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ENTRAINMENT OF SOLVENT IN AQUEOUS STREAM FROM CINC V-5 CONTACTOR

S. D. Fink
M. L. Restivo
T. B. Peters
M. D. Fowley
D. B. Burns
W. M. Smith, Jr.
F. F. Fondeur
S. L. Crump
M. A. Norato
D. T. Herman
C. A. Nash

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Waste Processing Technology Section
Savannah River National Laboratory
Aiken, SC 29808

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EXECUTIVE SUMMARY

Personnel completed a rapid study of organic entrainment during operation of a CINC V-5 contactor under prototypical conditions covering the range of expected MCU operation. The study only considered the entrainment of organic into the strip acid effluent destined for the Defense Waste Processing Facility. Based on this work, the following observations are noted.

- Concentrations of total organic from the contactor discharge, based upon modifier measurements, in the acid typically averaged 330 ppm_m, for a range to 190 – 610 ppm_m.
- Entrained droplet sizes remained below 18 microns for samples collected at the decanter outlet and below 11 microns for samples taken from the contactor discharge.
- Scouting tests showed that a vendor coalescer material promotes coalescence of smaller size droplets from the decanter effluent.
- Personnel observed a previously unreported organic impurity in the solvent used for this study. Additional efforts are needed to ascertain the source of the impurity and its implication on the overall process.
- Process throughputs and planned operating conditions result in very stable hydraulics, suggesting that the MCU stripping stages will have spare operating capacity.
- The V-5 contactors show operated with relatively cool surfaces under the planned operating conditions.
- If operating conditions result in an imbalance of the relative mixing and separation conditions within the contactor, a very stable emulsion may result. In this instance, the emulsion remained stable for weeks. The imbalance in this study resulted from use of improperly sized weir plates.
- Personnel demonstrated an effective means of recovering emulsified solvent following a non-optimal equipment configuration. The protocols developed may offer benefit for MCU and SWPF operations.
- This study developed and demonstrated the effectiveness of several analytical methods for support of the Caustic-Side Solvent Extraction process including infrared spectroscopy and droplet size measurement by a MicroTrac™ S3000. Interfacial tension measurements also showed sensitivity to purity of the solvent suggesting that this technique may prove valuable for future process diagnostics. The study highlighted limitations of the current gas chromatography configuration for determination of modifier content of samples.

Additional development of analytical methods for determining composition – and particularly – modifier content – of organic and mixed aqueous-organic samples is warranted. Infrared spectroscopy shows particular promise.

Additional full-scale studies are warranted to investigate the entrainment of organic in the aqueous effluent from the extraction operation. Since waste composition may differ appreciably for the process, this stream may exhibit much wider variance in hydraulic behavior, organic entrainment, and may pose a greater risk for poor hydraulics.

Conducting contactor studies at SRNL allowed numerous personnel to view operations and facilitated the training of staff members. Members of the Design Authority, Engineering, and Training groups benefited from tours. Consideration should be given to procuring and installing a full-scale contactor at SRNL for future support and for assistance during commissioning of the MCU.

Additional study of surface and interfacial tension is recommended. This tool may also offer economical and rapid process diagnostics for future operations.

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ADS	Analytical Development Section
ASTM	American Society for Testing and Materials
BOBCalixC6	calix[4]arene-bis(t-octylbenzo-crown-6)
CINC	Costner Industries of Nevada Corporation
Cs	cesium
CSSX	Caustic-Side Solvent Extraction
Cs-7SB	1-(2,2,3,3-tetraflouropropoxy)-3-(4-sec-butylphenoxy)-2-propanol, also known as modifier, CAS #308362-88-1
DOE	Department of Energy
DSS	decontaminated salt solution
DWPF	Defense Waste Processing Facility
EDL	Engineering Development Laboratory
GC/MS	Gas Chromatograph – Mass Spectroscopy
gph	gallons per hour
gpm	gallons per minute
HLW	High Level Waste
HPLC	High Performance Liquid Chromatography
ICP-ES	inductively coupled plasma emission spectroscopy
Isopar® L	hydrotreated heavy naphtha, CAS #7133465-00
MCU	Modular Caustic-Side Solvent Extraction Unit
NTU	nephelometric turbidity units
ORNL	Oak Ridge National Laboratory
ppm_m	parts per million on a mass basis
ppm_v	parts per million on a volume basis
SE	strip effluent
SPF	Saltstone Production Facility
SRNL	Savannah River National Laboratory
SVOA	semi-volatile organic analysis
SWPF	Salt Waste Processing Facility
TOA	trioctylamine-CAS #1116-76-3
WPTS	Waste Processing Technology Section
WSRC	Westinghouse Savannah River Company
VFD	variable frequency drive

INTRODUCTION AND BACKGROUND

The Modular Caustic-Side Solvent Extraction (CSSX) Unit (MCU) and the Salt Waste Processing Facility (SWPF) will remove radioactive cesium from Savannah River Site high-level wastes using an organic solvent system. Both designs include decanters while the SWPF also includes coalescers to recover suspended solvent droplets from the decontaminated salt solution (DSS) and strip effluent (SE) streams. The project conceptual designs limit organic solvent entrainment in these streams to <50 ppm_v.

The basis for the 50 ppm_v value is not well documented. Personnel chose this value chiefly from an economic evaluation. Prior demonstrations of the solvent extraction process with actual waste resulted in entrainment values in the range of 0.002 0.051 vol % (or 20 – 510 ppm_v).[1,2,3] Values in larger scale testing using simulated waste proved higher typically as ~0.012 vol % but reaching as much as 0.33 vol % for the organic in the decontaminated waste stream.[5] These findings – as well as parallel testing on simulated wastes – identified the need for additional mitigation steps to recover the valuable solvent. Limited studies examined the use of a coalescer and other means for this purpose.[4,5] Those preliminary tests suggest that the process could achieve a 10-fold reduction in organic content of the aqueous stream after treatment using a commercial coalescer.

Subsequent pilot testing of the integrated process using simulated waste resulted in higher than anticipated entrainment of organic with limited effectiveness of the coalescer configuration.[6] The scouting tests also suggested smaller droplet size of the entrained organic and foaming of the solvent may have contributed to the higher than anticipated entrainment. However, the scouting work is reported as not sufficiently designed to reflect final process performance. The study indicated a need for future testing.

Subsequent review of the prior test data resulted in a realization that the droplet size data reported in earlier studies appeared flawed and unreliable. SRNL rapidly conducted a series of scoping studies to examine the droplet size of solvent within the two aqueous streams using photo microscopy and laser particle sizing methods. Those studies indicate a very high fraction of the organic present in droplets well under 50 micron in diameter.

Recently, the MCU project conducted an evaluation of the facility design configuration to establish waste acceptance limits for solvent carryover in aqueous process streams going to the Defense Waste Processing Facility (DWPF), Saltstone Production Facility (SPF), and Tank 50H. This evaluation identified organic concentration limits lower than the conceptual design bases for MCU and SWPF (i.e., significantly below 50 ppm_v).

To resolve these concerns, management charted an integrated team to define mitigation options. Part of that overall effort required collection of information on the concentration and size of organic droplets in the effluent streams from contactors and from the downstream decanters. This report describes the results of experiments using a single centrifugal contactor of near-prototypical design for the strip section of the MCU process. The study used a V-5 contactor (i.e., 5-inch rotor) model from Costner Industries of Nevada Corporation (CINC). A parallel study examined organic entrainment in aqueous effluents from both the strip and extraction stages using 2-cm centrifugal contactors.[7]

APPROACH

Equipment Configuration

Testing used a CINC V-5 centrifugal contactor obtained on loan from the Parsons-SRS. Personnel assembled the contactor and associated decanter in the 786-A Engineering Development Laboratory (EDL) at SRNL in a vacant fume hood. Below is a picture of the installation. This location mitigated flammability and combustibility risks. [The solvent is listed as a Group D, Division 2 hazard per NEC Code requirements, chapter 5.] SRNL replaced the as received motor on the contactor with an identical but explosion proof model to meet NFPA requirements. This motor was removed upon return of the contactor to Parsons-SRS. All materials contacting the MCU solvent were limited to glass, stainless steel, and Teflon™ to avoid contamination of the solvent by leaching of plasticizers from plastic materials. Appendix A contains additional details for the specifics of the equipment. Included are process schematics and equipment layouts. The calibration curves are provided in the laboratory notebook.[12]

Figure 1. Installed Contactor and Decanter Equipment.

Analytical Methods

The analytical approach for entrained organic parallels in part that of the concurrent study using 2-cm centrifugal contactors.[7] Personnel collected samples either directly from the contactor outlet or from the sampling line on the decanter tank. Analysis used gas chromatography coupled with mass spectrometry (GC/MS).[8] Analytical separations occurred on an Agilent 6890 gas chromatograph, equipped with a 30 m DB-XLB (Part No. 200-0012) column, with 0.18 mm diameter and 0.25 μm film thickness. Quantitation used a Hewlett Packard 5973 mass selective detector. Personnel confirmed the mass spectrometer tuning within 24 hours prior to each measurement using perfluorotributylamine. The claimed accuracy of the Isopar® L determinations – based on standards – is ±10%. Analyses focused primarily on Isopar® L content – i.e., the flammable organic constituent – but personnel also analyzed selected samples for triocytlamine (TOA) and modifier as time allowed. Personnel sealed these samples in glass vials with Teflon™ lined caps to avoid evaporation of the volatile organic. Personnel filled the vials nearly full to minimize losses to the vapor space of the vials.

For each test, personnel collected at least one sample from the decanter discharge and held that sample for 2 hours per the protocol established in the parallel study. Personnel then immediately analyzed that sample via MicroTrac™ S3000 for droplet size information.[9]

Personnel made turbidity measurements on selected samples immediately after collection. They verified the operational reliability of the meter before and after measurements by checking the response to a 0 and a 40.0 NTU standard. All sample measurements fell between these values. Two obvious outliers occurred in the initial measurement of the 40.0 NTU standard although the paired, final analysis of the standard agreed well with expectations. Without removing the outliers, we determined a standard deviation of 15.2% for the 40.0 NTU standard measurements. Excluding the outliers, the standard deviation is reported as 0.9 NTU (or 2.3% of full scale).

Solvent Preparation

Parsons-SRS – the Engineering, Procurement, and Construction Contractor for the SWPF – loaned solvent to SRNL for the testing. The solvent had been used in earlier pilot testing[6] and contained excess Isopar® L. SRNL purified the solvent by rotary evaporation – see details in Appendix B – to return the composition to within the range typical for the process. During this process, personnel also identified the presence of a limited volume of entrained caustic as well as extensive solids. The source of the solids is unknown. Personnel decanted the second liquid phase and filtered the solids. Full details are provided in the appendix.

The solvent upon receipt had a distinctly yellow color, more extreme than seen in prior testing at SRNL. Personnel performed analyses of the as-received solvent and identified the presence of an organic contaminant in trace amounts. The contaminant does not match degradation by-products previously reported for the solvent. Gas chromatography - mass spectrometry did not detect the impurity, suggesting a concentration less than 10 mg/L. Fourier Transform Infrared spectroscopy – with attenuated total reflectance – of the solvent did identify the chemical nature of the impurity. Details are provided in the appendix.

In addition to the distinct discoloration, the solvent appeared to foam more than expected when stirred. Also, the prior test report[6] mentions a pronounced foaming behavior. Since the current testing investigates interfacial behavior – i.e., organic entrainment – as well as hydraulics, the authors decided to also perform surface tension and interfacial tension measurements of the solvent. The resulting values – see Appendix A – differ slightly from literature data for this solvent as well as from the parallel measurements for a control sample of fully-qualified solvent. However, the offset proved minimal. Hence, the authors do not believe the presence of the impurity significantly alters the findings from this study.

Experimental Design

Personnel designed a test matrix to examine organic entrainment as a function of process flow rates and rotation speed of the contactor. The matrix used the prior information from pilot testing[6] as a reference point. That prior testing indicated the contactor could achieve full nominal throughput at ~75% of maximum rotation speed (i.e., 2700 rpm). Hence, the MCU operation uses 2700 rpm as the nominal operating condition. The nominal MCU aqueous and organic flow rates – 0.36 gpm strip acid and 1.8 gpm organic – were provided by Seth Campbell of the Design Authority. Note that these combined flows fall well short of the nominal throughput capacity of the V-5 contactor but represent the nominal flows supported by the integrated design of the Actinide Removal Process and the MCU. The maximum flows through the MCU strip contactors are at or below 75% of the nominal contactor throughput advertised by CINC.

Table 1 provides the initial experimental design. The test matrix provided for the contingency of unstable hydraulic operation in the event that foaming proved problematic as reported in the earlier pilot test study. Tests 1-3 examine the impact of rotational speed on organic entrainment at nominal flow rates and concentration factor[1]. Tests 2B and 3B were contingency tests in the event that stable hydraulics did not occur at the fastest (i.e., 3100 rpm) and slowest (i.e., 2300 rpm) values examined. Since stable hydraulics did occur, personnel did not conduct tests 2B or 3B.

Table 1. Experiment Design for Hydraulic Testing

Test ID	Aqueous Flow Rate		Organic Flow Rate gpm	Contactor Speed (rpm)
	gpm	Gph		
Concentration Factor of 15				
1	0.36	22.5	1.8	2700
2A	0.36	22.5	1.8	3100
2B	0.36	22.5	1.8	2900
3A	0.36	22.5	1.8	2300
3B	0.36	22.5	1.8	2500
4	0.55	34.1	2.75	2700
5	0.27	17.1	1.33	2700
Concentration Factor of 12 (contingent upon failure to obtain higher value)				
6	0.69	42.6	2.75	2700
7	0.33	20.7	1.33	2700
Mitigation method: air flotation (contingent upon selection by Systems Engineering Team)				
8	0.36	22.5	1.8	2700
Mitigation method: coalescer material (contingent upon selection by Systems Engineering Team)				
9	0.36	22.5	1.8	2700

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[1] Concentration factor refers to the overall process volumetric reduction between the entering aqueous waste and the final effluent aqueous from cesium stripping operations. The design target value is 15 although smaller-scale laboratory demonstrations with actual waste using 2-cm centrifugal contactors did not always obtain that value.

Tests 1, 4 and 5 examine the impact of flow rates on organic entrainment at nominal rotational speed. The extreme values represent the maximum expected range of operations based on integrated operation of the Actinide Removal Process and the MCU.

Tests 6 and 7 represent contingent experiments at lower concentration factor (of 12) assuming stable hydraulic operation did not occur at nominal process values. Since stable hydraulics did occur, personnel did not conduct these tests.

Tests 8 and 9 address potential design mitigation strategies to reduce organic entrainment. A parallel systems engineering assessment[10] -- by the "Systems Engineering Team" – of options eliminated air flotation from consideration prior to reaching this step in the experimental matrix. Hence, personnel omitted Test 8. Test 9 allowed for initial assessment of coalescer material should the parallel systems engineering assessment define an acceptable material in time for testing. Personnel obtained three media very late in the testing period and inserted the most promising of the candidate media for testing. Two quick trials occurred.

Personnel set the duration of each test as stable operation for three decanter volumes of flow – i.e., 120 gal on strip acid. During the initial tests, personnel collected beakers of the organic and aqueous discharges from the contactor and observed visually for clarity. Typically, personnel photographed these samples prior to returning to the system. After three decanter turnovers, personnel collected a sample from the discharge of the decanter (i.e., aqueous recycle feed to the contactor). This sample received analysis for organic content by GC/MS, visual observation by photographic microscopy, entrained droplet size distribution by MicroTrac™ S3000, and turbidity. Turbidity measurements used an Orbeco-Hellige Model 965 turbidimeter calibrated and operated using a WPTS procedure.[11] The sample handling and analytical protocols are described in the parallel report for the 2-cm contactor testing.[7] MicroTrac™ S3000 analysis occurred two hours after sample collection such that the decant time for that sample represents the decanter residence time plus 2 hours. This time interval is consistent with that used in the 2-cm contactor testing.

As testing proceeded, personnel noted the concern that operating under recycle of the strip acid through sequential tests would cause a process bias for the analytical data. To help address this concern in later tests, personnel started collecting samples directly from the contactor discharge for analysis of organic content as a function of operating time. [Theses samples are in addition to the decanter discharge sample at the end of the test.] Also, after three tests, personnel replaced the acid in the system.

RESULTS

Initial Operation – Isopar® L Trial

Initial operations consisted of a brief trial (~15 minutes) using Isopar® L and dilute nitric acid (0.001 M)[2]. [Additional details of operation are recorded in a laboratory notebook.[12]] This initial trial and all routine contactor operations followed an operating procedure.[13] Personnel added 20.3 kg of Isopar® L to the Solvent Receipt Tank, transferred it to the Solvent Feed Tank, and added 40 gallons of acid to the decanter. They heated the system to operating temperature and then established flows to the contactor. The contactor operated stably with no gross entrainment of organic into the aqueous decanter. A small amount of aqueous phase – estimated by visual observation as ~100 mL – did transfer to the Solvent Receipt Tank and collected as a separate phase around the bottom of the Solvent Receipt Tank which has a rim along the outer wall. Personnel did not observe any significant foaming or frothing. Personnel emptied the solvent tanks and the contactor in preparation for the first experiment.

