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CENTRIFUGAL CONTACTORS FOR LABORATORY-SCALE SOLVENT EXTRACTION TESTS

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ABSTRACT

A 2-cm contactor (minicontactor) was developed and used at Argonne National Laboratory for laboratory-scale testing of solvent extraction flowsheets. This new contactor requires only 1 L of simulated waste feed, which is significantly less than the 10 L required for the 4-cm unit that had previously been used. In addition, the volume requirements for the other aqueous and organic feeds are reduced correspondingly. This paper (1) discusses the design of the minicontactor, (2) describes results from having applied the minicontactor to testing various solvent extraction flowsheets, and (3) compares the minicontactor with the 4-cm contactor as a device for testing solvent extraction flowsheets on a laboratory scale.

INTRODUCTION

The Argonne centrifugal contactor was developed in the early 1970s for carrying out solvent extraction operations required in the nuclear industry (1). However, it is very versatile and should find application wherever solvent extraction processes are required. A schematic of an operating contactor is given in Fig. 1. Detailed discussion of contactor operation can be found elsewhere (1,2). A typical solvent extraction flowsheet contains multistage extraction/scrub sections that recover

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discusses (1) the design and operation of the minicontactor, especially the changes that were required to make it work over the full range of organic-to-aqueous (O/A) flow ratios, (2) the application of the minicontactor to various solvent extraction flowsheets, emphasizing any problems with the contactor or the process, and (3) the differences between the minicontactor and the 4-cm contactor as devices for testing solvent extraction flowsheets on a laboratory scale.

DESIGN AND OPERATION OF MINICONTACTOR

A 2-cm contactor built in the past could not be operated below an O/A flow ratio of 0.8; therefore, the unit was not entirely satisfactory for testing solvent extraction flowsheets (2). The problem was failure of the phases to invert. The continuous phase would not change easily from organic to aqueous at O/A flow ratios below 1.0. Tests done at the time indicated that performance might be improved by (1) increasing the rotor length and (2) increasing the annular gap between the spinning rotor and the stationary housing (the Couette gap).

The goal of designing a new 2-cm contactor was to eliminate the problems with the original (referred to hereafter as "old") design. Further testing of the hydraulic performance in 2-cm and 4-cm contactors at Argonne indicated that, while increasing the rotor length does help, the Couette gap should be decreased for a rotor of a given radius. As the gap is decreased, the range of O/A flow ratios for which either phase can be the continuous phase also decreases. Most important, the dispersion in the mixing zone becomes aqueous continuous before the O/A flow ratio drops so low (about 0.5) that the dispersion viscosity increases rapidly. If the dispersion does not pass through a phase inversion before the O/A ratio drops below 0.5, the dispersion develops a structural viscosity similar to foam that causes operating problems (2).

To characterize the 2-cm contactors, two dimensionless quantities are determined. The first is a dimensionless Couette gap, N_G , given by

$$N_G = \frac{r_{Hi} - r_{Ro}}{r_{Ro}} \quad (1)$$

where r_{Hi} is the inside radius of the stationary housing around the rotor, and r_{Ro} is the outside radius of the rotor. The second is a dimensionless rotor length, N_L , given by

$$N_L = \frac{L}{2 r_{Ro}} \quad (2)$$

where L is the length of the separating zone inside the rotor, that is, the length from the rotor inlet to the less-dense-phase (lower) weir. The N_G values for the old 2-cm contactors were from 0.31 to 0.45; the N_L values were from 1.05 to 1.49 (2). The contactors with the higher N_L values gave better, but still not satisfactory, performance.

For the new 2-cm contactor discussed here, N_G was decreased by a factor of two, to 0.15, and N_L was increased by 60%, to 2.36. Hydraulic tests with the new minicontactor showed that this solved the problem with phase inversion (5). For these tests, mixing-zone operation was viewed by use of a transparent housing made of an acrylic resin. Typical mixing-zone operation is shown in Fig. 2.

