. As is readily evident, the greatest benefit arising from use of the smaller contactors comes from the reduced start-up and rinse volumes due to the lower contactor holdup. The effluent volume numbers suggest that, if the processes are run continually, there is substantial benefit to operating with the larger contactor because the start-up and rinse cycles may be shortened or perhaps eliminated (if material accountancy can be managed). By contrast, batch operation favors smaller units due to the smaller liquid-waste volumes generated. As the UREX feed volume, based on stream 611B, is significantly larger than that used in the earlier analyses, the start-up and shut-down effluent volumes constitute a relatively small fraction of the overall liquid volumes generated as wastes. This condition mitigates some of the advantage gained from smaller holdup volumes.

TABLE 8 Process Effluent Volumes Generated for V-2, V-5, and V-3.5 for the UREX Flowsheet for 260 L of Feed

Process Volumes (L)	V-2 (1 L/min)	V-5 (15 L/min)	V-035 (5 L/min)
Raffinate generated during cold start-up	11	137	58
Product generated during cold start-up	17	207	88
Raffinate generated processing fuel	351	351	351
U Product from fuel treatment	533	533	533
Raffinate generated during U rinse	16	173	73
Dilute product generate during U rinse	24	263	111

TABLE 9 Process Effluent Volumes Generated for V-2, V-5, and V-3.5 for the UREX Flowsheet for 625 L of Feed

Process Volumes (L)	V-2 (1 L/min)	V-5 (15 L/min)	V-035 (5 L/min)
Raffinate generated during cold start-up	11	137	58
Product generated during cold start-up	17	207	88
Raffinate generated processing fuel	844	844	844
U Product from fuel treatment	1281	1281	1281
Raffinate generated during U rinse	16	173	73
Dilute product generate during U rinse	24	263	111

3 CONCLUSIONS

This report is an update of the first analysis of contactor requirements completed in May 2012 [1]. The tradeoff between the two extreme contactor sizes remains clear. The V-2 requires a substantially longer processing time, up to 19 hours. The V-5 reduces the processing time by a factor of nine, but the volumes of waste that are generated increase by a factor of ten or more. This study added an intermediate contactor size, designated V-3.5, based on the results of the earlier study, additional information obtained from SHINE Technologies, process flow diagrams for the Mo-99 production process, and discussions with CINC Industries. The quantities of cold chemicals needed simply to start up and shut down the V-5 are more than halved for the V-3.5. On the other hand, the processing time required for the V-3.5 is approximately double. The length of the V-3.5 contactor is less than that of the V-5, although there is some room for reducing the calculated lengths by about 20% based on the unit installed in the MCU facility at SRS. The smaller V-3.5 would free up additional shielded-cell space for other operations such as solvent clean-up or equipment maintenance, as well as any unforeseen tasks.

Perhaps the most significant determinant in the initial evaluation [1] was the processing time. For the feed batch volume of 260 L used in the first calculation, the V-2 requires 19 hours, which was deemed too long to allow completion in a single day, while the V-5 requires approximately 3 hours, and the V-3.5 requires 5.7 hours. Given the reduced holdup, cold-feed requirements, and waste volumes, the V-3.5 would appear to be a nearly optimal scale for the 260-L feed batch volume. However, the major benefit of the larger contactor, namely, reduced processing time, becomes more significant for the larger 625 L UREX feed batch volume estimated from PFD stream 611B. The non-linear characteristics of the contactor yield an increase in the V-5 processing time to only 4.5 hours, while the V-3.5 requires about 10 hours and the V-2 nearly 41 hours. Note that the characteristics of the V-3.5 are derived from the calculations made for the V-2 and V-5, where key design parameters were available for input to the ROTOR model to predict contactor operational characteristics. Because these data are not yet available for the V-3.5, the predicted values are more uncertain, and processing time, holdup, and footprint will be somewhat, perhaps significantly, different than shown here.

4 FURTHER WORK

For future work, the interface between UREX processing of fuel solutions and the other key facility operations should be systematically studied to attain greater confidence in the contactor selection, particularly the relationships among available tankage, timing of operations, and waste-form production. Given the solution volumes and processing times, facility functionality will be substantially affected if a sub-optimal contactor is placed in service. The benefit of combining the start-up and rinse steps, as well as semi-continuous processing, should be evaluated to determine any impact on the ultimate product quality.

There would appear to be some difference in the expected composition of the feed used for the flowsheet calculation and that delineated in the 5-1-13 PFDs provided by SHINE Medical Technologies. This difference arises from changes that have been made to the baseline facility flowsheet as the overall process has evolved. Consequently, the UREX flowsheet should be recalculated once these process flows are more fully fixed. As a first course, the flowsheet should be evaluated and, possibly, revised on the basis of the 5-1-13 PFDs using the Argonne Model for Universal Solvent Extraction (AMUSE) code. It is also recommended that a sensitivity analysis be carried out using AMUSE to determine the robustness of the flowsheet to changes in operation, whether due to changes in upstream operations or variability in process flows and feed compositions. Although UREX flowsheets are generally robust, a sensitivity analysis should be done to determine where the designed flowsheet may break down. Typically, this would involve varying a number of process parameters, such as flow rate and feed concentrations, and calculating the system's response.

In terms of equipment, there is substantial room for optimization of contactor design. Based on the 260 L feed batch volume and time constraints, the V-3.5 may be near-optimal scale. However, with a larger feed batch volume, this is no longer clear and requires a detailed analysis of the impacts on upstream and downstream processing. It would appear that the V-5 is closer to optimal for the larger feed volume. Since contactors have significant turndown ratios, the V-5 throughput can be reduced by a factor of ten with relatively small reductions in performance.

Even for the standard CINC V-5 contactor, key design elements must be detailed. Since CINC has experience with designing remotely operated contactors for nuclear installations, many desirable external features are already identified. In terms of the internals, the weirs which regulate phase separation within the rotors can be optimized by using the ROTOR model. The appropriate weir dimensions are critical to throughput, high stage efficiency, and reduced carryover; CINC units are equipped with multiple weirs, from which the user selects that most suited to the process of interest, but the choice should be verified experimentally. The number of vanes and their orientation, the width of the annular gap, and the optimal liquid height in the mixing zone should also be experimentally analyzed during the design process.

Finally, the holdup and associated phase ratios that are used in this document should be confirmed experimentally using the V-5, such as the unit on-hand at Argonne, for the several O:A phase ratios that occur in the UREX flowsheet. The processing estimates can then be revised with these experimental values. If the V-3.5 unit is preferred by SHINE, 3-D printer technology can be used to produce a single unit in a matter of days to collect the required data and verify the ROTOR predictions. The detailed CINC design parameters can be incorporated into the ROTOR model once CINC develops a design for eventual manufacture. These design parameters would yield a better estimate of processing time, feed and effluent volumes, and in-process holdup.

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