Initial Operational Upset – Emulsion and Recovery

Personnel transferred 46.6 ± 0.3 lb_m (~6.57 gal) of solvent to the Solvent Receipt Tank and then to the Solvent Feed Tank. They established aqueous flow to the contactor at the target rate (0.36 gpm). Approximately 4 minutes after starting organic flow to the contactor, personnel observed overflow into the decanter with a few, small organic droplets apparent immediately on the aqueous surface. They proceeded to stabilize the organic flow rate to the target value of 1.8 gpm. Within several minutes, the appearance of the decanter contents turned to a milky white color (see Figure 2). Personnel continued operation and requested consultation from the activity manager. The manager reported to the scene approximately 15 minutes after start of organic flow. By that time, the solvent as observed in the rotameter sight glass had also taken on the milky white appearance. No solvent remained in the Solvent Receipt Tank. Personnel halted operations.

Sampling and observation of the contents of the various vessels determined that the entire solvent inventory had emulsified in the aqueous. The emulsion proved extremely stable; the phases failed to separate even after weeks of storage. SRNL personnel began numerous efforts to investigate means to recover the solvent from the emulsion. Appendix C discusses these efforts.

Consultation with the technical representative from CINC and with personnel from Parsons-SRS led to the discovery that the contactor configuration included the aqueous weir plate (2.8 inch diameter opening) appropriate for extraction operation rather than stripping (2.45 inch). This configuration led to excessive mixing with little separation within the contactor. Also, the vendor representative commented that the subsurface delivery of the aqueous effluent to the decanter tank may also have contributed to inappropriate hydraulics.

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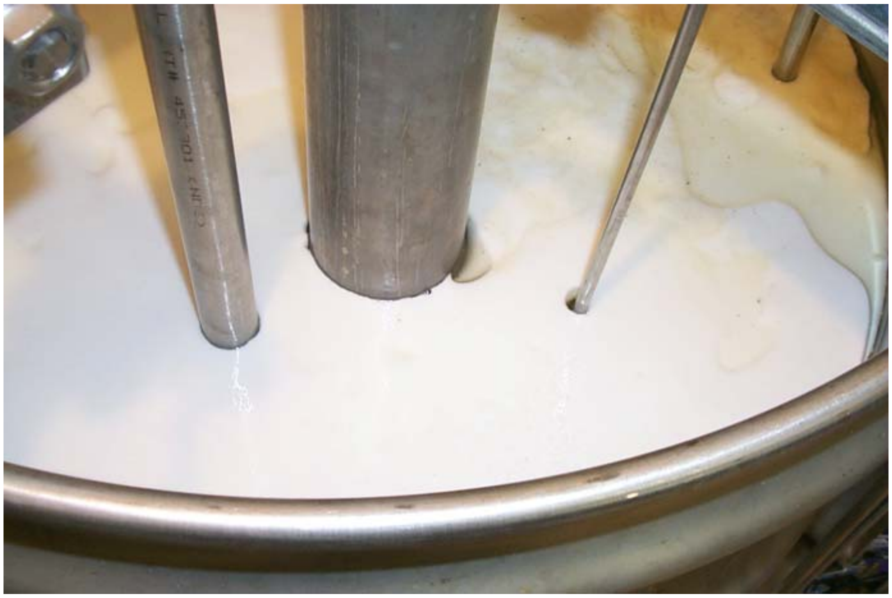
[2] Personnel periodically measured the pH of the acid throughout testing and added minor amounts of fresh acid when needed to maintain the target value. Most measurements slightly exceed a pH of 3. At the completion of all testing, the pH of two drums of acid measured 3.45 and 3.47, respectively.

Figure 2. Photograph of Decanter Contents after Initial Operational Upset.



Parsons-SRS personnel rapidly (i.e., ~ 2days) shipped the appropriate replacement weir. In parallel, SRNL personnel reconfigured the piping to avoid subsurface discharges and to add vent lines to the aqueous effluent piping.

Testing by SRNL personnel indicated that centrifuging emulsion samples for long durations (i.e., 20 minutes at 1500 rpm) successfully separated the phases. With this in mind, personnel reconfigured the contactor – with the 2.45 inch weir installed – for feed only from the Solvent Feed Tank. By feeding only from the Solvent Feed Tank under gravity flow and running under very low rate, personnel achieved a residence time of ~20 minutes inside the contactor. The low flow rates (~0.16 gpm) fell below the rotameter scale; hence, personnel verified the flow rate by measuring the volume collected in a beaker over a time period. Personnel operated the contactor at rotor speed of 2800 rpm.

Personnel first processed the emulsion drained earlier from the contactor and obtained a relatively pure organic phase in the Solvent Receipt Tank. Personnel removed this solvent from the system and when the operating volume in the Solvent Feed Tank dropped appreciably, added batches of aqueous from the upper layers of the decanter to the Feed Tank. By adding sequential batches of aqueous from the upper section of the decanter and removing solvent as it collected in the Solvent Receipt Tank, personnel recovered ~5.95 gal – calculated volume – over a period of 7 hours. Figure 3 shows a photograph of one of two pails containing the recovered solvent. This represented a recovery of ~90.5%. Additional recovery efforts continued in parallel.

Figure 3. Photograph of Recovered Solvent

Experiment Sequence and Sampling Plan

After recovering the solvent from the initial upset, personnel prepared fresh strip acid and resumed operation per the defined test matrix. Test protocol required personnel to achieve stable operating conditions and operate through 3 decanter volume turnovers – i.e., process 120 gal of aqueous – before collecting the final sample from the decanter.

Tests occurred in the following sequence – and approximate operating duration at steady state – to best make use of multiple shifts: 1 (5.6 h), 3A (5.6 h), 5 (7.4 h), 4 (3.6 h), and 2A (5.6 h). Tests 1 and 4 started with fresh strip acid whereas the other tests used the acid from prior experiments.

Appendix D, Table 7 and Table 8 contain the various analytical data (i.e., turbidity measurements, chemical analysis, and droplet size determinations) for this study. Table 7 lists the "Analysis / Sample Time". For samples analyzed using the MicroTrac™ S3000, this time is the time for that analysis. When the sample received a turbidity measurement but no droplet size measurement, the listed time reflects the time of the turbidity measurement (which typically occurred immediately after sample collection). When samples received both determinations, a comment embedded in the parent Excel worksheet provides the time of the turbidity determination (and sample collection). For samples undergoing GC/MS analysis, the listed time is that of sample collection.

For all the samples that received droplet size measurements, Table 8 provides the MicroTrac™ S3000 analysis for volume distribution (assuming spherical droplets). The analyses also included number distribution data (i.e., the reported number of droplets detected within given size ranges). That information is contained within the laboratory notebook.

Thermal Profile of Contactor during Routine Operation.

Personnel obtained a thermal (infrared) image of the contactor during routine operation. Temperature of the aqueous decanter contents and of the Solvent Feed Tank contents remained within range (33 ± 3 °C; 91.4 F) throughout the experiments. Figure 4 shows the installed contactor while Figure 5 shows an infrared image of the operating contactor. The surface temperature of the contactor – see thermal scale of Figure 5 – remained much cooler, closer to the air temperature, except near the upper penetration of the rotor into the body of the contactor. The hot spots are the motor and the bearing. Personnel also circulated deionized water at 33 °C – see two red lines in figure leading from contactor – through the jacket of the contactor.

Figure 4. View of V-5 Contactor.

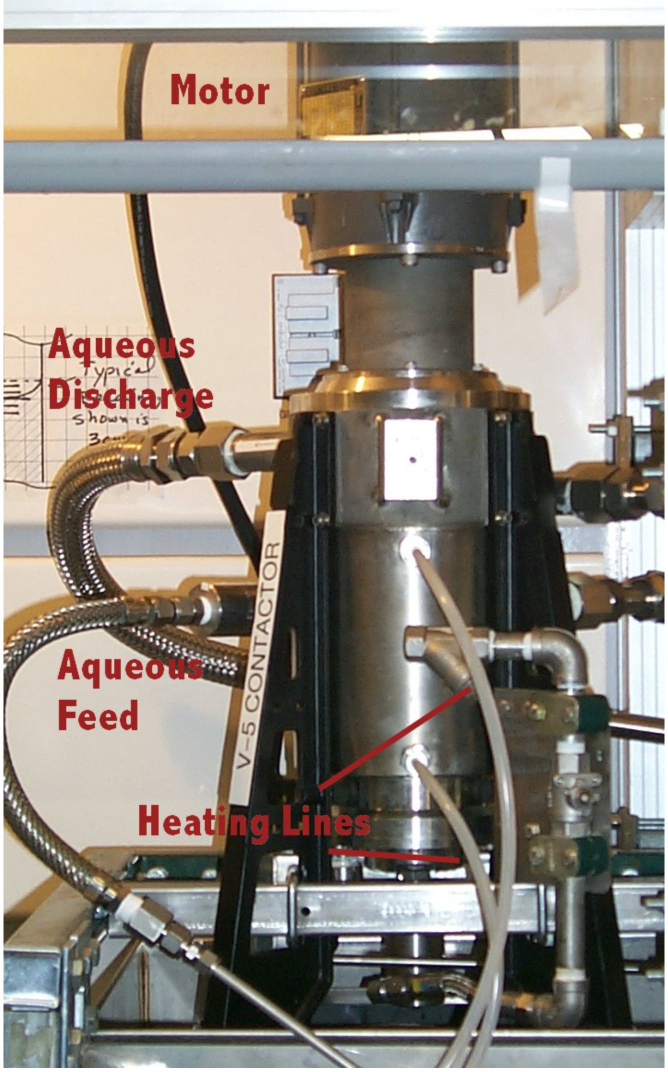
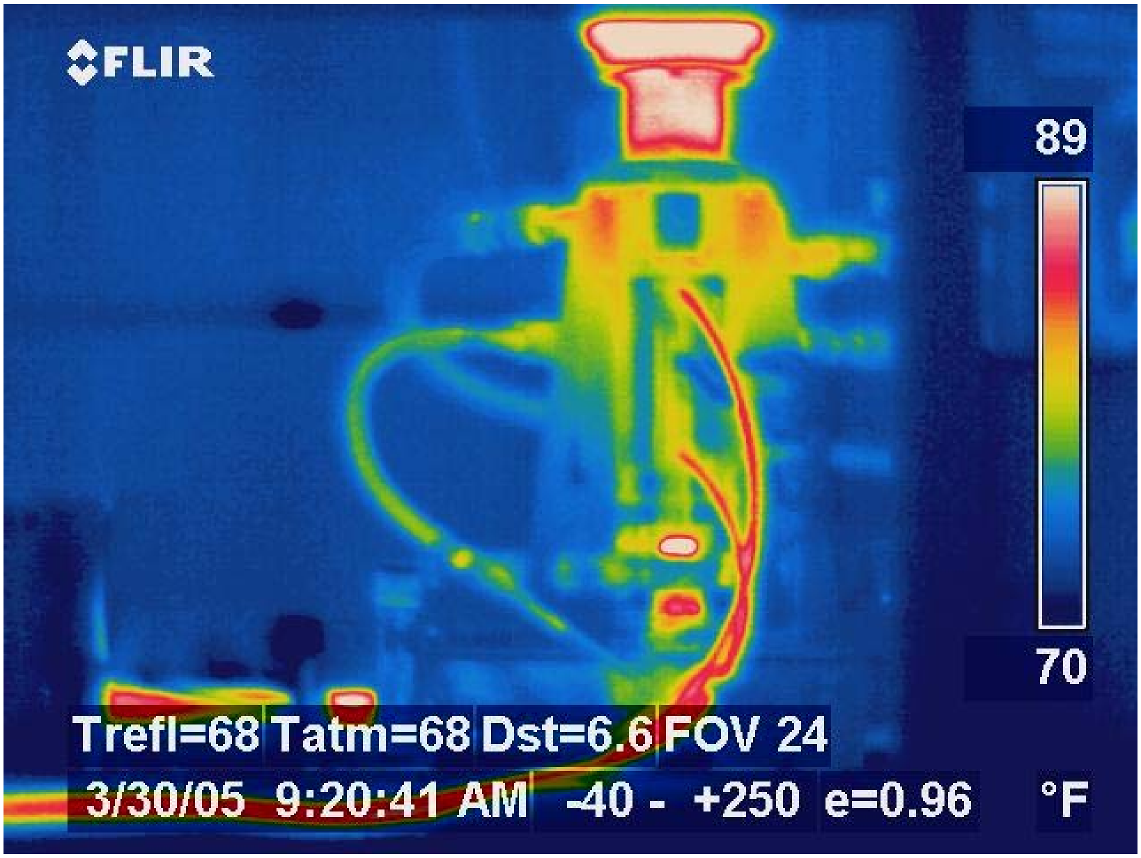


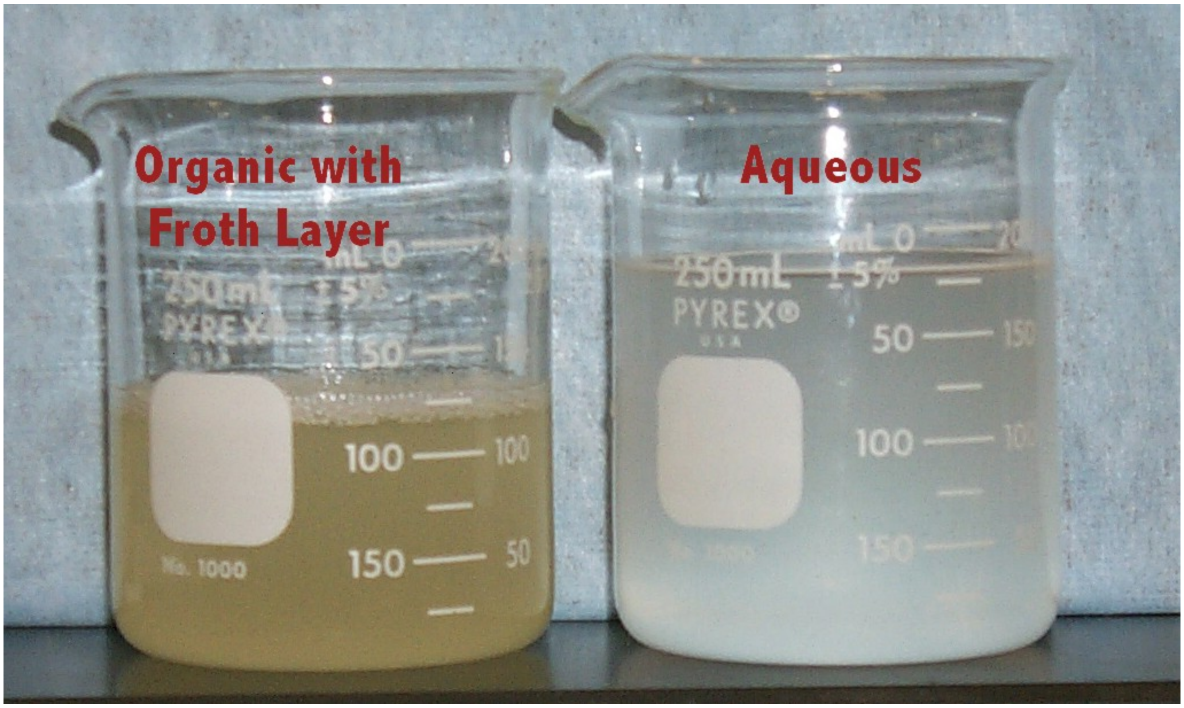
Figure 5. Thermal Profile of Contactor during Routine Operation.



Hydraulic Behavior during Routine Operation

During the experiments, hydraulic behavior remained stable. Minor frothing occurred in the Solvent Receipt tank from the solvent free falling to the surface. The time required for the bulk of the froth to dissipate from samples collected directly from the discharge line ranged from ~1-3 minutes. Given the limited volume of solvent available (~6 gal) and the organic flow rates (1.33 – 2.75 gpm), personnel occasionally observed minor cloudiness in the sight glass of the rotameter on the feed line to the contactor. Frothing in the aqueous samples collected from the discharge line persisted slightly longer than noted for the organic samples. Figure 6 shows a typical set of the organic and aqueous phases.

Figure 6. Typical Appearance of Organic (left) and Aqueous (right) Samples collected from Discharge of Contactor. Note minor froth on top of organic sample.



The bulk of the organic entrained in the aqueous decanter separated fairly quickly from the discharge stream and created a floating oil layer on the surface. Figure 7 shows the decanter contents viewed from above during typical operation. Testing recycled the aqueous stream to minimize waste. After multiple tests, the amount of organic present in the decanter increased. Figure 8 shows the decanter contents during Test 2A – the final experiment. The organic comes for two sequential experiments plus any residual organic not flushed from the tank after removing the aqueous from the first three tests. Visual appearance of the decanter suggested the highest organic content in the aqueous occurred at this time. This conclusion seems consistent with the nature of the last two tests: Test 4 represented the highest total flow test while Test 2A used the highest rotational speed. Hence, one expects these two tests to result in the maximum organic entrainment.

Figure 7. Typical Appearance of Decanter Contents (Test 1).

Figure 8. Floating Organic in Decanter After Multiple Tests (Test 2A).