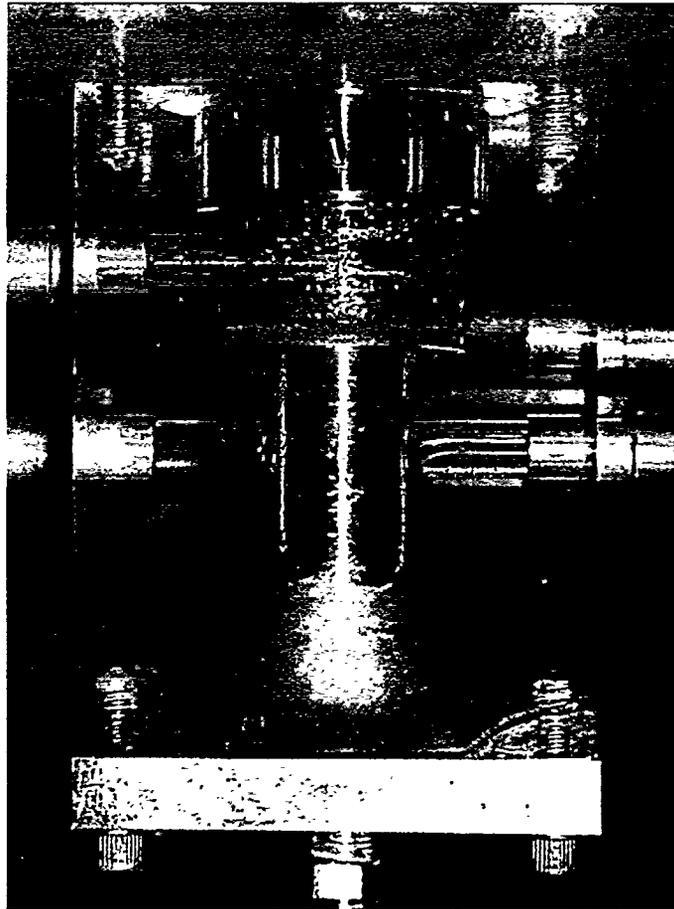


FIGURE 2. Mixing-zone operation in the new minicontactor.

Other changes to the old 2-cm contactor design were to (1) eliminate the o-ring drive belt and (2) reduce the rotor speed. The former makes it easier to add stages and disposes of the possibility of drive belt breakage. Decreasing rotor speed from 6000 rpm to 3600 rpm caused a proportional loss in throughput. However, this was partially offset by the increased length of the separating zone. In addition, the decreased rotor speed and elimination of the drive belt make it possible to use an off-the-shelf electric motor from Bodine Electric (Chicago, IL, model 710). A key design question was the first natural frequency of this small motor spinning the new elongated rotor. For satisfactory operation, the first natural frequency of the motor/rotor assembly should be at least 72 Hz, that is, 20% above the operating speed of 3600 rpm (60 Hz). A vibrational analysis of this system indicated that the first natural frequency of the motor/rotor assembly would be in the range from 125 to 144 Hz (6). This is well above the required value of 72 Hz, which may explain why the contactor operates so quietly.

When the rotors were first built, a small set screw (6-32 UNC, 1/8-in. [3.2-mm] long) in the rotor shaft was used to attach it to the motor shaft. Subsequently, a larger set screw (10-32 UNC, 1/8-in. [3.2-mm] long) was used to replace the smaller one because the rotor had occasionally come loose during operation. Because a larger hex-bar wrench is used with the larger screw, the rotor shaft can be secured more tightly. In addition, the set screw is partially screwed into the rotor-shaft cavity before assembly with the aid of a special drill-rod guide that is flat on one side. With the set screw in position, the motor shaft can only be inserted into the hole in the rotor shaft if the flat on the motor shaft is directly under the set screw. This facilitates contactor assembly.

In the design of the rotor, the less-dense-phase (lower) weir was chosen so that the total liquid volume in the rotor is only 6 mL. The more-dense-phase (upper) weir is slightly larger. Its exact value was chosen through a trial-and-error process. The appropriate upper-weir radius for a specific process would accommodate the range of densities expected for the two phases at all O/A flow ratios. In the old 2-cm contactors, the average mass transfer for uranium was 94%, with a range from 90 to 97%. Since the new minicontactor has a narrower Couette gap, its extraction efficiency should be even higher.

The new multistage minicontactor was built in banks of four stages. Three banks of the new minicontactor are shown in Fig. 3. Note how the inlet and outlet lines for each stage are fully accessible. Thus, stages can be allotted, as needed, to extraction, scrub, strip, and wash sections. The two guide posts for each motor make it easy to replace a motor/rotor assembly during remote-maintenance operations in a shielded-cell facility. They are at different heights so that the motor/rotor assembly

can be inserted in two steps. First, it is located on the high post. Then the assembly is moved around until the other post is located.

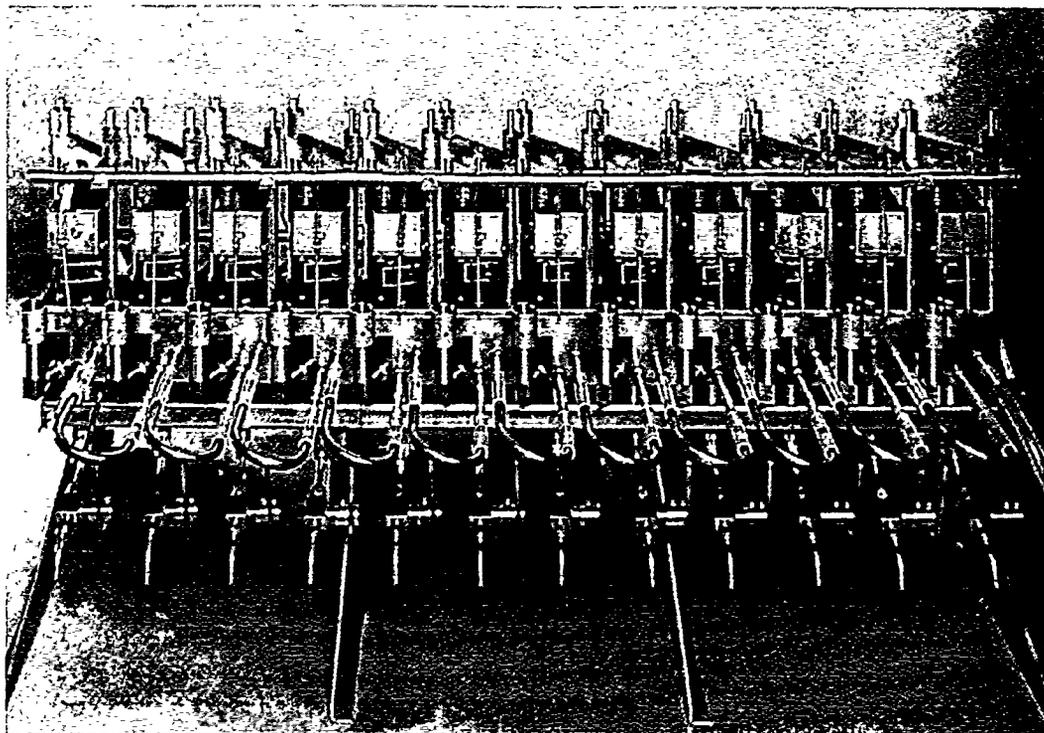


FIGURE 3. Twelve minicontactor stages.

The electrical plug for a stage goes into an electrical junction box that is just above the motor. The box has a recessed male plug that fits the female plug on the electrical cord. This design, which is used on most computer equipment, keeps electrically hot prongs from being exposed. It also eliminates any wires dangling from the motor/rotor assembly as it is being removed or reseated.

The purge-air manifold and the clips that hold it in place are now a part of the basic contactor design. This design feature keeps the manifold easily accessible but out of the way. The purge air flows down the motor shaft toward the rotor. The purge air extends motor life by keeping water vapor and acid fumes away from the motor.

When the contactor is being operated, the unit must be filled with the more-dense phase, typically the aqueous phase, before the less-dense phase is fed to the unit. A hot (radioactive) feed should only be introduced after both phases are exiting from their expected exit ports. Since (1) the volume of liquid in a contactor stage is 15 to 20 mL, including the liquid in the interstage lines (as noted above, the rotor volume contribution is 6 mL, that of the mixing zone is about 3 mL), and

(2) the maximum throughput (both phases) is 40 mL/min, a 20-stage minicontactor can reach steady state (based on three residence times through the unit) in less than 30 min.

APPLICATION TO SOLVENT EXTRACTION FLOWSHEETS

The new minicontactor has been used to test a variety of solvent extraction flowsheets. A number of these tests are reviewed here, with an emphasis on process and contactor problems encountered. This discussion includes (1) how the minicontactor was able to cope with process problems and (2) and what we did to fix contactor problems.

TRUEX

The first use of the minicontactor was for testing TRUEX flowsheets to verify the Generic TRUEX Model (GTM) (7). An overview of the GTM and the verification tests is given by Vandegrift et al. (8). The TRUEX solvent extraction process is used for removing transuranics from nuclear waste solutions containing a wide range of nitric acid and nitrate salt concentrations. In this process, a typical solvent contains a bifunctional extractant called octyl(phenyl)-N,N-diisobutylcarbamoylmethylphosphine oxide (CMPO) with tri-n-butyl phosphate (TBP) as a modifier and a hydrocarbon or chlorocarbon diluent (9). In the minicontactor tests, the TRUEX solvent consisted of 0.2M CMPO and 1.4M TBP in a hydrocarbon diluent of normal dodecane (nDD). Analysis of data from a single-stage test with nitric acid indicated that the mass-transfer efficiency for a stage was $95.0\% \pm 3.4\%$ (10).

Interstage flow

When the flow of liquids is low in the 2-cm contactor, the flow becomes intermittent in interstage lines that are not wetted by the flowing liquid. Such flow, which is controlled by the surface tension of the liquid, is a concern since it will reduce stage efficiency. On a qualitative basis, the longer the time between slugs of flowing liquid, the greater the reduction in stage efficiency. We use fluorinated ethylene propylene (FEP) tubing for the interstage lines. Because of its transparency, we can view the liquid flowing between the stages and determine if any liquid backup has occurred in the interstage lines. The organic phase wets the FEP tubing; therefore, it flows smoothly through the interstage lines. The aqueous phase does not wet the FEP tubing; therefore, it flows through the interstage lines intermittently.

To smooth out this flow, inserts were placed into the aqueous inlets at each stage. The normal inside diameter is 9.5 mm (3/8 in.) for the interstage lines and

7.9 mm (5/16 in.) for the inlet tubes. The opening at the end of the flow restrictor tube has a diameter of 1.0 mm and a length of 3.2 mm. The outlet from this restrictor is 0.8 mm from the surface of the spinning rotor. These flow restrictors did not solve the intermittent interstage flow. When they were used, liquid backups occurred in various interstage lines, especially the organic interstage lines, in an erratic fashion that was not reproducible. A contributing factor may have been one stage that had a large gap between the slinger ring on the rotor and the lip of the collector ring. Also, one motor had a bent shaft. To decrease the effects of the intermittent flow, the flow rate was not allowed to go below 2 mL/min. This was expected to keep the interval between slugs of flowing liquid to less than 1 min. By using this guideline, proper performance was obtained without the flow restrictors.