Organic Entrainment in Acid Stream

Personnel analyzed the samples for organic content using GC/MS.[14,15,16,17] Additional samples received analysis for turbidity as a gross indicator of entrained organic content. Samples examined came both from the decanter outlet and directly from the aqueous discharge line from the contactor. Table 7 contains the results of the measurements for chemical composition and turbidity of the samples

GC/MS Data

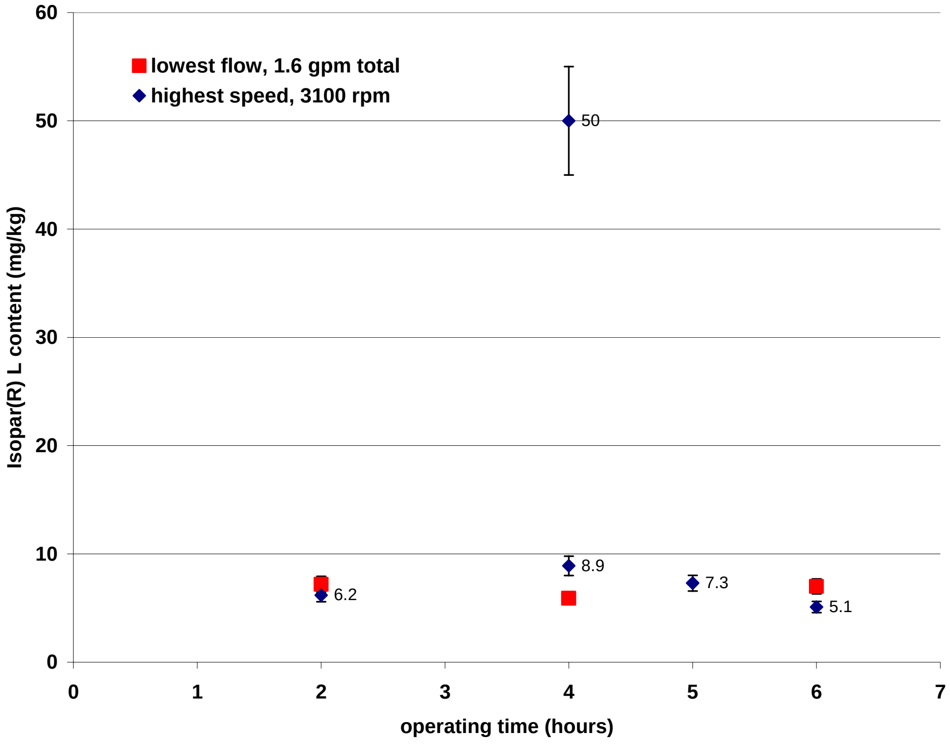
The GC/MS analytical preparation involved the extraction of the entrained organic into a carrier solvent. Personnel extracted the entire sample. Sample volumes ranged from 10 to 75 mL. The expected mass ratios of components in the solvent are 554.2, 239, and 1 for Isopar® L, modifier, and TOA, respectively. In all samples from the decanter or contactor discharge, the TOA fell below the detection limit for the method.

The response of the GC column to Isopar® L and to TOA in the standards proved very good. However, the modifier response proved less reliable when processing large sample volumes. Modifier interacts with the column strongly and tails on subsequent analyses. Modifier peak shapes in the larger samples proved broad. The data suggest retention of modifier on the column following samples containing very large amounts of entrained organic. This same modifier retention and elution behavior persists for pure solvent samples. For these reasons, all the reported modifier concentration data may be low. For samples whose total organic mass did not overwhelm the column – i.e., did not show severe peak broadening for the modifier – the mass reported ratio of modifier to TOA reached values as high as the expected value of 239 (e.g., 240 for Sample 1 of Test 5 – see Table 7). Therefore, the reported modifier results appear approximately valid.

In contrast, the Isopar® L data appears more suspect. The mass ratio of Isopar® L to modifier averages only about 5% of the expected value in all samples. The low relative concentrations of Isopar® L compared to modifier almost certainly does not represent evaporation in the decanter tanks. Personnel analyzed a sample of solvent recovered from the aqueous decanter liquid approximately 2 weeks after testing. Recovery involved collecting and filtering the liquid through a Millipore Glass Fiber Pre-filter, catalog number APFF09050, rated at 0.7 micron. Recovery occurred over several days and involved vacuum filtration for overnight periods. Personnel took no special precautions to avoid evaporation. The density measured 0.8711 ± 0007 g/mL (21.7°C) indicating an evaporation of ~20% of the Isopar® L. In contrast, personnel also measured the density of the recovered solvent transferred to Parsons-SRS after the testing as 0.8541 ± 0004 g/mL (21.7°C). Hence, the solvent in the equipment experienced very little if any evaporation of Isopar® L. These data suggest a maximum evaporation under 20%, much less than the ~95% loss measured for nearly all the GC/MS samples.

The GC/MS measured a maximum Isopar® L concentration in the samples from the decanter at the end of the tests of 11 mg/kg. The maximum measured concentration (i.e., 50 mg/kg) of Isopar® L came from a sample from the contactor discharge line in the highest speed test. Figure 9 shows the measurements for contactor discharge samples collected in the tests with highest rotational speed and with lowest overall throughput. One would expect the variance across these sets to reflect the maximum contrast in organic entrainment. Other than the single extreme sample in the high speed test, the data are remarkably consistent.

Figure 9. Variation in Organic Content of Contactor Discharge Samples.



The modifier concentrations are appreciably larger than the Isopar® L data. Table 2 contains the measured values for final decanter outlet samples as well as the range of values for contactor discharge samples in the two tests where personnel collected such samples. The CSSX solvent contains 0.2967 weight fraction modifier. Table 2 also contains the calculated, or implied, total organic content for these samples assuming the modifier is present at the original concentration. The average concentration measured for all samples is 293 mg/kg (or $\mathrm{ppm_m}$) which corresponds to ~345 $\mathrm{ppm_v}$ (or 0.0345 vol %). The maximum measured value is 808.9 mg/kg (or $\mathrm{ppm_m}$) which corresponds to ~952 $\mathrm{ppm_v}$ (or 0.0953 vol %). These values compare favorably with prior larger scale testing using simulated waste in which entrainment typically measured ~0.012 vol % but reached as much as 0.33 vol % for the organic in the decontaminated waste stream.[5] When one considers only the contactor discharges samples – available only for Test 2A – the entrainment averaged 329 $\mathrm{ppm_m}$ with a range of 192 t0 606 $\mathrm{ppm_m}$.

Table 2. Modifier and Implied Total Organic Content of Samples by GC/MS.

	GC/MS Modifier (mg/kg)		Implied Total Organic (mg/kg)	
Test	**Decanter Outlet Sample (Final)**	**Contactor Outlet Samples**	**Decanter Outlet Sample (Final)**	**Contactor Outlet Samples**
1	31	--	104.5	--
2A	3.5	57 to 180	11.8	192 to 606
3A	41	--	138.2	--
4	85	--	286.5	--
5	240	75 to 98	808.9	250 to 330

Immediately after completion of testing, personnel emptied the contents of the systems and weighed the amount of recovered solvent. The collected amount corresponded to ~5.64 gal versus the starting amount of ~5.95 gal. This implies that the 80 gal of strip acid from the first tests may contain as much as 0.31 gal (0.39 vol %) organic. This amount agrees reasonably well with the maximum entrainment reported in prior large scale tests[5] with simulated waste (i.e., as much as 0.33 vol %). The 0.31 gal (0.39 vol %) is notably greater than the instantaneous values based on modifier content. However, the total carry over in the drums includes organic from all start up periods and disruptions – during which organic entrainment is appreciably larger. Given this corroborating information from the gross material balance, we believe the modifier data provides a realistic implication of the total organic content from the contactor discharge.

Turbidity Data

The turbidity measurements from the various decanter samples ranged between 10.44 to 42.5 NTU. See Table 8 for the droplet size data. For samples collected from the contactor discharge line, the values ranged from 10.05 to 32.9 NTU, a comparable spread. Neither data set shows obvious trends with respect to time. Also, no correlation between contactor and decanter effluent samples is apparent. Figure 10 shows data from Test 4 which used a fresh supply of 0.001 M acid in the decanter. A plot of data for Test 2A – the test with highest rotational speed – shows similar behavior (Figure 11). [Since Test 2A re-used acid from an earlier test, visual observation of the decanter does not aid in assessing the amount of organic entrained during the experiment. Photos – not included here – show that one can not visually discern a difference in the amount of solvent floating on the surface of the aqueous in the decanter.] A plot of turbidity – not included in report – versus Isopar® L concentration by GC/MS shows no apparent correlation.

Figure 10. Turbidity Measurements of Samples from Test 4.

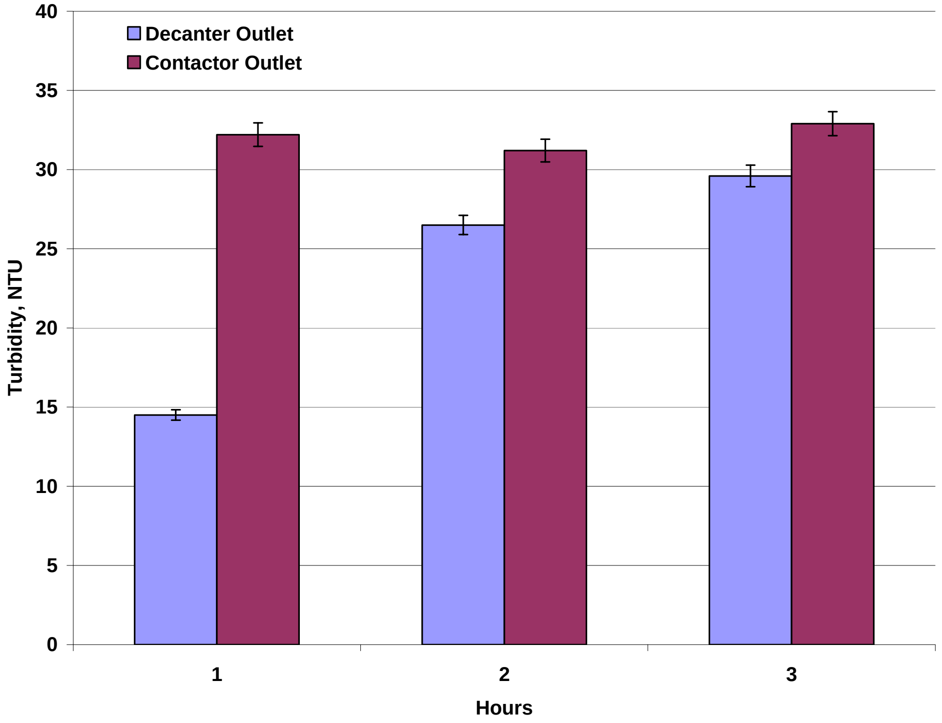
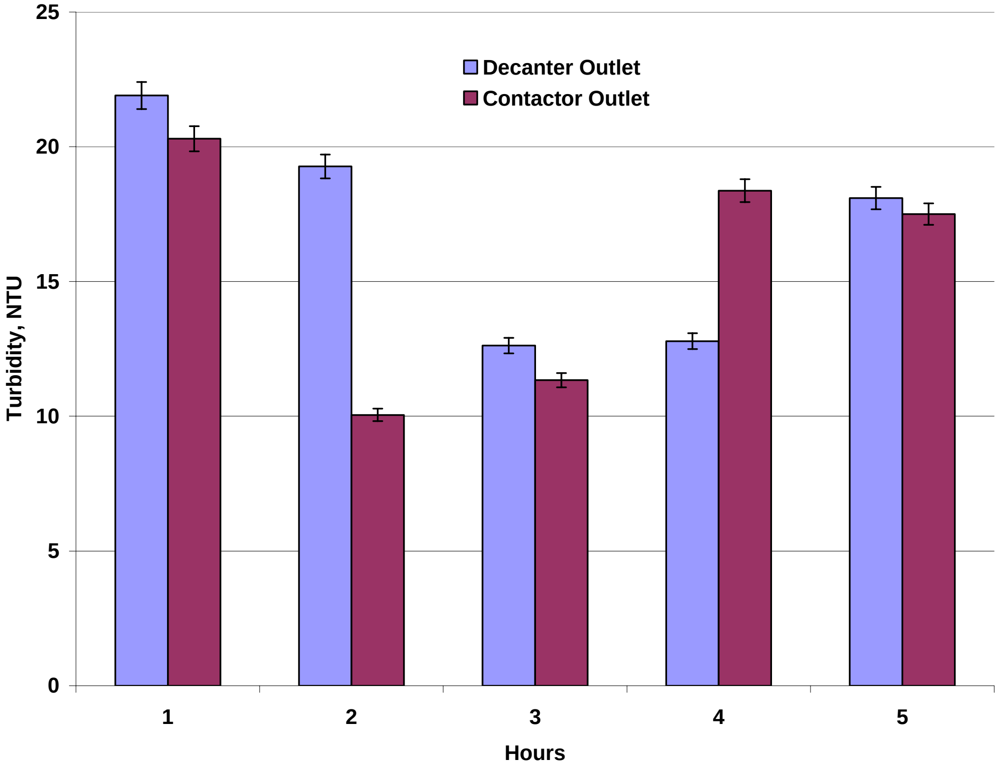


Figure 11. Turbidity Data from Test 2A (3100 rpm).



Droplet Size in Acid Stream

Personnel analyzed selected samples by the MicroTrac™ S3000 and by photographic microscopy for droplet size determinations. Most of the MicroTrac™ S3000 samples analyzed in this study contained too little organic to provide reliable statistical data as reported by the software package. Nevertheless, the authors report the trends for the data as they appear insightful.

The microscopy data supports the findings that the droplet sizes are generally less than a few microns in size. Figure 12 shows an example of the micrographs showing droplets from Test 5 with all observed droplets less than 7 µ in diameter. The great majority of the microscopy photos show droplets of this size and smaller.

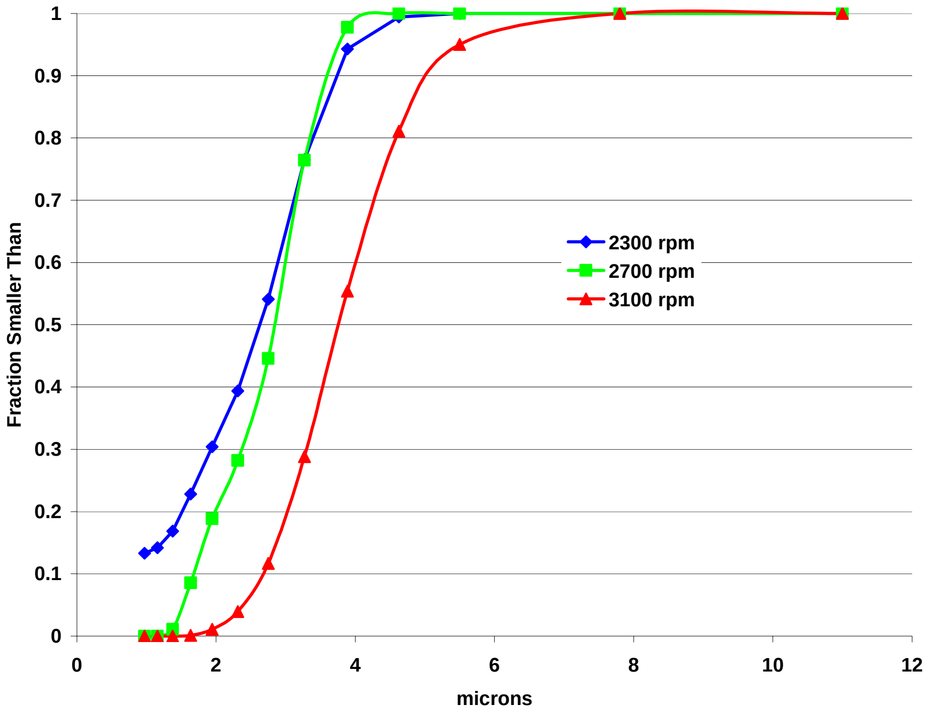
Figure 12. Photo Micrograph of Sample Decanter Sample from Test 5.

Samples from Decanter Contents and Effluent

Personnel collected samples at the completion of three decanter turnovers and aged these two hours per the protocol established in the parallel study for 2-cm contactors. Since every test did not use a fresh acid supply, the decanter contents typically contained organic droplets from the prior test(s) in addition to fresh organic entrained from the contactor. This attribute of the testing undoubtedly adds some "smoothing" of the data sets between experiments. Nevertheless, the data sets show discernable trends that appear to support expected behaviors.

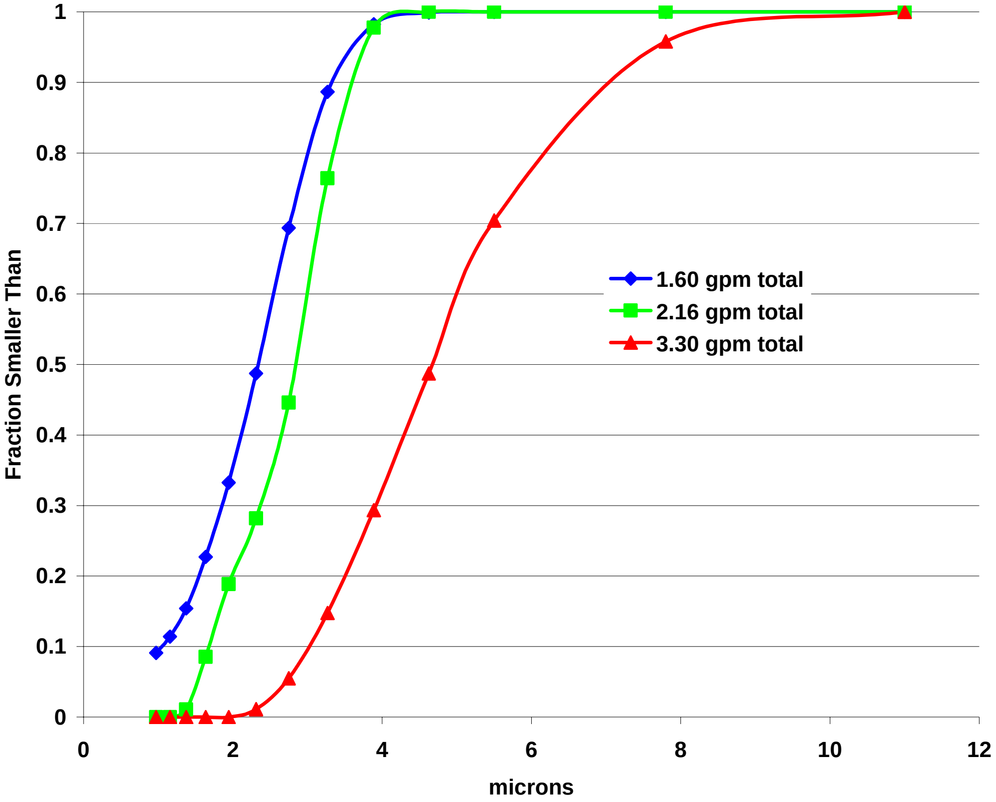
Tests 3A (2300 rpm), 1 (2700 rpm), and 2A (3100 rpm) examined the impact of rotational speed on droplet size at nominal throughput for the MCU. One expects an increase in rotational speed to cause a decrease in droplet size if the equipment is operating more as a mixer than a separator. Figure 13 shows the data from this study. Apparently, the contactor and coupled decanter system operated effectively as a separation system at the nominal processing speed (i.e., 2700 rpm) for the MCU. At the highest speed (i.e., 3100 rpm) examined, separation efficiency declines as evidenced by an increase in the number of large drops exiting the decanter. The GC/MS trend for Isopar® L concentration may also support this conclusion although the variance is less apparent for the data. Figure 9 shows that Test 2A (3100 rpm) did have the most extreme instantaneous concentration of Isopar® L for samples from the contactor discharge.