Interstage sampling

Six interstage sampling methods were tested and found to be usable for obtaining component concentrations. However, they have operational problems that limit their usefulness. The first method is to pump a liquid sample from an interstage line. However, the long residence time in the pump system means that the concentration in the interstage line is at steady state for a long time before the pumped sample reaches steady state. The second method is using a capillary tube and allows liquid to drip from the line. The drip system has the problem of the capillary tube clogging.

The third and fourth methods involve draining the more-dense phase from the bottom of the rotor stage. Either the pump system (method three) or the drip system (method four) can also be used. The same operational problems are experienced as with the first two methods. In addition, these two methods have the possibility of some of the less-dense phase accumulating with the more-dense phase.

The fifth method, similar to method three, employs a low-volume (3-mL) pump system to extract aqueous samples from the bottom of an operating stage at 0.33 mL/min (11). This method was used to collect samples for the TRUEX-SREX process described below. This system worked well for short periods. However, as soon as the process was upset for any reason, organic phase became entrained in the aqueous phase being sampled. When this occurred, the small-diameter (0.8-mm ID) Viton tubing of the sampling system swelled. Eventually, the Viton tubing closed completely and stopped the sampling process (12). Thus, this low-volume pump system is also of limited usefulness for stage sampling.

The sixth method, which we recommend for stage sampling, provides a quick shutdown when the contactor is at steady state. During a quick shutdown, all of the rotor motors and pumps are turned off at once. The two phases are drained from each stage and their volumes measured. Then, they are equilibrated by shaking.

Problems with the quick shutdown are that (1) pump flow may continue for 5 to 15 s, and (2) rotors do not stop turning for 10 to 40 s. As they turn, the rotors continue to pump liquid from stage to stage. Thus, stage volumes and concentrations will be partly a reflection of which rotors stopped first. However, measuring component concentration in stage samples gives a general understanding of the stage-to-stage concentration profile. The error in this analysis is estimated by comparing the stage samples to the external effluent in stages having an external effluent. Good values for the distribution ratios (D values) of the various components in the stage have been obtained from the equilibrated stage samples. Stage-to-stage concentration profiles have been reconstructed from these D values by using stage-to-stage calculational techniques, such as the Spreadsheet Algorithm for Stagewise Solvent Extraction (SASSE) given by Leonard (13).

TRUEX-SREX

The TRUEX process for removing transuranic elements from nuclear waste solutions was combined with the SREX process for removing strontium to create a new process, the TRUEX-SREX process (14,15). The combined TRUEX-SREX solvent contains (1) the TRUEX extractant, CMPO, (2) the SREX extractant, 4,4'(5)-di-t-butylcyclohexano-18-crown-6 (a crown ether) referred to here as CE, (3) a modifier to prevent third-phase formation, and (4) a hydrocarbon diluent. The hydrocarbon diluent used in the minicontactor tests of the TRUEX-SREX process was a high-purity, narrow-cut, isoparaffinic solvent called Isopar L (Exxon, Houston, TX). The solvent modifier was either diamyl amyolphosphonate (DAAP) or TBP. Various solvent compositions were evaluated using the dispersion number and process solvent 12 (PS 12) was chosen for the TRUEX-SREX process (16). The composition of PS 12 is 0.2M CMPO, 0.2M CE, and 1.2M DAAP in Isopar L. In the strip section, tetrahydrofuran-2,3,4,5-tetracarboxylic acid (THFTCA) was dissolved in water and used as the stripping agent. Since it was found that THFTCA works better when NaOH is added to form the mono sodium salt of THFTCA (MST) and some of the di sodium salt of THFTCA (DST), the strip feed has been 0.3M THFTCA with 0.4M NaOH, which can also be written as 0.2M MST and 0.1M DST. The various laboratory-scale tests of the TRUEX-SREX process with the minicontactor are discussed below.

Cold tests

Process solvent 12 was used in a series of five cold (nonradioactive) tests of the TRUEX-SREX flowsheet, four of them done in the minicontactor (12). It was found that PS 12 degraded with time. This degradation was inferred from color changes in the solvent, decreases in the dispersion number, and significant other-phase carryover in the aqueous raffinate (6%) and in the aqueous strip effluent (13 to

18%). Based on these results, the PS 12 solvent was reformulated and replaced by process solvent 15 (PS 15), which has a composition of 0.15M CMPO, 0.15M CE, and 0.12M TBP in Isopar L. Both solvents were developed by E. P. Horwitz and his group in the Chemistry Division at Argonne National Laboratory. The PS 15 solvent was used in the TRUEX-SREX tests with uranium and other radioactive materials.

Uranium tests

A series of three uranium tests in an eight-stage minicontactor demonstrated the superior performance of the PS 15 solvent (12). At the same time, the new stripping agent, consisting of 0.3M THFTCA and 0.4M NaOH, was evaluated at three O/A flow ratios (0.33, 0.67, and 1.25) in the strip section. These tests showed that as the O/A flow ratio decreased, the material balance for the various components went from essentially full recovery (79 to 107%) to a limited recovery (49 to 88%) for the lowest O/A flow ratio. At this low O/A flow ratio, the nitric acid in the organic phase entering the strip section neutralizes all the NaOH in the strip feed. At high NaOH concentrations, the THFTCA stays mainly in the aqueous phase as MST and DST. At low NaOH concentrations or no NaOH, the THFTCA stays mainly in the organic phase. Thus, at an O/A flow ratio of 0.33, the THFTCA was pinched in the strip section. This 8-stage test was run for 4.5 residence times, based on a total flow rate (both aqueous and organic phases) in the 4-stage strip section of 16 mL/min. The hydraulic performance during this test was good up until a residence time of 2.2. At that time, high liquid levels were observed in the interstage lines feeding the last strip stage (where the strip feed was being introduced) and the first stage of the two-stage carbonate-wash section. At 4.0 residence times, high interstage liquid levels were also seen in the next-to-last strip stage. If the run had been continued, it is likely that the strip section would have become completely inoperable. For the higher O/A flow ratios in the strip section (0.67 and 1.25), the operation of the contactor and the process was acceptable based on (1) good material balances for the various feed components and (2) hydraulic performance.

Hot tests

Based on these three uranium tests, an O/A flow ratio of 1.0 was chosen for the strip section of the hot (radioactive) test of the TRUEX-SREX flowsheet in a 24-stage minicontactor (17). This process had three scrub sections: an oxalate scrub to keep zirconium out of the strip section, an aluminum scrub to keep oxalate out of the strip section, and a low-acid scrub to keep nitric acid out of the strip section.

The overall process worked well with good hydraulic performance. However, there were three problems. First, the decontamination factor for several elements (Eu, Np, and Sr) in the extraction section was lower than expected. This may be due to the

formation of a second organic phase there. It is not known whether this phase was present during the test or only formed after the test was finished. Second, a material balance showed that most of the bismuth in the process feed was lost in the contactor. This loss appears to have occurred at the oxalate feed stage. Third, a precipitate was found on the wall of that rotor after the test. The precipitate was mostly bismuth oxalate, which confirms the low material balance for bismuth. Thus, while the stage-to-stage concentration profiles of many components showed good agreement between the GTM values and the experimental results, some components showed significant differences. Overall, the hot minicontactor test showed that, while the TRUEX-SREX process works well, some additional work is needed before it will be fully operational.

SREX-NEPEX

The SREX process for removing strontium from nuclear waste solutions was combined with the NEPEX process (where NEPEX is short for neptunium-plutonium extraction) to separate Sr, Np, and Pu from the lanthanides (Ln), Am, and Ba. The extractant for the NEPEX process is TBP in a hydrocarbon diluent. Two minicontactor tests were made of the SREX-NEPEX process. After the adverse results of the first test, a new solvent with a new diluent was used for the second test.

Isopar-L diluent

In the first minicontactor test of the SREX-NEPEX process, the process solvent (PS 16) had a composition of 0.15M CE and 1.2M TBP in Isopar L (17). The flow ratios for the extraction and first scrub sections were chosen carefully, and the first scrub section was a high-acid scrub designed to obtain a good separation of strontium from barium. The second scrub section, a low-acid scrub, prepared the solvent for the THFTCA/NaOH strip section.

Initially, the hydraulic performance of the 20-stage minicontactor was very good, with no appreciable backup of liquid in any interstage line. At 3.5 residence times (one residence time was about 10 min) into the run, which ran for a total of 12.3 residence times, a resistance to flow was noted at stage 11, the first stage that the solvent enters in the low-acid scrub section. This observation is based on a high liquid level for both phases in the interstage lines feeding this stage. At 6 residence times, the backup of liquid in the organic interstage lines had extended to stage 10, the last stage of the high-acid scrub section; at 10 residence times, the backup extended to stage 5, the aqueous feed stage for the extraction section; at the end of the test, the backup was all the way to stage 2. At 8.1 residence times, the backup of liquid in both the organic and aqueous interstage lines had extended from stage 11 to stage 12, the second stage in the low-acid scrub section consisting of four stages; at

9.8 residence times, it had extended to stage 13, the third stage in the low-acid scrub section.

After the run, a white sticky precipitate, which was a precipitate of strontium and the crown ether (referred to here as Sr/CE), was found in stages 11 and 12. In stage 11, the precipitate was on the outside of the rotor, on the inside of the rotor, and in the bottom of the contactor housing up to the top of the bottom vanes. In stage 12, the precipitate was on the outside of the rotor from the bottom to about half way up the side of the rotor. The amount of precipitate in stage 11 was great enough that this rotor was no longer able to turn. This rotor probably stopped turning during the test; however, the stopped rotor was not discovered until we tried to flush out the contactors after the test. The Sr/CE precipitate was removed from stages 11 and 12 by dissolving it with ethanol.

A sudden drop in the organic effluent flow rate indicated that the stage-11 rotor had stopped turning between 5 and 7 residence times into the test. The higher, but less than steady state, flow rates of the organic effluent at later times suggest that, with sufficient backup of liquid, some organic phase was forced through stage 11. However, for all practical purposes, process operation in the minicontactor stopped when the rotor in stage 11 stopped turning.

In reviewing the test, we learned that the Sr/CE precipitate had been observed in test-tube equilibrations, but only after the tube had sat for 24 to 48 h. In the contactor, this precipitate formed in less than 0.5 h. Since a precipitate was never seen in the test tube equilibrations when 1-octanol was used as the diluent, 1-octanol was chosen as the diluent for the second SREX-NEPEX test.