Figure 13. Impact of Rotational Speed on Droplet Size.



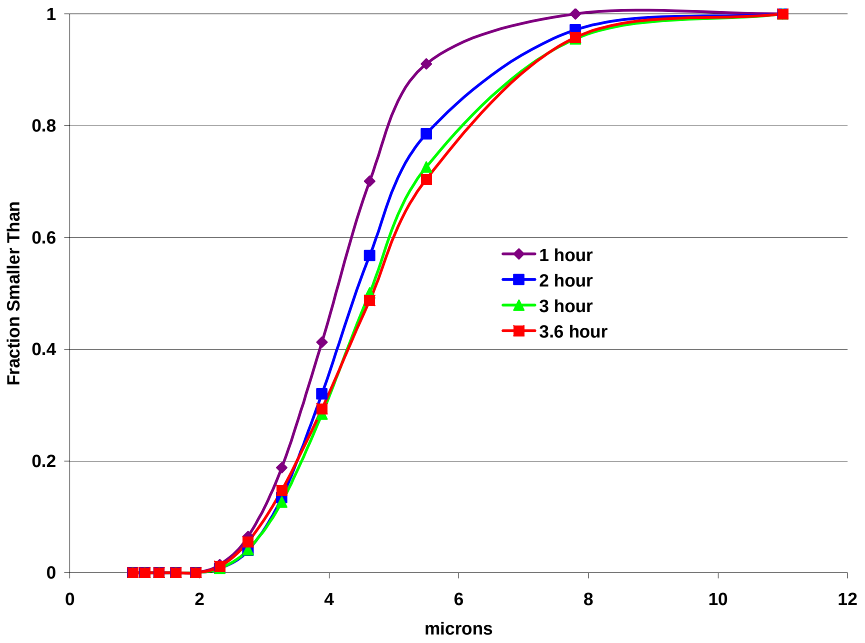
Tests 5 (1.60 gpm), 1 (2.16 gpm), and 4 (3.30 gpm) examine the impact on increasing total flow rate on droplet size at the nominal operating speed of 2700 rpm chosen for the MCU. Figure 14 shows the results of these tests. As flow rate increases, residence time within the contactor – and within the decanter – declines. Shorter residence time allows less separation and hence larger droplets exit the decanter. [For this comparison, the GC/MS data are too sparse to provide insight to the behavior.]

Figure 14. Impact of Flow Rate on Droplet Size.



Test 4 had the highest total throughput and hence the shortest duration. One may expect that the test would show the greatest variability of droplet size as a function of time. Recall that Test 4 also started with a fresh supply of 0.001 M nitric acid added to the decanter and hence should show less impact from prior tests (other than Test 1). Figure 15 shows the droplet size of samples collected from the decanter outlet as a function of operating time during Test 4 (3.30 gpm total flow). [The analytical time for these samples varied by about one hour and did not correlate rigorously with the collection sequence for the samples. However, inspection of the data suggests minimal or negligible impact from this variation.] Note that the droplet size increases during the course of operations, suggesting that the decanter does indeed result in coalescence of smaller droplets over time. The variance decreases at longer times, suggesting the droplet size distribution is reaching steady state after three decanter turnovers, confirming an assumption of the test design.

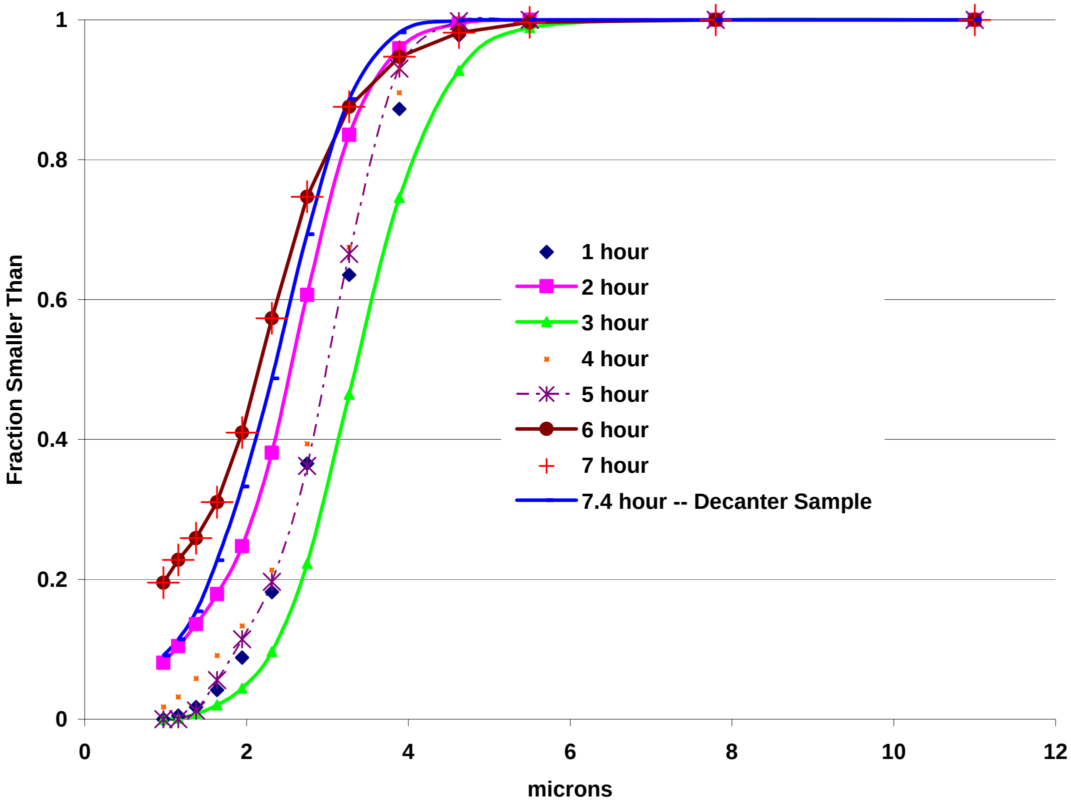
Figure 15. Impact of Operating Time on Droplet Size from Decanter.



Samples from Contactor Effluent

Since the project re-used the acid in the decanter, personnel grew concerned about the bias that would result for samples collected from the decanter outlet. Hence, in the longest duration experiment, Test 5, personnel collected hourly samples directly from the discharge line entering the decanter and analyzed these using the MicroTrac™ S3000. Figure 16 shows that data.

Figure 16. Impact of Operating Time on Droplet Size from Contactor.



The droplet size in these instantaneous samples from the discharge of the contactor show low correlation with time; the droplet sizes vary over the complete range exhibited by samples collected from the decanter outlet. However on average, the instantaneous droplet sizes appear larger than those from the decanter outlet although the difference may not prove statistically significant. [Note that the GC/MS data also shows no appreciable change in Isopar® L concentration from the contactor outlet versus the decanter outlet. However, these data are less definitive on this point given that the decanter holds aqueous from two prior experiments.]

Feasibility Test of Coalescer Material

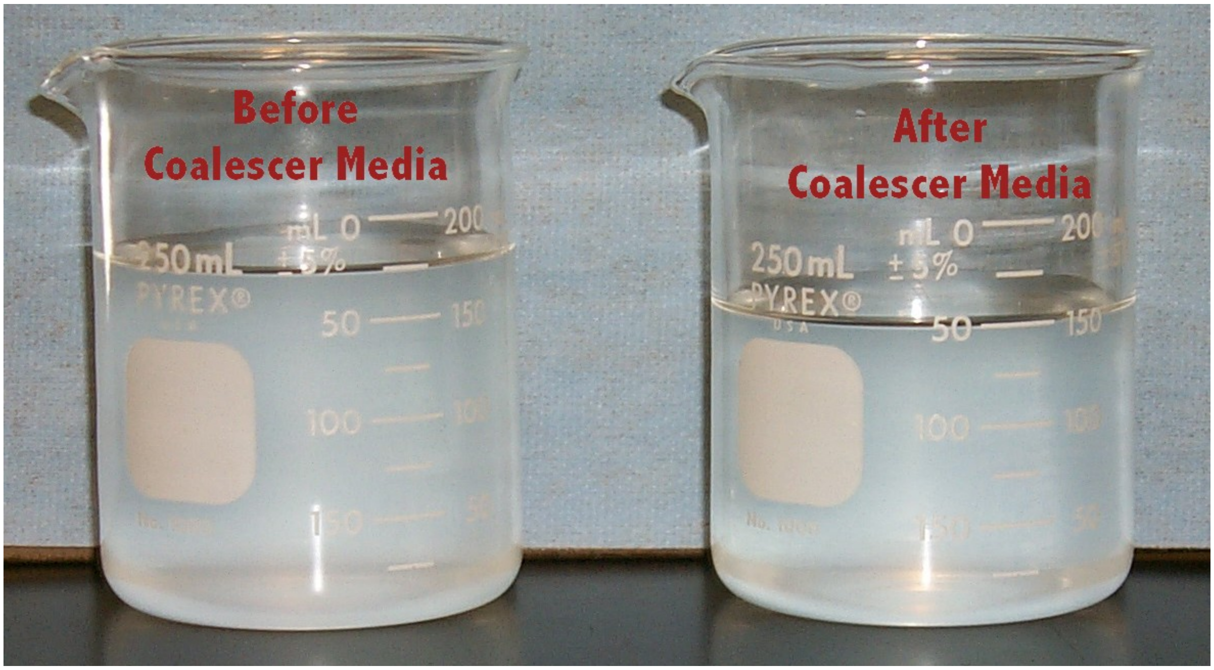
Earlier testing by SRNL and efforts to recover the emulsified solvents each demonstrated the effectiveness of Millipore Glass Fiber Prefilter, catalog number APFF09050, rated at 0.7 micron, to coalesce the fine organic droplets and promote coalescence of trace organic phase from the aqueous stream. Personnel identified a vendor coalescer material– Fiberglass Mat Interceptor-Pak™ by ACS Separations, Inc. – that resembled this filter material and obtained samples for testing. Personnel wrapped the coalescer material around the inlet of the stilling tube from the decanter. Figure 17 shows the installed coalescer material for testing.

Figure 17. Coalescer Material Configuration.

The design consists of two layers of the medium wrapped in cylinders around the 2.5 inch outer-diameter pipe. The layers are wrapped to provide butt joints with the joints for the two layers offset by 180°. The layers extend 3 inches above the end of the pipe and are tied by wire. The bottom ends of the coalescer layers wrap around a glass beaker approximately 3 inches high and are similarly attached by a wire. The glass beaker acted as an end cap for the design. A gap of 4 inches existed between the bottom of the pipe and the top of the glass beaker.

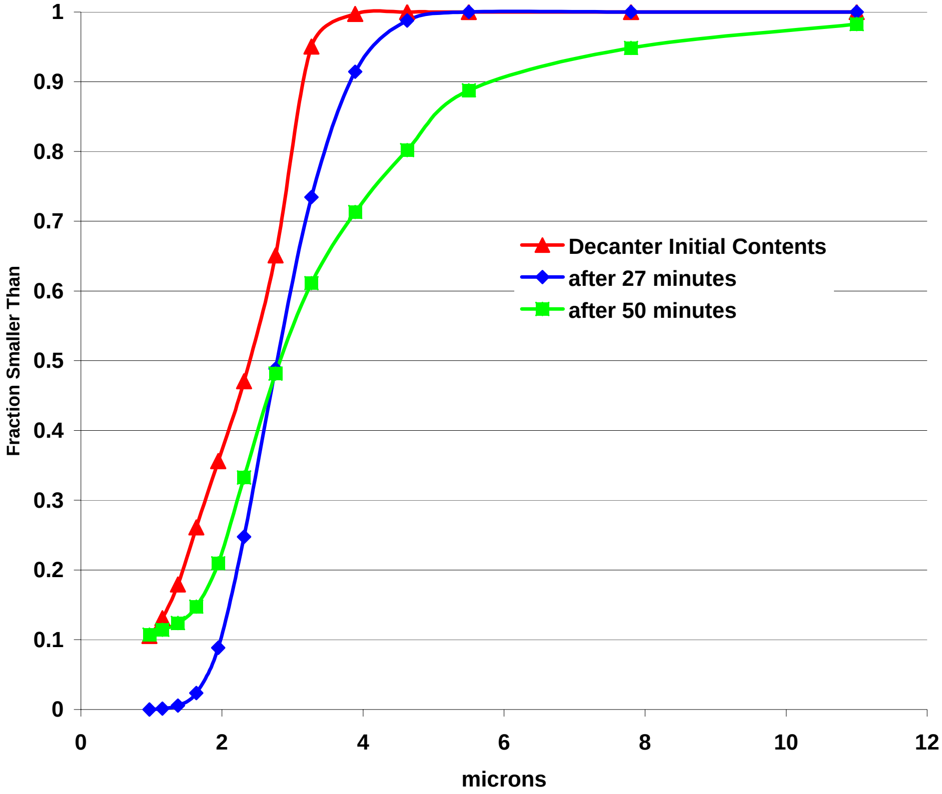
Personnel operated the aqueous stream under recycle at a flow rate of 0.36 gpm. The flow bypassed the contactor; testing did not include an integrated test with the contactor. This flow rate falls well within the vendor claims for acceptable rates for this material. Personnel completed two tests lasting approximately 1.5 hours each. The decanter sat idle for approximately 1 hour before the start of the first test; the second test occurred the following day. Every 30 minutes, personnel obtained samples from both the bulk contents of the decanter and from the outlet stream from the stilling tube. Visual observations indicated minimal to negligible difference in the haziness of the aqueous samples. Figure 18 – from the first test – shows a typical set of samples before and after treatment by the coalescer material. Only a very slight reduction in haze is noticeable. Personnel suspected that the difference in entrained organic content would fall within the analytical accuracy and hence elected to omit chemical analysis.

Figure 18. Typical Samples from Coalescer Test: Before (left) and After (right) Flow Through Coalescer Material.



Personnel submitted samples from one test for droplet size determination by the MicroTrac™ S3000. The analysis indicated relatively few drops of organic present either in the samples directly from the decanter or from the stilling tube after flowing through the coalescer material. Hence, the data may not prove statistically reliable. Nevertheless, the trend in the data does prove interesting. Figure 19 shows a graph depicting the measurements. The data appear to show that the material causes coalescence of smaller droplets. As time passed, the droplet size downstream of the coalescer material increased – i.e., the initial data represent decanter contents that did not pass through the material. [Note: 27 and 50 minutes correspond to 0.24 and 0.45 decanter volume turnovers, respectively.]

Figure 19. Impact of Coalescer Material on Droplet Size.