1-Octanol diluent

In the second minicontactor test, the process solvent (PS 19) had a composition of 0.10M CE in 1-octanol (17). Because this solvent has neither CMPO nor TBP, it will not extract Np or Pu. Thus, this solvent is really a SREX solvent. The process flowsheet for this SREX test is shown in Fig. 4.

The hydraulic performance of the 24-stage minicontactor was very good throughout this test. When the aqueous flow was high, 20 mL/min or higher, the aqueous interstage flows were continuous. At lower aqueous flows, 2 to 8 mL/min, the flow was intermittent, that is, the liquid flowed as discrete slugs of liquid in the interstage lines. Solvent recycle was started at 15 residence times (one residence time was about 12 min) into the test. The test ended at 20 residence times.

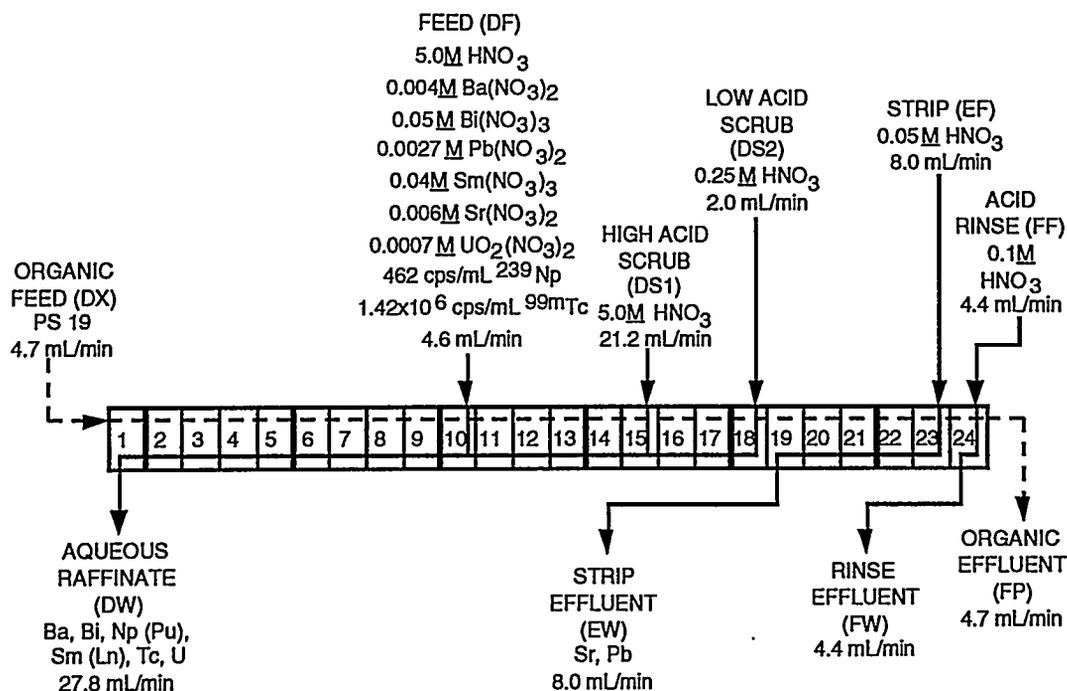


FIGURE 4. Flowsheet for SREX test.

Analysis of effluent compositions showed that this test had met its basic objective, that is, strontium had clearly been separated from barium. However, the decontamination factor for strontium in the 10-stage extraction section was 200, not the 10^5 that had been expected based on 100% stage efficiency. The concentration profile for strontium in the extraction section indicates that the stage efficiency for strontium in the extraction section was only 60%.

Analysis of the aqueous and organic effluents showed that most neptunium ended up in the aqueous raffinate from the extraction section. After the test, when the stage samples were equilibrated, most neptunium was found in the organic phase. The neptunium distribution ratios ranged from 100 to 7 in the extraction and first (high-acid) scrub sections, from 3.5 to 0.6 in the second (low-acid) scrub section, and from 0.2 to 0.6 in the strip section. Thus, based on an analysis of the stage samples, most of the neptunium should have appeared in the aqueous strip effluent, not the aqueous raffinate. This suggests that the oxidation state of neptunium changed in the 2 to 3 h after the test, before the two phases from each stage were equilibrated. The chemical changes that could have caused this are discussed further by Leonard et al. (17). Overall, the neptunium results were unexpected.

For the other components that were analyzed (Ba, Tc, and HNO₃) and for strontium outside of the extraction section, a stage efficiency of 100% works well when experimental results are compared with stage-to-stage values generated using

SASSE. This comparison is shown in Fig. 5 for Ba and Sr, Fig. 6 for Tc, and Fig. 7 for HNO_3 .

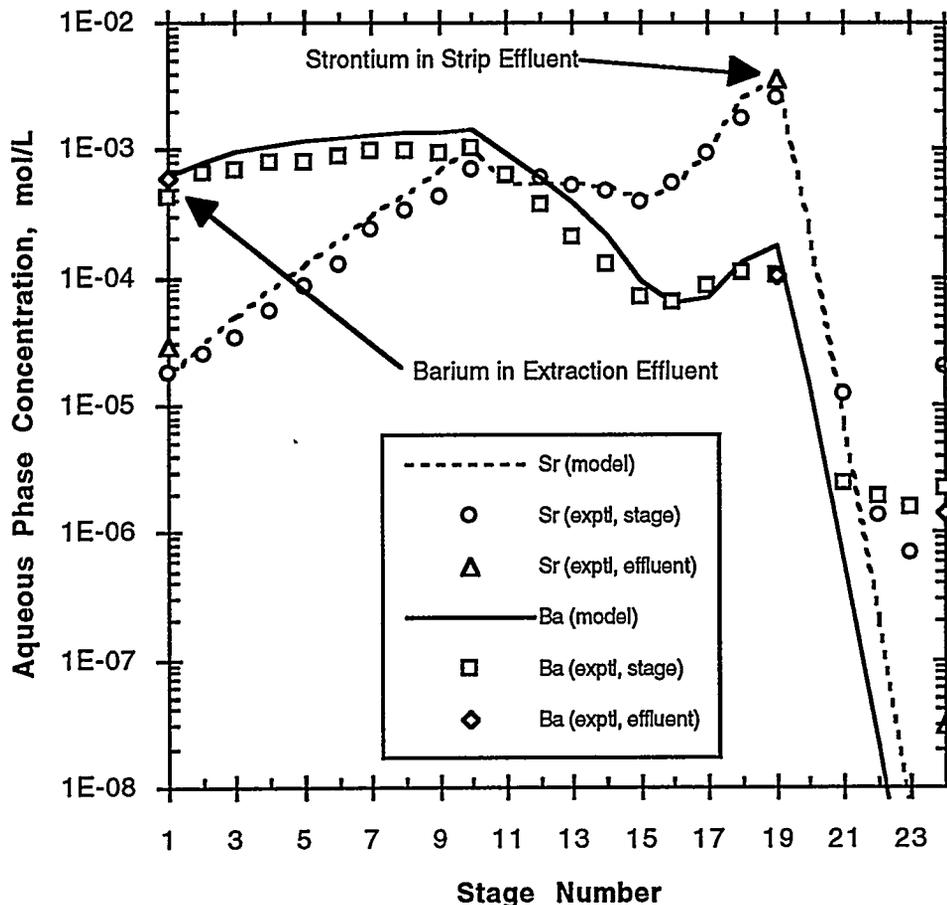


FIGURE 5. Concentration profile for Ba and Sr at the end of the SREX test. The SASSE model assumed that the strontium extraction efficiency was 60% in the extraction section.

Since the strontium stage efficiency was also low in the extraction section of the hot TRUEX-SREX test, it appears that the residence time in the mixing zone of a contactor stage, about 6 s for these tests, is too short. Batch tests in a test tube, which mix the two phases for 60 s, show good extraction efficiency. It may be that strontium extraction by a crown ether is slower than has been observed for CMPO or TBP with other metal ions. These two extractants typically have 95 to 100% extraction efficiency in centrifugal contactors. Even if strontium extraction with crown ether is slow, the centrifugal contactor can still be used if (1) it is redesigned to

increase the residence time in the mixing zone, or (2) more extraction stages are added. For the SREX test discussed here, 12 to 14 more extraction stages would be required.

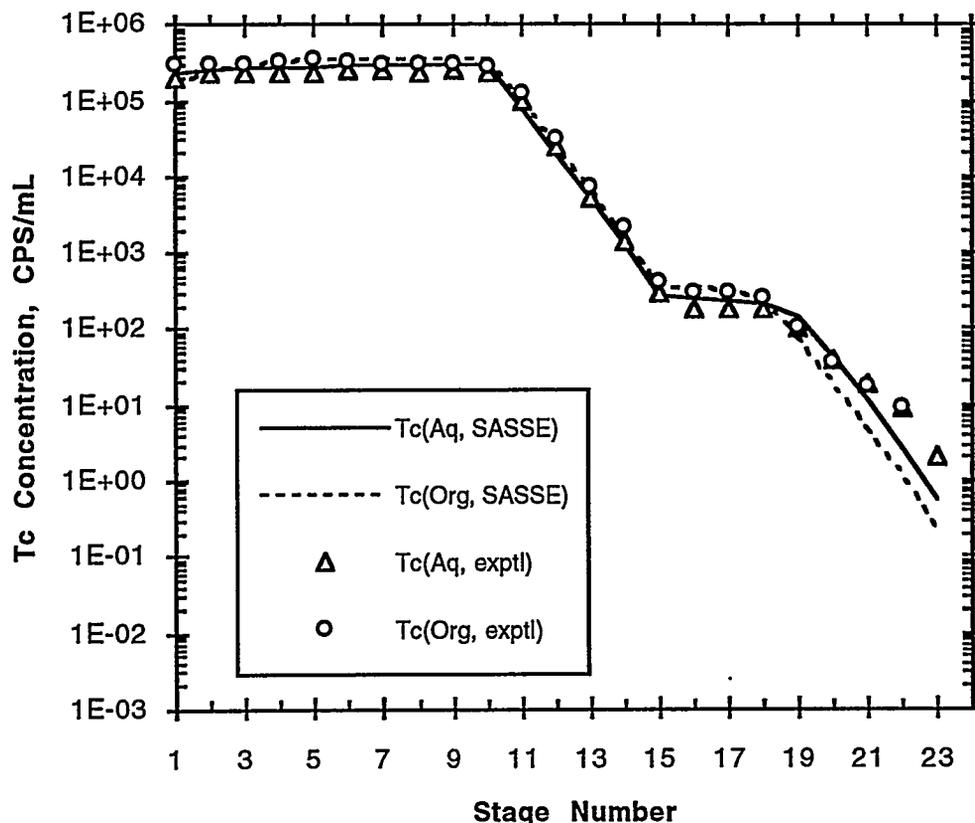


FIGURE 6. Concentration profile for technetium at the end of the SREX test.