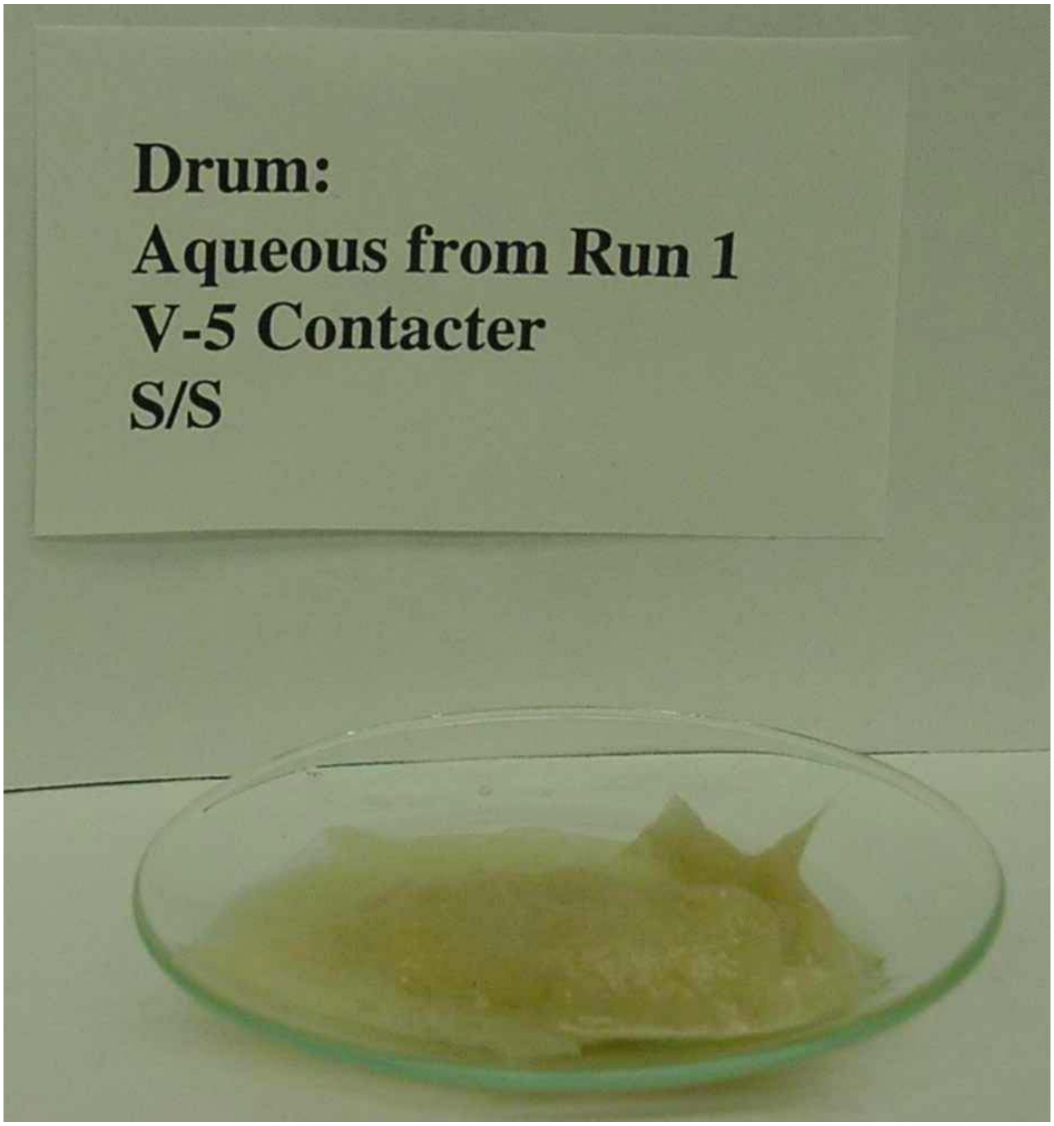


Recovery of Organic from Stored Decanter Contents

At the end of testing, personnel stored the spent aqueous in 55 gal drums. After allowing 2-3 weeks of undisturbed storage, personnel inspected the drums for purpose of siphoning any floating organic. One drum contained the aqueous from the initial operation that had emulsified the solvent. The floating layer in that drum proved much different from the drums container "routine" decanter contents. The layer took on an almost solid appearance – see Figure 20, with a rather densely concentrated organic phase.

After the storage period, personnel recovered ~ 800 mL of separated solvent from the surface of one of the drums. This amount corresponds to an entrainment value as high as 0.53 vol % over the course of the tests for that selected material. Hence, a per test value may reach as high as 0.18-0.26 vol % ignoring any contributions from flushing the solvent loop. [The drums also contained flush water from disassembly of the equipment and hence this sample may also contain solvent from that flushing process.] These values are of the same order of magnitude as previously reported (i.e., up to 0.33 vol %) for entrainment of organic in the decontaminated waste from earlier tests.[5] One expects slightly larger values for the current tests since organic entrainment proves worse in the lower density strip acid than in the waste stream.

Figure 20. Recovered Organic from Surface of Drum with Remnants Emulsified Solvent.



CONCLUSIONS

Personnel completed a rapid study of organic entrainment during operation of a CINC V-5 contactor under prototypical conditions covering the range of expected MCU operation. The study only considered the entrainment of organic into the strip acid effluent destined for the Defense Waste Processing Facility. [Schedule limits prohibited an investigation of the extraction effluent or a more complete study of operating conditions.] Based on this work, the following observations are noted.

- Concentrations of total organic from the contactor discharge, based upon modifier measurements, in the acid typically averaged 330 ppm_m, for a range to 190 – 610 ppm_m.
- Entrained droplet sizes remained below 18 microns for samples collected at the decanter outlet and below 11 microns from the contactor based on MicroTrac™ S3000 laser measurement.
- Scouting tests showed that a vendor coalescer material promotes coalescence of smaller size droplets from the decanter effluent but thickness must be significantly larger than those tested here to achieve process-significant organic removal.
- Process throughputs and planned operating conditions result in very stable hydraulics, suggesting that the MCU stripping stages will have spare operating capacity.
- The V-5 contactors show operated with relatively cool surfaces under the planned operating conditions.
- Personnel observed a previously unreported organic impurity in the solvent used for this study. Additional efforts are needed to ascertain the source of the impurity and its implication on the overall process.
- If operating conditions result in an imbalance of the relative mixing and separation conditions within the contactor, a very stable emulsion may result. In this instance, the undisturbed emulsion remained stable for weeks. The imbalance in this study resulted from use of improperly sized weir plates.
- Personnel demonstrated an effective means to recover emulsified solvent following a non-optimal equipment configuration. The protocols developed may offer benefit for MCU and SWPF operations.
- This study developed and demonstrated the effectiveness of several analytical methods for support of the Caustic-Side Solvent Extraction process including infrared spectroscopy and droplet size measurement by a MicroTrac™ S3000. Interfacial tension measurements also showed sensitivity to purity of the solvent suggesting that this technique may prove valuable for future process diagnostics.

- The study highlighted limitations of the current gas chromatography configuration for determination of modifier content of samples.

RECOMMENDATIONS/PATH FORWARD

Additional development of analytical methods for determining composition – and particularly – modifier content – of organic and mixed aqueous-organic samples is warranted. Infrared spectroscopy shows promise as a rapid method with adequate accuracy and lower waste generation. Such a method also offers great promise for at-line or on-line application.

This study did not investigate the entrainment of organic in the aqueous effluent from the extraction operation. Additional full-scale studies for that purpose are warranted. Since the waste composition may differ appreciably for the process, this stream may exhibit much wider variance in hydraulic behavior, organic entrainment, and may pose a greater risk for poor hydraulics.

Conducting these studies at SRNL allowed numerous personnel to view operations and facilitated the training on staff members. Members of the Design Authority, Engineering, and Training groups benefited from tours. Strong consideration is appropriate for procuring and installing a full-scale contactor at SRNL for future support and for assistance during commissioning of the MCU.

Additional efforts are needed to ascertain the source of the unknown contaminant in the solvent. The impact of this contaminant on mass transfer chemistry is unknown; hence, scouting experiments to measure cesium distribution performance with the solvent are desirable. Additional efforts to isolate and positively identify the impurity are highly advisable. Finally, chemists should assess whether an additional decomposition mechanism is active other than those identified in past studies.

Additional study of surface and interfacial tension is recommended. This tool may also offer economical and rapid process diagnostics for future operations.

ACKNOWLEDGEMENTS

The staff of the Engineering Development Laboratory provided exceptional support during design, installation, operation, and disassembly of the equipment. John Steimke provided the heat transfer calculations for sizing the temperature control system. Tim Steeper assisted in various aspects of the design and especially proved instrumental in the decanter and coalescer design elements. Duane Adamson provided exceptional support tracking progress and readiness during the hazards assessment effort. Susan Hatcher provided coordination of the procurements, completed most of the hazards assessment and mitigation activities, and supervised the technician staff.

Mike “Doc” Armstrong completed the bulk of the fabrication, piping, and assembly operations. He also performed virtually all of the contingent repairs and configuration changes as issues emerged. Mike performed a large share of the direct operations and displayed exceptional insight into the principles of operation. Jimmy Mills, Andy Foreman, and Vernon Bush assisted in calibrations, installation, and operations.

Henry Bolton, Jeannine Mills, Dennis Lewis and Sharon Smith provided sample collection and preparation support for the experiments including turbidity measurements. Each also assisted in the investigations to define a recovery path for the emulsified solvent. Kim Wyszynski performed many of the surface and interfacial tension measurements on the solvent samples.

Annie Still prepared the numerous samples for the gas chromatography work. Ronny Rutherford assisted with the MicroTrac™ measurements and Cynthia Foreman helped with the microscopy of samples. Cecil May assisted in the purification of the solvent received from Parsons-SRS. David Herman assisted in recovery efforts for the emulsified solvent and made key observations regarding the purity of the as-received solvent. Doug Walker provided technical consultation for a number of aspects of the program. Seth Campbell assisted in the initial identification of the test matrix.

Chuck Terhune and Jack Kasper of Parsons-SRS approved the loan of solvent and a V-5 contactor to SRNL for these studies. Rich Smalley, Bob Leugemors, Darryl Wolfe and Don Haberkost assisted in arranging and coordinating the loan of solvent and the V-5 contactor from Parsons-SRS. Alan Stephens led the effort to identify the weir configuration of the loaned contactor and, along with Bill Brasel, arranged the shipment of the replacement aqueous weir plate to SRNL on short notice. Without these generous acts of cooperation, this study would not have been possible.

Glynn Dyer, Renee Spires, Brent Gifford, and Earl Brass arranged the funding for this effort.

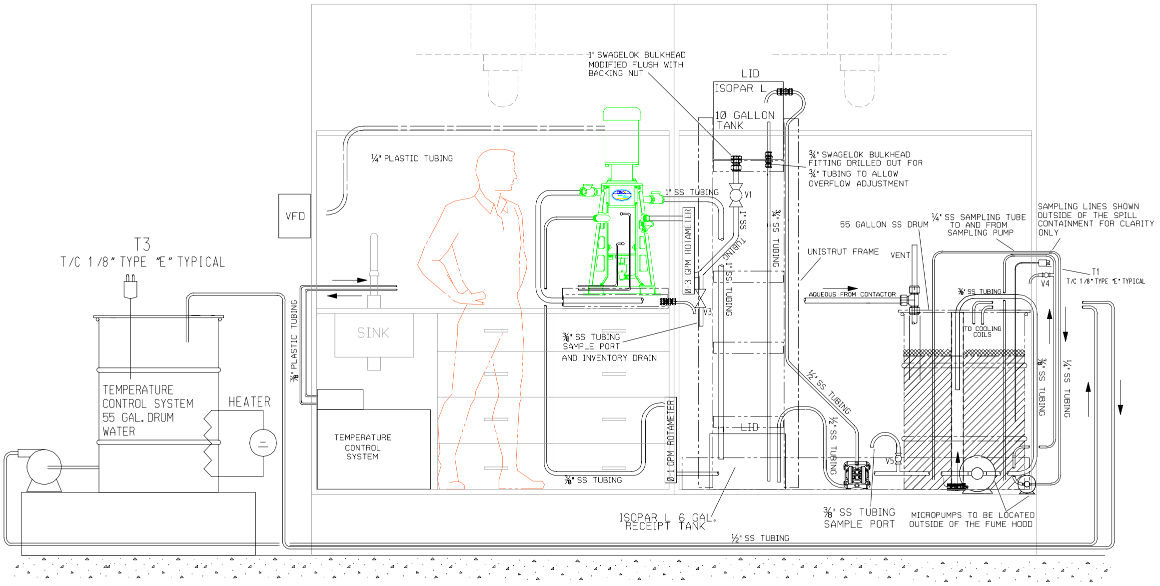
Mitch Peel provided the excellent project budget and schedule liaison function for this task allowing the research team to concentrate fully on meeting the accelerated schedule.

APPENDIX A– EQUIPMENT DETAILS

The contactor itself was found to leak from the bottom rotary seal. This seal was temporarily plugged with a threaded fastener (thereby defeating the clean in place (CIP) system, which was not used during testing. The plug was removed prior to returning the contactor).

The overall equipment arrangement is shown below in an elevation view, Figure 21.

Figure 21. V-5 Contactor and Decanter Layout.



The Piping and Instrument Diagram is shown below in Figure 22.

Figure 22. V-5 Contactor Installation, Piping, and Instrument Diagram.

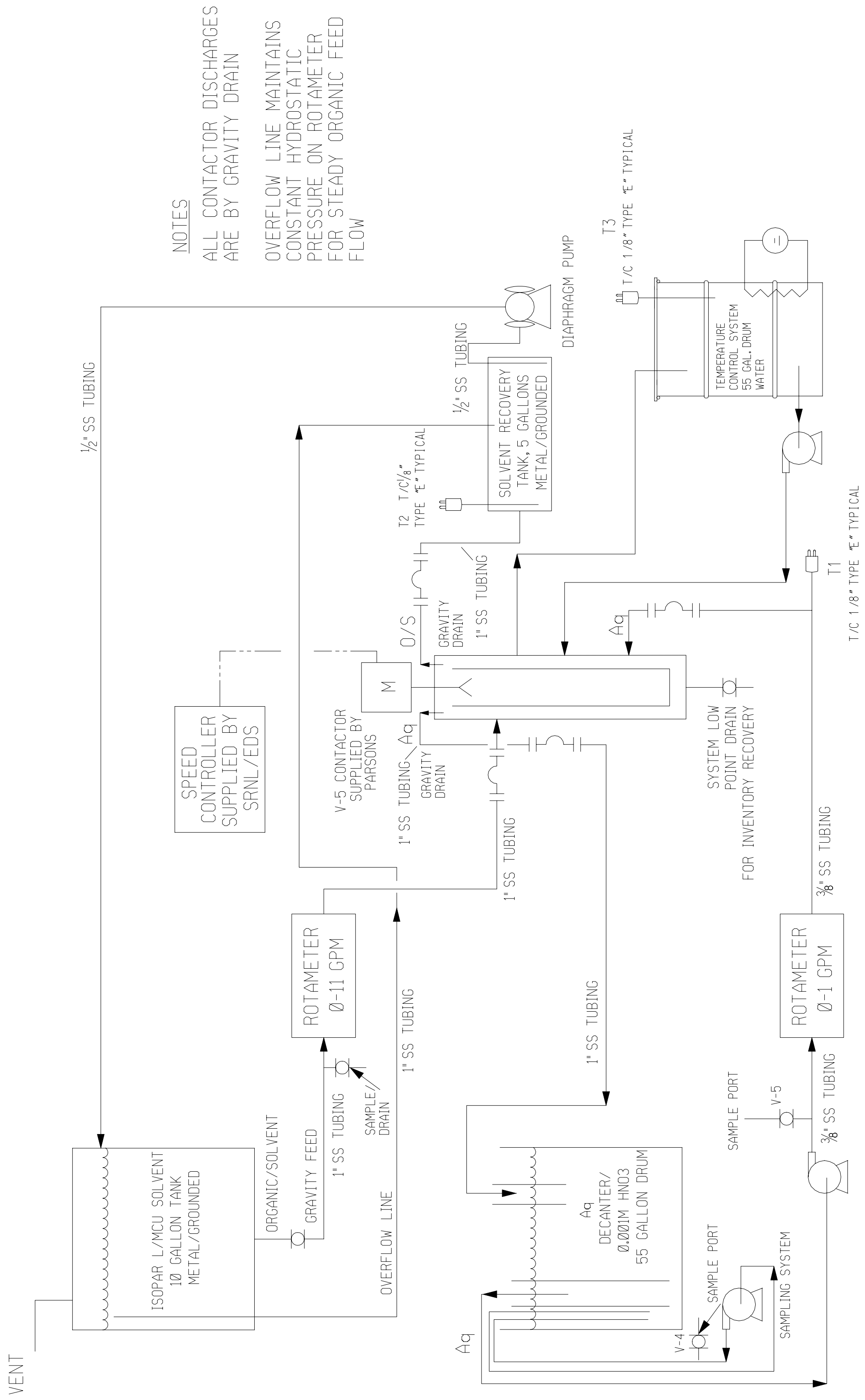
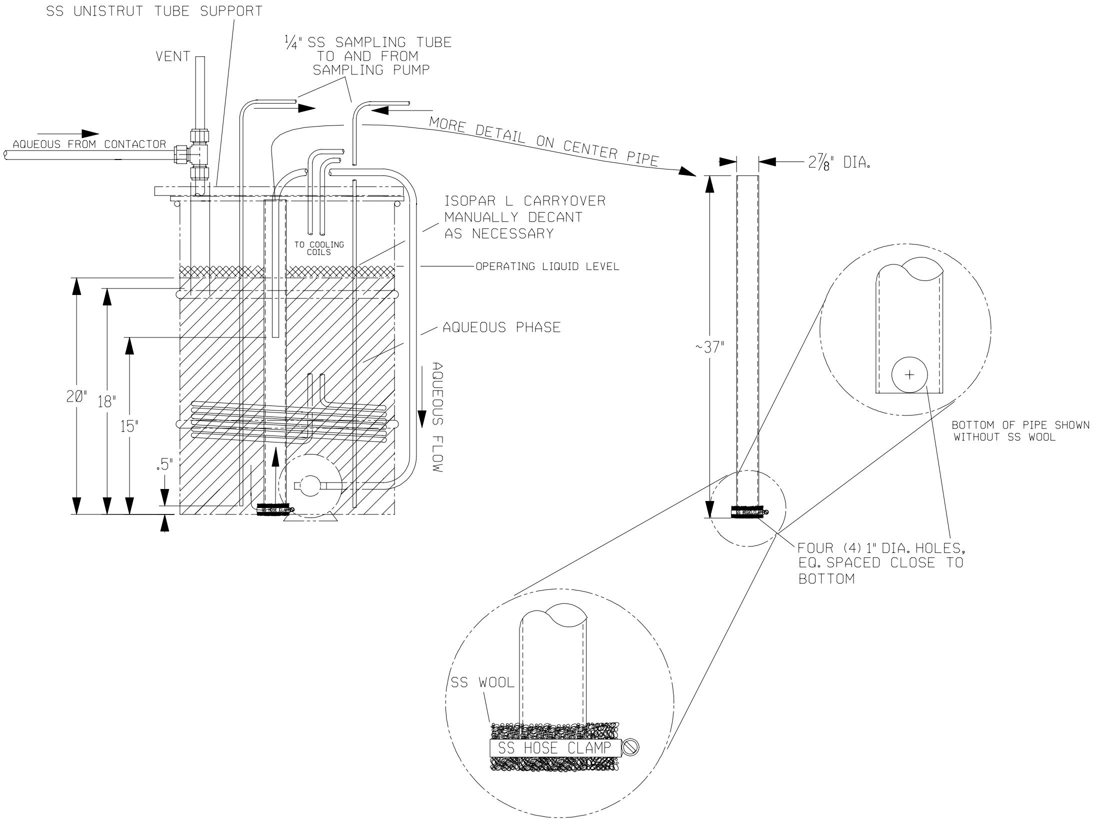


Figure 23 below is the design used for the V-5 Contactor Aqueous Decanter.

Figure 23. Aqueous Decanter Design.



A control system maintained the temperature of the aqueous solution to 33 ± 3 °C. This system consisted of a 55 gallon drum half filled with water, with three rheostat-controlled, 1 kw drum-band heaters, a flexible impeller pump, and the necessary ½" tubing to reach from the 55 gallon drum to the aqueous decanter heating coils. This system remained at approximately 40 °C during all testing. An additional lab size control system supplied the contactor jacket with water maintained at 33 ± 3 °C.

The aqueous system involved the constant recycling of 0.001M nitric acid strip solution, as per the site MCU system design. The design included an aqueous decanter system to limit any organic carryover into the aqueous feed system. This decanter system was designed to be as prototypical as possible given the compacted test schedule. Discussions with the Design Authority for the MCU system led to the determination of a minimum needed residence time of 45 minutes.

The decanter tank also served the aqueous feed tank, minimizing the amount of strip solution required. Because of the low expected carryover of aqueous into the solvent system, the design did not include a decanter into the solvent system. The solvent system recycle loop pulled solvent from 1" off the bottom of the Solvent Receipt Tank, allowing visual detection of any accumulation of aqueous. Testing resulted in negligible aqueous accumulation. Because of the limited amount of solvent, the maximum residence time in the Solvent Receipt Tank at the highest solvent flow rate proved less than 1 minute, minimizing the effectiveness of any installed solvent decanter.

Table 3. List of Measurement and Test Equipment (M&TE).

Instrumentation	M&TE	Range	Accuracy (full scale)
0-11 gpm Rotameter	TR-03766	0 to11 gpm	± 0.12 gpm
20-80 gph Rotameter	TR-03615	20 to 80 gph	± 1.95 gph
Type J Thermocouple	TR-01051	0 to 750 °C	± 1.7 °C
Type J Thermocouple	TR-01036	0 to 750 °C	± 1.7 °C
Type E Thermocouple	TR-03726	-200 to 900 °C	± 1 °C

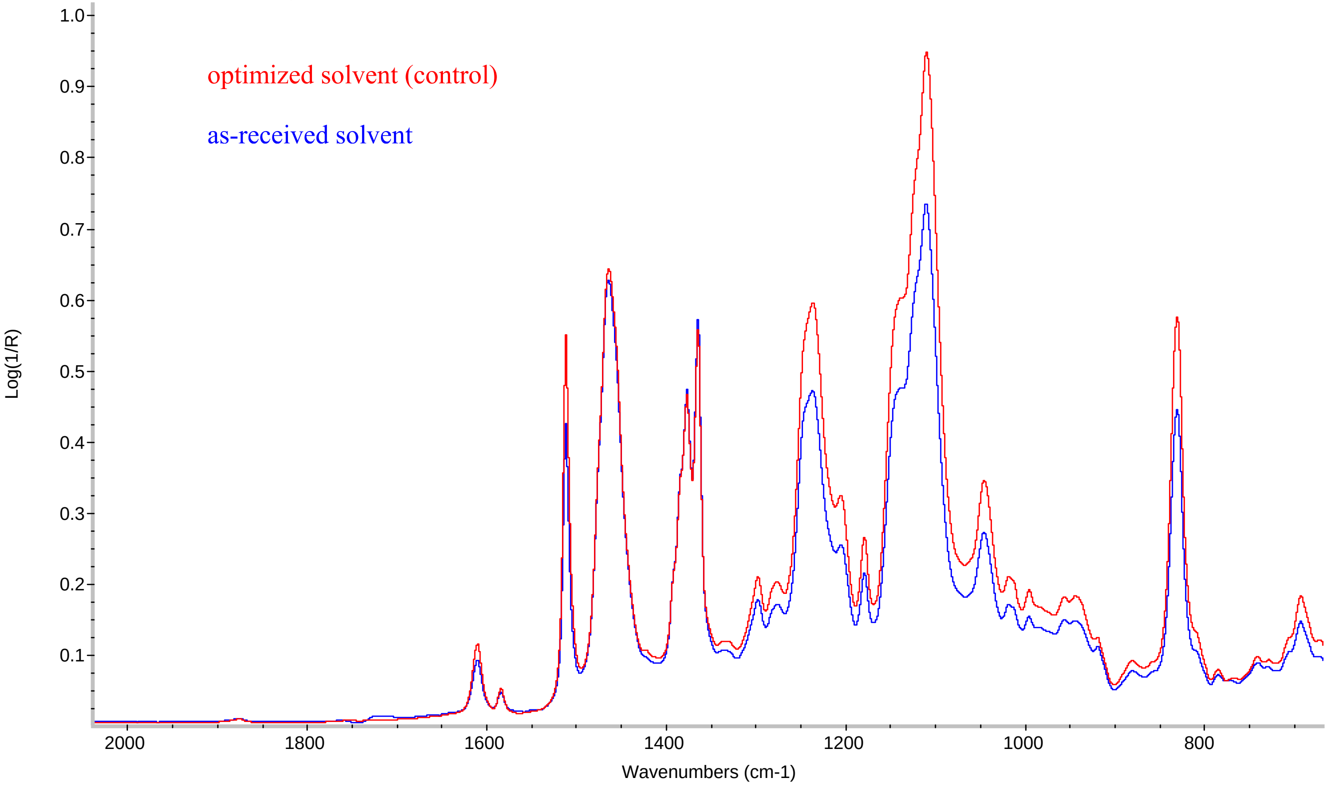
APPENDIX B – SOLVENT PURIFICATION

The MCU solvent (referred to hereafter as "Parsons Solvent") arrived at SRNL on March 2, 2005, in a stainless-steel 30 gallon drum in a secondary containment drum. The container was sealed with security tape. Parsons-SRS personnel estimated the drum contained a total of ~9 gallons of material.

Personnel tracked all operations with the solvent using strict custody control procedures. They recorded all custodial changes involving > 10 mL samples of the solvent tracked in a Solvent Log Book. At the end of each day of operations, personnel placed a tamper-resistant seal on the drum. Only a limited number of people were authorized to manipulate the solvent or the tamper-resistant seals. To prevent solvent contamination, personnel stored or manipulated the solvent only in glass, steel or Teflon™ containers.

Parsons-SRS personnel notified SRNL that the solvent contained an extra quantity of flush material from a final flush of the contactors. The flush material was 2 gallons of Isopar® L, making the solvent ~29% more dilute than the required 7 gallons. Figure 24 shows infrared spectra of the as-received solvent in contrast with a sample of qualified solvent (with composition determined by chemical analysis to agree with process specifications). Comparison of peak heights at ~1100 and 1250 cm$^{(-1)}$ wavenumbers – attributed to modifier – confirms ~2 gal of excess Isopar® L.

Figure 24. Infrared Spectra of As-Received and Optimized Solvent



First inspection of the solvent revealed a distinct yellow color (Figure 25). Personnel first believed the yellow color may reflect the presence of 4-*sec*-butylphenol, a known degradation product from the modifier that results during routine operation. However, washing with dilute caustic (0.01 M NaOH) – up to 5 equal volume batch washed –did not remove the yellow color as expected, indicating that the yellow color may be due to an unknown impurity or degradation product. Personnel performed additional infrared spectroscopy of the material and compared to a spectrum for a batch of qualified solvent without discoloration. The difference spectrum shows that the yellow solvent has a carbonyl containing species (1727 cm-1). This species is probably due to oxidation of the secondary alcohol group on the Cs-7B modifier.

Figure 25. Photo of As-Received Solvent with Distinct Yellow Color.

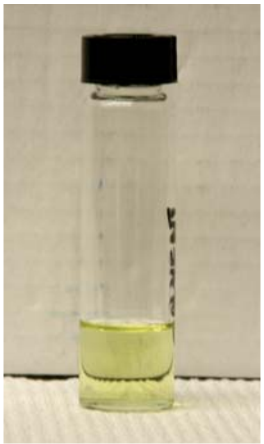
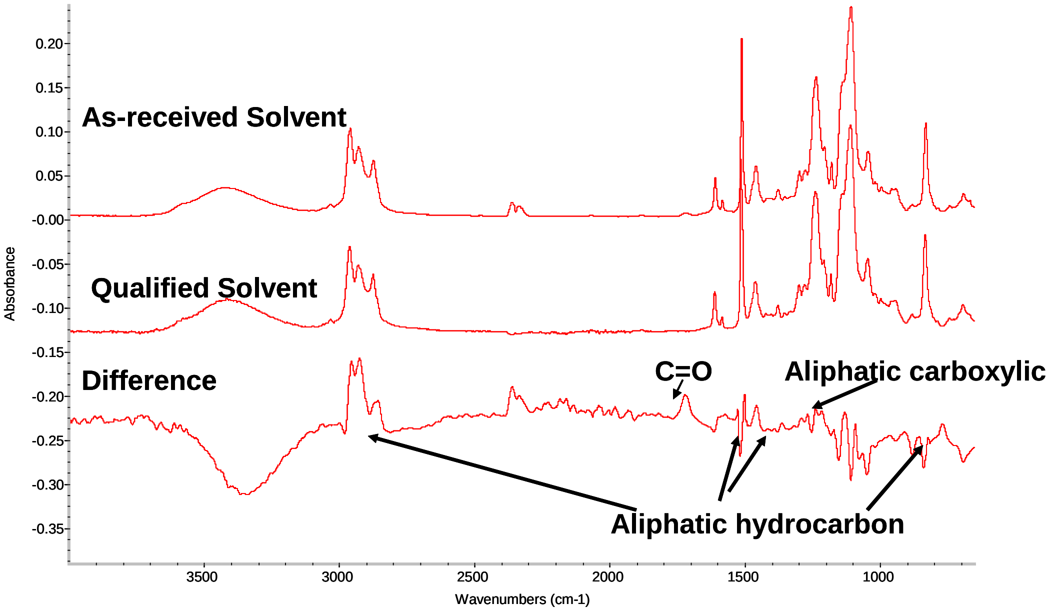


Figure 26. Identification of Impurity in As-Received Solvent.



Personnel next analyzed a sample of the concentrated solvent resulting from rotary evaporation of ~50% of the volume – i.e., removing a large portion of the Isopar® L. This allowed obtaining a more detailed infrared spectrum of the unknown impurity for direct comparison against a comparable spectrum of 4-*sec*-butylphenol. Figure 27 shows the two spectra.

SRNL decided to use a rotary evaporator (Figure 28) to distill off at reduced pressure the required amount of Isopar® L. Personnel cleaned the rotary evaporator ("rotovap") and installed a secondary cold trap (of dry-ice/isopropanol) connected to a high-capacity vacuum pump. Before any distillation of the actual solvent occurred, SRNL tested the rotovap with a small quantity of Isopar® L to identify the operating parameters and to verify no organic impurities transfer from operation of the equipment. The test involved distilling ~300 mL of Isopar® L from a ~ 1 L supply at ~75 ºC and 5-10 torr. Distillation started slowly at ~10 torr, and increased as the pressure decreased. It took approximately 10 minutes to distill over ~50% of the volume after reaching 10 torr operating pressure. Analysis of samples of the original and distilled material by SVOA analysis detected no contaminants.

Figure 27. Comparison of Impurity with 4-*sec*-butylphenol.

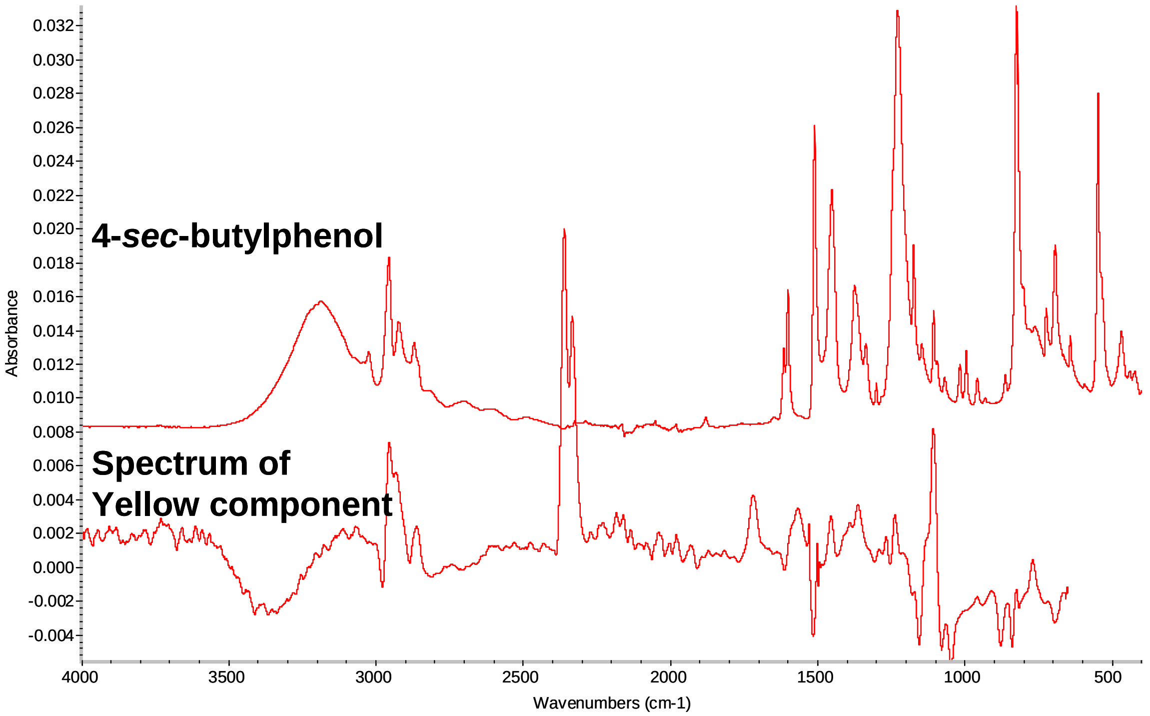
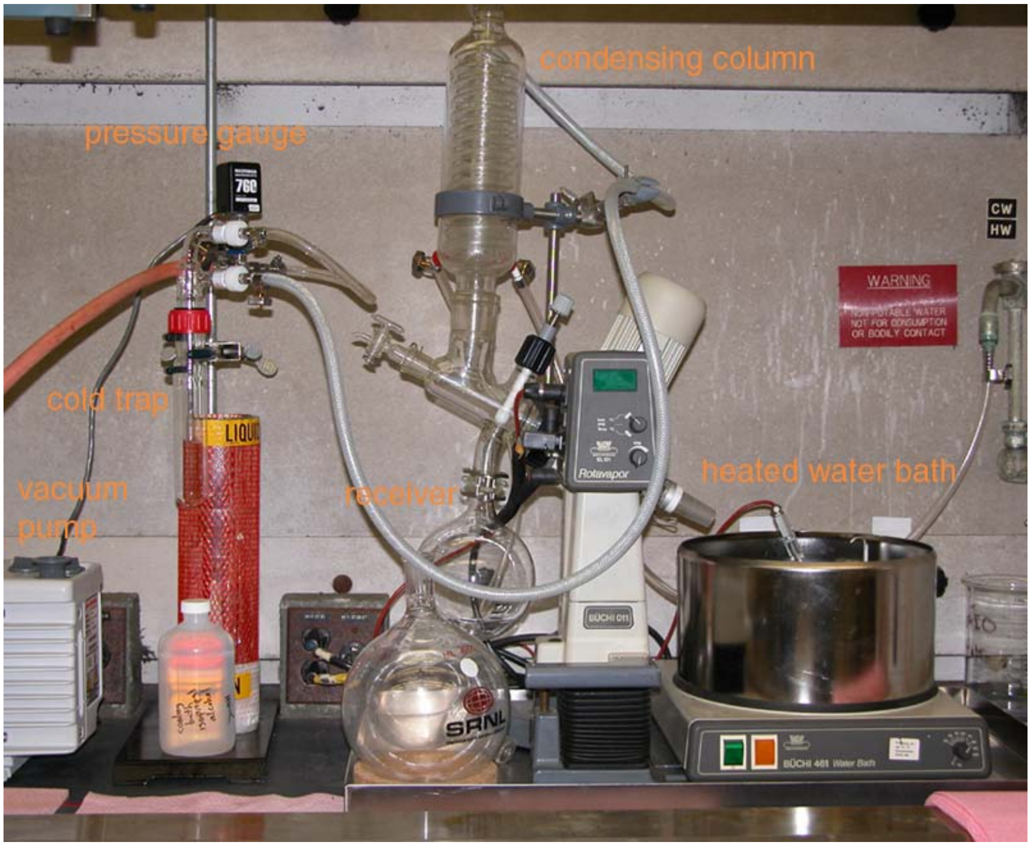


Figure 28. Rotary Evaporator.