COMPARISON WITH 4-cm CONTACTOR

In comparing the minicontactor with the 4-cm contactor, we concluded that the 4-cm contactor has two clear advantages: (1) a higher throughput, 400 mL/min vs. 40 mL/min, and (2) less effect of surface tension on the interstage flow. The 2-cm contactor also has two clear advantages: (1) less feed volume, 1 L vs. 10 L, and (2) less space needed for the unit itself. In several ways, both units exhibit no differences; both have (1) easy access to interstage lines for combining external feeds with interstage flows and for assigning stages as required to the various sections, (2) 95%

extraction efficiency for most elements and extractants, and (3) short residence times, 10 to 30 s per stage, when operating near maximum throughput.

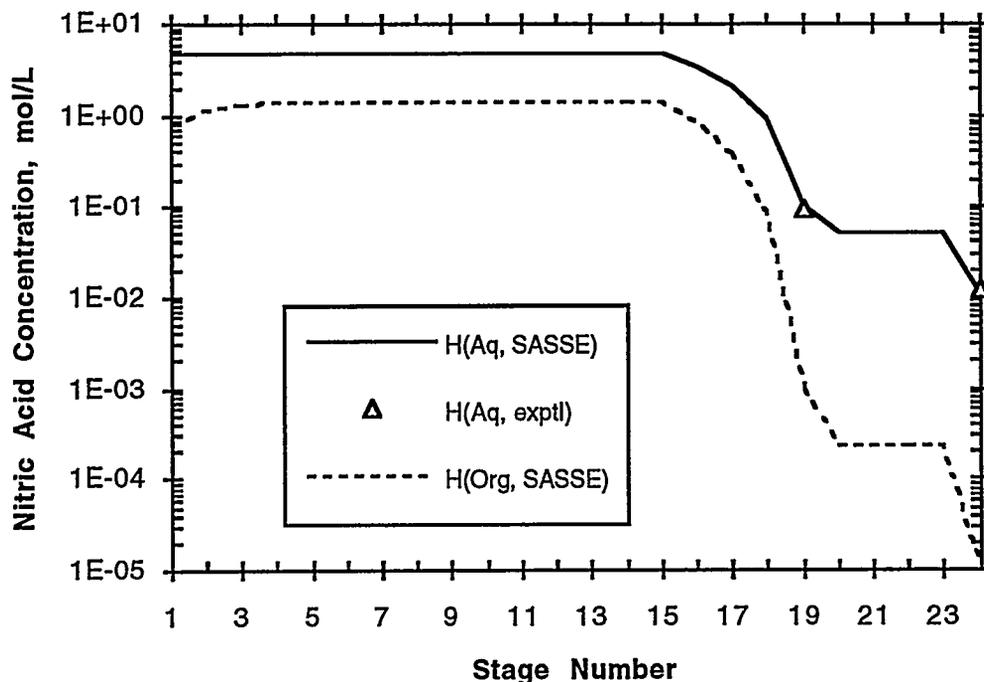


FIGURE 7. Concentration profile for HNO_3 at the end of the SREX test.

If one needs to process a large quantity of solution in the laboratory, then the 4-cm unit is clearly the right choice. For most other cases, the minicontactor is recommended because the difference in feed volumes can be important in at least eight ways. (1) The real or simulated waste feed may be difficult to obtain or costly to prepare. (2) The other aqueous and organic feeds are also reduced by a factor of 10 and, therefore, take up less space. (3) The effluent containers also take less space. (4) Less waste is generated, both radioactive and nonradioactive. (5) Radiation levels are reduced by a factor of 10. This is especially important when tests are being done in a hood or glovebox. (6) If a new extractant, which is still expensive, is being tested, the cost savings can be significant. (7) If the new extractant is only available in limited quantities, one might not be able to obtain enough material for a 4-cm contactor test. (8) Because of the difference in throughputs, for the same volume of feed, the 2-cm contactor can complete a run that is ten times as long.

CONCLUSIONS

With the design, building, and operation of the new 2-cm centrifugal contactor, we have demonstrated that a minicontactor can be used for testing solvent extraction processes at various O/A flow ratios with total throughputs (both phases) up to 40 mL/min. In running the minicontactor with a variety of solvent extraction flowsheets, we have been able to identify problems such as poor solvent quality, precipitate formation, third-phase formation, changes in distribution ratio, and low extraction efficiency. Thus, the minicontactor is a valuable tool in the development of solvent extraction processes since it allows one to identify and address potential plant-scale problems at a very early stage.

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