Evaporation of the Parsons solvent proceeded almost exactly as the Isopar® L distillation. The temperature remained between 67 and 69 ºC throughout, and the pressure dropped to as low as 4 torr. Personnel used a 1L working volume flask, allowing removal about half the volume of each 1L batch of sample. Each distillation batch required about 30 minutes after the start of distillation to remove ~50% of the batch volume. Including set up time, it took about 1 hour to remove 500 mL of Isopar® L from each 1L of Parsons solvent.

SRNL calculated that approximately 6-7 L of Isopar® L needed removed from the Parsons solvent to return it to specifications. After removing ~6.5 L from the solvent, personnel poured the resulting distillation bottoms back into the solvent drum. They mixed the drum contents with an overhead stirrer for several minutes and collected a sample for density measurement. Using a calibrated 5 mL volumetric flask, 5 mL of reworked solvent was weighed in triplicate. Table 4 shows the results of the density measurement.

Table 4. Density Measurements.

	Density (g/mL)
Literature Value for solvent[18]	0.8516 (25 °C) 0.8557 (20 °C)
Measurement 1	0.8488 (23.3 °C)
Measurement 2	0.8492 (23.3 °C)
Measurement 3	0.8493 (23.3 °C)
Average	0.8491 (23.3 °C)

The average density is ~0.6% below the target value; SRNL declared the solvent to be within acceptable parameters. Personnel collected several analytical samples for conformational SVOA and HPLC analyses.

While collecting samples, personnel used a flashlight to inspect the solvent and noted the presence of solids in bottom of the drum (Figure 29). Personnel collected a sample of the material from the drum (Figure 30). This operation also identified the presence of entrained caustic in the solvent drum. A test showed that the solids settled quickly into the water layer. Using a Medium glass fritted filter (10-20 microns), personnel filtered a sample of the aqueous/organic slurry. The filtration proceeded at a reasonable rate and cleared all visible evidence of solids from the liquid layers. The brown solids looked like rust, and exhibited some magnetic behavior.

Figure 29. Solids in the Received Drum.

Figure 30. Contents from Bottom of Shipment Drum.

After noting the presence of solids in the solvent, SRNL decided to filter the solids from the liquid layers. Using a Medium glass frit (10-20 microns) and a large vacuum flask, personnel filtered all of the (now volume reduced) Parsons solvent. This operation lasted a total of 5 days. Personnel submitted a sample of the brown solids for digestion and ICP-ES analysis. The results of the ICP-ES analysis (Table 5) indicate the primary constituents are iron, aluminum, calcium, magnesium and sodium. The iron is rust (from color and magnetic observations), while the rest of the main constituents are probably from impure water. Reportedly, Parsons-SRS used process water for the pilot testing. If this water did serve as the primary source of these solids, the use of process water may represent an appreciable risk for routine operations.

Because of the presence of an unexpected organic impurity and the relatively frothy appearance of the solvent when agitated, researchers performed a series of surface tension measurements on various liquids used in the solvent extraction hydraulic demonstration at EDL. Using a Model 21 Fisher Surface Tensiomat (

Figure 31), personnel recorded several surface and interfacial surface tension measurements. This equipment employs the classical du Nouy ring design and this testing approximated the protocols defined in the ASTM standards.[19,20] Due to the accelerated schedule needs, personnel altered the recommended cleaning procedure for the platinum ring, omitting the heating step in a flame. Cleaning consisted of rinsing the ring twice with acetone and drying in a forced air stream.

Figure 31. Surface Tension Measurement Device.

Table 5 Elemental Composition of Solids in As-Received Solvent

Element[*]	Result (µg/g)	% Uncertainty
Al	37900	10.1
Ba	276	11.1
Ca	48700	10.0
Cr	201	26.1
Cu	1510	10.1
Fe	120000	10.0
Mg	15900	10.0
Mn	1810	10.0
Na	43500	10.0
Ni	545	13.4
P	8430	10.3
S	2510	10.1
Si	7850	10.0
Sr	111	11.5
Ti	264	10.1
Zn	868	10.4

[*] Elements present below detection limit omitted from table.

Personnel performed each measurement at least three times as listed in Table 6. For measurements of interfacial tension between two liquid phases, personnel first inserted the platinum ring into the lower phase than slowly added the lighter phase to avoid gross mixing.

Table 6. Surface and Interfacial Tension Measurements

Interface Measured	Surface Tension (dyn/cm) at ambient temperature (~22 °C)[3]
Parsons Reworked Solvent / Air	26.1, 26.1, 26.1
Optimized Solvent / Air	26.3, 26.3, 26.3
0.001 M HNO3 / Air	75.7, 74.8, 75.2, 74.5
Parsons Reworked / 0.001 M HNO3	12.9, 14.5, 14.6
Optimized Solvent / 0.001 M HNO3	15.3, 15.7, 15.2

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[3] Personnel failed to record the room temperature at time of measurement; this value reflects the typical measurement for the room.

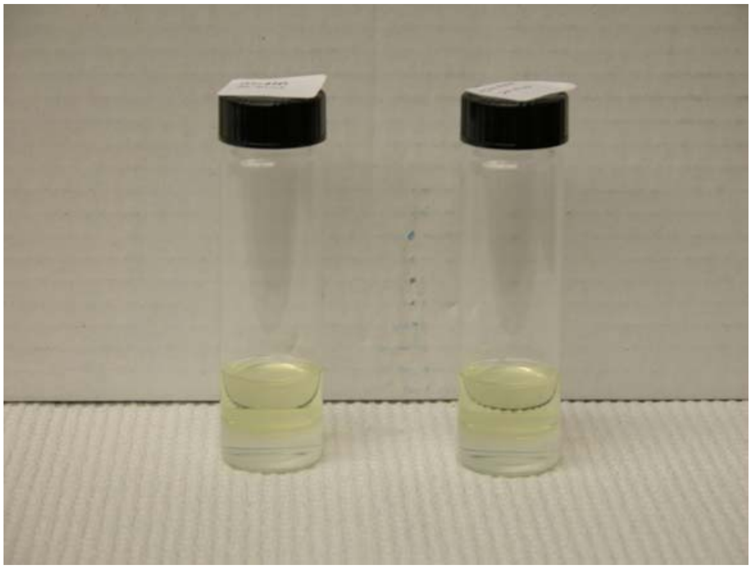
APPENDIX C – EMULSION RECOVERY

Initial operation resulted in near complete emulsification of the solvent inventory in the 0.001 M nitric acid due to use of an improperly-sized aqueous weir plate. The resulting emulsion proved extremely stable lasting for weeks if undisturbed. Personnel considered a number of methods to break the emulsion including the following.

- Centrifugation (1500 and 3600 rpm)
- Acid washes (0.01 M and 2 M nitric acid, 1:1 volume ratio of acid to emulsion)
- Caustic washes (0.01 M and 3 M NaOH, 1:1 volume ratio of caustic to dilute emulsion)
- Isopar® L wash (1:20 volume ratio of organic to dilute emulsion)
- Filtration through various media
- Heating (approximately 40 and ≥50 °C)

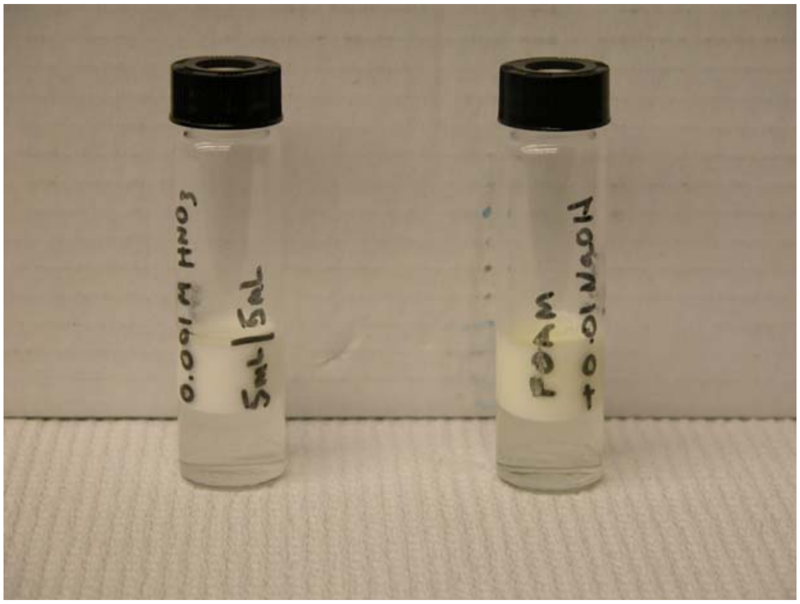
Direct centrifuging of the emulsion the day of formation resulted in good recovery of the solvent. However, effective separation only occurred at relatively high rotational speeds (1500 to 3600 rpm) after periods of 20 minutes. Operation at 10 minutes only marginally began coalescence. Figure 32 shows replicate samples after centrifuging for 20 minutes at 1500 rpm. The V-5 contactor technical representative expressed concern that the need for these prolonged centrifuge periods may indicate that the current equipment would prove ineffective in recovering the solvent.

Figure 32. Centrifugation of Emulsified Solvent.



The first acid and caustic washes used equal volumes of emulsion and either 0.01 M nitric acid or 0.01 M caustic. Neither proved effective as shown in Figure 33. This work occurred within hours of creating the emulsion. Approximately 2 weeks later, personnel also attempted washes using equal volumes of either 2 M nitric acid or 3 M caustic combined with the >90% more dilute, or "lean",[4] emulsion remaining from after the successful recovery of the initial emulsion. The washes did not result in quick recovery of the solvent. However, centrifuged (1500 rpm for 10 and 20 minutes) samples of the washes showed more efficient organic recovery than comparable centrifuged samples of the dilute emulsion. Addition of Isopar® L at a 1:20 volume ratio with the lean emulsion also provide relative poor or negligible recovery of the entrained organic – both with simple mixing and after centrifuging.

Figure 33. Acid and Caustic Wash of Emulsified Solvent.



Earlier studies showed that a Millipore 0.7 μ glass fiber material could effectively remove micron, and submicron, droplets of entrained organic from 0.001 M nitric acid. Personnel filtered an aliquot of the emulsion through such media using gravity flow. Filtration started within hours of creating the emulsion but filtration rates proved very slow, requiring several hours to finish for the limited volume examined. The filtrate – Figure 34 – showed two distinct liquid phases indicating complete breaking of the emulsion. While the results proved encouraging the slow filtration rates made recovery of the 40 gallons on emulsion impractical within the testing schedule. Over the next several days, personnel tried filtering samples with larger pore size media with the objective of obtaining similar recovery efficiency but at higher flux rates. Media testing include 0.1 μ Mott, 0.5 μ Pall M020, and 2.0 μ Pall M200 scintered stainless-steel. Flux rates proved only marginally better. Larger pore size correlated with faster rates but the greater thickness of the steel filter – compared to the glass filter – offset the gain in flux. Also, none of the filtrates from the steel media approached the clarity of that from the glass filter. Figure 35 shows the comparative performance for the various filter media. Testing also showed that applying trans-membrane pressure by vacuum or by pressure (to 40 psig) did not improve flux rates sufficiently to make this approach practical within the allowed schedule.

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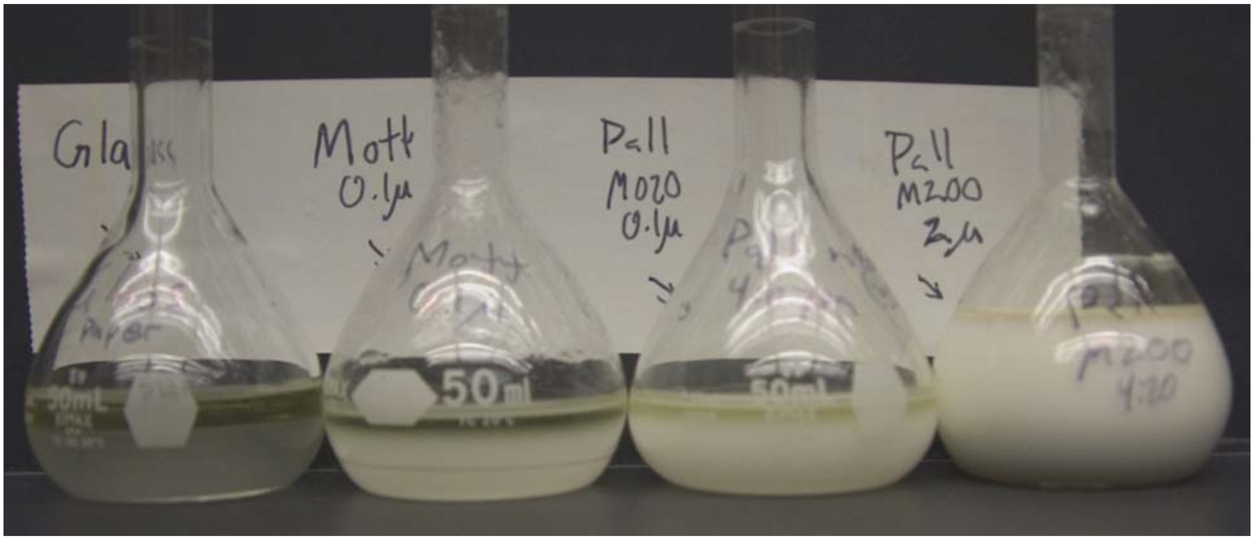
[4] The mass balance on the recovered solvent places the approximate organic content of the lean emulsion between ~4000 and 10,000 ppm at the time of these trials.

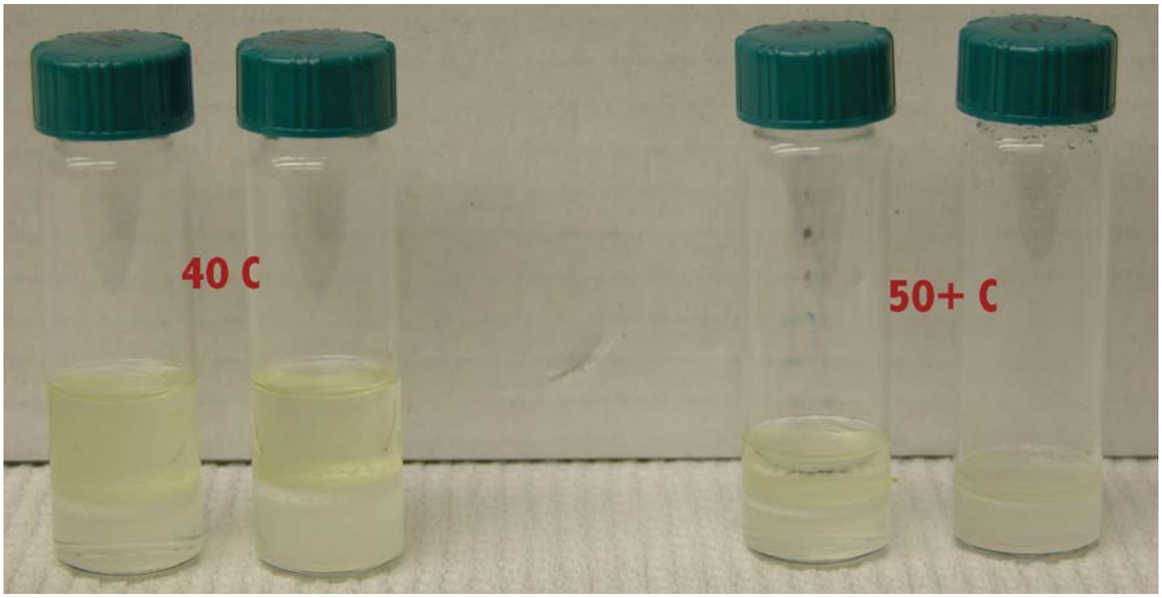
Figure 34. Filtration of Emulsified Solvent through Glass Fiber Material.

Figure 35. Filtration of Emulsified Solvent with Various Media.



Consultation with Parsons-SRS personnel indicated that minor emulsions formed in their testing separated more quickly when heated. SRNL personnel attempted heating of portions of the emulsified solvent approximately 24 hours after formation. Heating to 40 and ≥50 °C resulted in slow separation of the solvent from the 0.001 M nitric acid. Personnel selected 40 °C as a base temperature as this represents the highest allowed temperature for activities with solvent in the approved hazards assessment for the EDL where larger volume recovery efforts may occur. Maintaining the samples at 50 °C proved difficult with the rapidly assembled temperature bath and hence these samples experienced brief periods of heating well beyond the target value. Due to this concern, these samples were not included in the solvent returned to Parsons-SRS. Figure 36 shows replicate samples heated to 40 and to ≥ 50 °C. Close visual inspection of the samples shows the presence of three liquid phases, indicating incomplete separation. Figure 37 contains an expanded view of the samples heated to 40 °C. The intermediate density phase shows a deeper white color than the bulk of the emulsion and begins to assume a texture suggesting presence of solids.

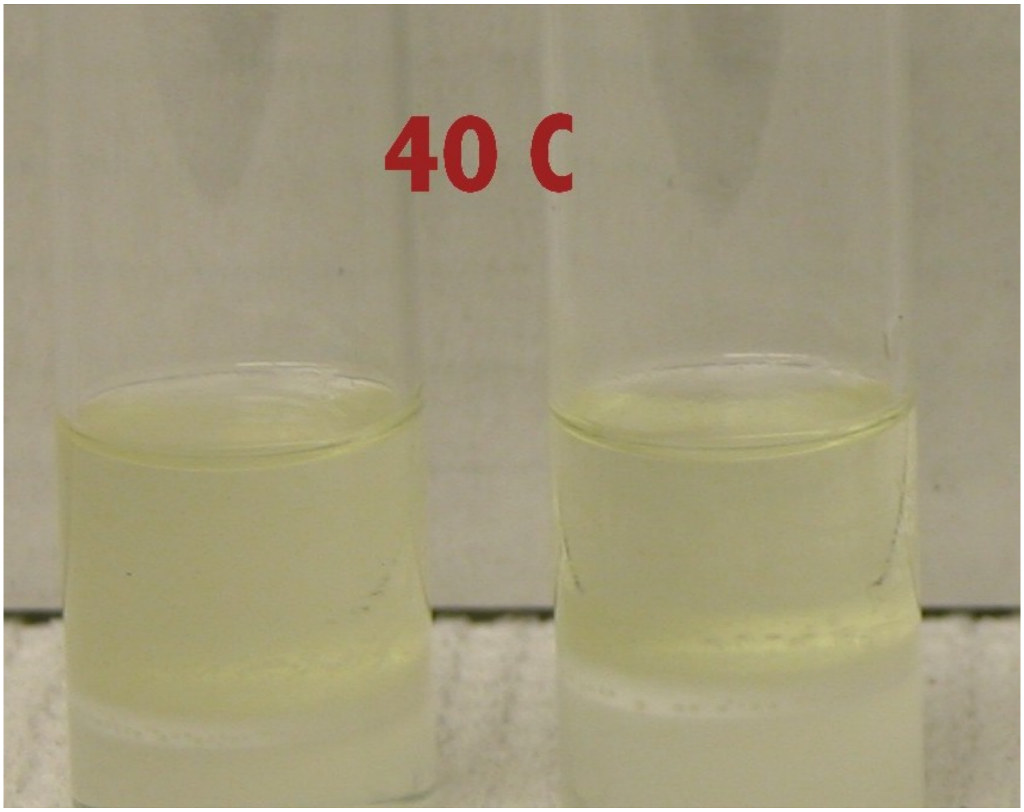
Figure 36. Heating of Emulsified Solvent.



Based on these results, SRNL personnel selected centrifugation as the most promising of the proposed methods for recovering the emulsified solvent. Personnel reconfigured the equipment to allow feed only from the Solvent Feed Tank and removed the rotameter from the solvent feed loop. [Personnel reasoned that removal of the rotameter would allow finer control of the flow to the contactor by reducing the pressure drop in the feed line and avoiding the use of two valves to control flow. In retrospect, recovery efforts likely did not need this equipment change.] They added approximately 0.5 gallons of the emulsified contents of the aqueous decanter to the contactor to establish liquid seals. Next, they added the prior contents of the contactor – i.e., the “richest” portion of the emulsified solvent to the Solvent Feed Tank. They also added several gallons of the contents from the upper region of the aqueous decanter – again, choosing to process the “richer” organic fraction of the contents first.

Operation of the contactor started at 2300 rpm. Personnel started gravity flow from the Solvent Feed Tank at minimal valve opening. Personnel observed initial discharge from the contactor to the aqueous decanter as expected– i.e., the emulsion contained primary aqueous and hence discharge occurred first from the aqueous overflow. Discharge to the Solvent Receipt Tank started approximately 3 minutes later although at substantially slower rates. The organic discharge was notably frothy but clearly had a high organic content. Personnel increased the rotational speed to 3000 rpm but soon thereafter reduced to 2700 rpm since the two discharged phases showed immediate loss in clarity at the higher speed. Personnel timed the collection of a sample from the aqueous discharge line and adjusted the valve from the Solvent Feed Tank to reach a flow rate of ~0.16 gpm – a value that would provide approximately 20 minute residence time for the contactor thus matching the prolonged centrifuge time that resulted in successful recovery of the emulsion samples.

Figure 37. Expanded View of Emulsifed Solvent Heated to 40 °C.



Operation over 90 minutes produced an estimated 0.8 gal of clarified solvent in the Solvent Receipt Tank. As clean solvent collected in the Solvent Receipt Tank, personnel removed the clarified organic from the system and placed in stainless-steel pails. Solvent that appeared insufficiently pure was recycled to the Solvent Feed Tank. Hence, the organic loop operated under partial recycle with manual removal of a "slip stream". [In retrospect, this removal of organic was not necessary to achieve recovery of the solvent. However, at the time of the recovery effort the primary objective involved recovery of solvent for return to Parsons-SRS since the solvent represents a critical resource for planned V-10 contactor testing.]

At that point, personnel added an additional batch of the emulsified material from the decanter to the Solvent Feed Tank to re-establish inventory. Personnel adjusted the contactor operational speed between 2700 and 3400 over the subsequent 90 minutes in hopes of obtaining faster recovery. After 3 hours of operation, personnel estimated the recovered volume of solvent as 1.89 gal – or 29% of the starting inventory. The organic recovery rate slowed and personnel added a third batch of emulsified feed from the decanter to the Solvent Feed Tank. The organic recovery rate – or discharge rate to the Solvent Receipt Tank – remained slow over the next 75 minutes.

Personnel reconfigured to allow feed directly from the aqueous decanter and adjusted the flow rate to just above the zero point of the rotameter (i.e., below the scale of the sight glass). Operation continued under these conditions for the duration of the effort. After 7 total hours of operation, flow rate of purified organic in the discharge line to the Solvent Receipt Tank slowed to exceedingly low rates. Personnel calculated the removed volume as 2.25 gal. They reasoned that the contactor should contain an additional ~ 3 gal of purified solvent – i.e., as operation proceeded, purified solvent had to accumulate in the contactor internals.

Personnel stopped operation and emptied the contactor to the Solvent Receipt Tank. They also emptied the Solvent Feed Tank contents to the Receipt Tank. Unfortunately, personnel failed to inventory each volume of organic separately. By measuring the depth of the solvent in the two containers, personnel calculated the total volume of clarified solvent at ~5.95 gallons (i.e., ~90.5% recovery of the starting inventory).

The recovery rate averaged ~0.014 gpm versus a typical gross flow rate of 0.16 gpm. This suggests the system operated on average at an organic-to-aqueous ratio of 1:10. This value compares favorably with the approximate 1:6 volume ratio (i.e., ~6.6 gal of solvent emulsified in 40 gal of 0.001 M nitric acid) for the solvent contained within the entire contents of the aqueous decanter. Hence, the overall recovery efficiency proved relative high for the operating time.

From another perspective, the typical gross flow rate of 0.16 gpm for 7 hours equates to processing ~67.2 gal. Hence, for a total system volume of 46.6 gal, the system only operated for ~1.5 residence times. Hence, 90% separation within 1.5 residence times seems a rather efficient value.

Although emulsifying the solvent represented a substantial obstacle, the overall events proved beneficial and enlightening. Personnel could easily accommodate a modified version of this recovery protocol within the design for either the MCU or SWPF. By operating the system under a controlled sequence of total recycle at low flow rates, personnel may achieve recovery of solvent from any event that leads to emulsification within a practical time period.

APPENDIX D – ANALYTICAL DATA

Table 7. Gas Chromatography and Turbidity Data.

				GC/MS Data			Total Implied Solvent based on Modifier		
Sample ID	Comment	Analysis / Sample Time	LIMS #	Isopar (mg/kg)	Modifier (mg/kg)	TOA (mg/kg)	(mg/kg)	time (h)	turbidity NTU
Test 1 Sample 2	decanter discharge	3/28 1944	3-217273					5.6	14.5
Test 1 Sample 1	decanter discharge	3/28 1715	3-217252	3.7	31		104.5	5.6	
Test 2A Sample 1	decanter discharge	3/30 1410	3-217254	1	3.5		11.8	5.6	
Test 2A Sample 2	decanter discharge	3/30 1703	3-217255					5.6	10.44
Test 2A Decanter Outlet		3/30 0935						1	21.9
Test 2A Decanter Outlet		3/30 1035						2	19.27
Test 2A Decanter Outlet		3/30 1135						3	12.62
Test 2A Decanter Outlet		3/30 1235						4	12.79
Test 2A Decanter Outlet		3/30 1335						5	18.1
Test 2A Contactor Outlet		3/30 0935	3-217474	6.2	65	<1	219.1	1	20.3
Test 2A Contactor Outlet		3/30 1035	3-217475	8.9	76	<1	256.2	2	10.05
Test 2A Contactor Outlet		3/30 1135	3-217476	5.1	57	<1	192.1	3	11.34
Test 2A Contactor Outlet		3/30 1235	3-217477	50	180	<1	606.7	4	18.37
Test 2A Contactor Outlet		3/30 1335	3-217478	7.3	110	<1	370.7	5	17.5
Test 3A 0 hr	decanter outlet	3/28 1715						0	14.2
Test 3A 2 hr	decanter outlet	3/28 1955						2	
Test 3A 4 hr	decanter outlet	3/29 0121						4	39
Test 3A Sample 2	decanter outlet	3/29 0110	3-217259					5.6	39.3
Test 3A Sample 1	decanter outlet	3/29 0055	3-217258	4.8	41		138.2	5.6	42.5
Test 4 2000 Decanter		3/30 0102	3-2172xx					1	14.5
Test 4 2100 Decanter		3/30 0123	3-2172xx					2	26.5
Test 4 2200 Decanter		3/30 0143	3-2172xx					3	29.6
Test 4 Sample 1	decanter discharge		3-217262		85		286.5	3.6	
Test 4 Sample 2	decanter discharge	3/30 0051	3-217263					3.6	22
Test 4 2000 Contactor Outlet		3/30 0113	3-2172xx					1	32.2
Test 4 2100 Contactor Outlet		3/30 0133	3-2172xx					2	31.2
Test 4 2200 Contactor Outlet		3/30 0154	3-2172xx					3	32.9
Test 5 initial	decanter contents at start	3/29 0850						0.3	41.1
Test 5 initial 2	decanter contents at start	3/29 0850	3-217479	12	98	<1	330.3	0.3	40.5
Test 5 0830	contactor discharge	3/29 0830						0	25.8
Test 5 0930	decanter discharge	3/30 0002	3-217322					1	26
Test 5 1030	decanter discharge	3/29 1030	3-217480	7.2	80	<1	269.6	2	
Test 5 1030	decanter discharge	3/29 1841	3-217323					2	21.2
Test 5 1030	contactor discharge	3/29 1030						2	22.1
Test 5 1130	decanter discharge	3/30 0013	3-217324					3	18.5
Test 5 1230	decanter discharge	3/29 1230	3-217481	5.9	75	<1	252.8	4	
Test 5 1230	decanter discharge	3/29 1852	3-217325					4	16.7
Test 5 1230	contactor discharge	3/29 1230						4	21.1
Test 5 1330	decanter discharge	3/30 0024	3-217326					5	15.6
Test 5 1430	decanter discharge	3/29 1430	3-217482	7	77	<1	259.5	6	
Test 5 1430	decanter discharge	3/29 1904	3-217327					6	15.6
Test 5 1430	contactor discharge	3/29 1430						6	20.5
Test 5 1530	decanter discharge	3/30 0036	3-217328					7	19
Test 5 Sample 1	decanter discharge	3/29 1805	3-217264	11	240		808.9	7.4	
Test 5 Sample 2	decanter discharge	3/29 1823	3-217265					7.4	19.1
Fibermat 1	from V4 (?27 minutes after start)	3/30 ????	3-217358					0.0	
Fibermat 2	after 27 minutes from valve V5	3/30 1618	3-217360					0.5	
Fibermat 3	after 50 minutes from valve V5	3/30 1645	3-217359					0.8	
Aqueous Baseline 1	Drum of aqueous from emulsifed solvent after recovery	3/28 1626	3-217248						
Aqueous Baseline 2	Drum of aqueous from emulsifed solvent after recovery	3/28 1000	3-217249	980					
Test Rig Baseline 1	Decanter at start of Test 1	3/28 1633	3-217250						
Test Rig Baseline 2	Decanter at start of Test 1	3/28 1115	3-217251	21					

Sample loading low
Results may be unreliable

not applicable

Table 8. Droplet Size Data.

Sample ID	LIMS #	Particle Size Data (Volume fraction of particles smaller than indicated diameter in microns)													
		0.972	1.156	1.375	1.635	1.945	2.312	2.75	3.27	3.889	4.625	5.5	7.8	11	13.1
Test 1 Sample 2	3-217273	0	0	0.0109	0.0856	0.1887	0.282	0.4458	0.7642	0.9776	1	1	1	1	1
Test 1 Sample 1	3-217252														
Test 2A Sample 1	3-217254														
Test 2A Sample 2	3-217255	0	0	0	0.0014	0.011	0.0394	0.1166	0.2883	0.554	0.8109	0.9502	1	1	1
Test 2A Decanter Outlet															
Test 2A Decanter Outlet															
Test 2A Decanter Outlet															
Test 2A Decanter Outlet															
Test 2A Decanter Outlet															
Test 2A Contactor Outlet	3-217474														
Test 2A Contactor Outlet	3-217475														
Test 2A Contactor Outlet	3-217476														
Test 2A Contactor Outlet	3-217477														
Test 2A Contactor Outlet	3-217478														
Test 3A 0 hr															
Test 3A 2 hr		0.0842	0.0901	0.097	0.1076	0.1285	0.178	0.2867	0.459	0.637	0.7737	0.8622	0.9476	0.9865	0.9972
Test 3A 4 hr		0.1043	0.1186	0.1448	0.1902	0.2566	0.3571	0.5272	0.7532	0.927	0.9898	1	1	1	1
Test 3A Sample 2	3-217259	0.1331	0.1419	0.1686	0.2281	0.3039	0.3939	0.5411	0.765	0.9428	0.9945	1	1	1	1
Test 3A Sample 1	3-217258														
Test 4 2000 Decanter	3-2172xx	0	0	0	0	0.0074	0.0378	0.1219	0.2915	0.5486	0.8161	0.9614	1	1	1
Test 4 2100 Decanter	3-2172xx	0	0	0	0.0046	0.0162	0.0475	0.1225	0.2691	0.4876	0.7247	0.892	0.9921	1	1
Test 4 2200 Decanter	3-2172xx	0	0	0	0	0.0066	0.0306	0.0987	0.2418	0.4645	0.7166	0.8987	0.9959	1	1
Test 4 Sample 1	3-217262														
Test 4 Sample 2	3-217263	0	0	0	0	0	0.0113	0.0551	0.1476	0.2934	0.4872	0.7041	0.9581	1	1
Test 4 2000 Contactor Outlet	3-2172xx	0	0	0	0	0	0.0145	0.0647	0.1879	0.4125	0.7008	0.9105	1	1	1
Test 4 2100 Contactor Outlet	3-2172xx	0	0	0	0	0	0.0078	0.04	0.1343	0.3201	0.5675	0.7854	0.9716	1	1
Test 4 2200 Contactor Outlet	3-2172xx	0	0	0	0	0	0.008	0.0412	0.1266	0.284	0.5007	0.7261	0.9553	1	1
Test 5 initial															
Test 5 initial 2	3-217479														
Test 5 0830															
Test 5 0930	3-217322	0	0.0051	0.0173	0.0416	0.0883	0.1817	0.3654	0.6354	0.8726	0.9779	0.9989	1	1	1
Test 5 1030	3-217480														
Test 5 1030	3-217323	0.0805	0.1044	0.136	0.1788	0.2474	0.3808	0.6069	0.8356	0.9591	0.9946	1	1	1	1
Test 5 1030															
Test 5 1130	3-217324	0	0.0012	0.0075	0.0203	0.0441	0.0967	0.2225	0.4643	0.7459	0.9274	0.9888	1	1	1
Test 5 1230	3-217481														
Test 5 1230	3-217325	0.0177	0.0321	0.0584	0.091	0.1332	0.213	0.3937	0.675	0.8959	0.9814	0.9989	1	1	1
Test 5 1230															
Test 5 1330	3-217326	0	0	0.0122	0.0557	0.1141	0.1964	0.362	0.6651	0.9303	0.9981	1	1	1	1
Test 5 1430	3-217482														
Test 5 1430	3-217327	0	0	0.0231	0.1018	0.1645	0.2291	0.3875	0.7519	0.9804	1	1	1	1	1
Test 5 1430															
Test 5 1530	3-217328	0.1952	0.2277	0.2588	0.3104	0.4097	0.5733	0.7471	0.8756	0.9472	0.9817	0.9965	1	1	1
Test 5 Sample 1	3-217264														
Test 5 Sample 2	3-217265	0.091	0.1141	0.1541	0.2271	0.3326	0.4874	0.6935	0.8867	0.9822	0.999	1	1	1	1
Fibermat 1	3-217358	0.1053	0.131	0.1793	0.2612	0.356	0.4708	0.6512	0.9504	0.9973	1	1	1	1	1
Fibermat 2	3-217360	0	0.0014	0.0058	0.0236	0.0886	0.2476	0.4886	0.7345	0.9143	0.9879	1	1	1	1
Fibermat 3	3-217359	0.1072	0.1142	0.124	0.1474	0.2095	0.3327	0.4815	0.6112	0.7133	0.8019	0.8878	0.9485	0.9828	0.9977
Aqueous Baseline 1	3-217248	0.0486	0.06	0.0746	0.1009	0.138	0.165	0.177	0.1847	0.1927	0.2063	0.2292	0.2727	0.3052	0.336
Aqueous Baseline 2	3-217249														
Test Rig Baseline 1	3-217250	1	1	1	1	1	1	1	1	1	1	1	1	1	1
Test Rig Baseline 2	3-217251														

Sample loading low
Results may be unreliable

not applicable

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