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1/6TH SCALE STRIP EFFLUENT FEED TANK - MIXING RESULTS USING MCU SOLVENT

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EXECUTIVE SUMMARY

The purpose of this task was to determine if mixing was an issue for the entrainment and dispersion of the Modular Caustic Side Solvent Extraction (CSSX) Unit (MCU) solvent in the Defense Waste Processing Facility (DWPF) Strip Effluent Feed Tank (SEFT). The MCU strip effluent stream containing the Cs removed during salt processing will be transferred to the DWPF for immobilization in HLW glass. In lab-scale DWPF chemical process cell testing, mixing of the solvent in the dilute nitric acid solution proved problematic, and the Savannah River National Laboratory (SRNL) was requested to perform scaled SEFT mixing tests to evaluate whether the problem was symptomatic of the lab-scale set-up or of the solvent. The solvent levels tested were 228 and 235 ppm, which represented levels near the estimated DWPF solvent limit of 239 ppm in 0.001M HNO_3 solution. The 239 ppm limit was calculated by Norato in X-CLC-S-00141.

The general approach for the mixing investigation was to:

- Investigate the use of fluorescent dyes to aid in observing the mixing behavior. Evaluate and compare the physical properties of the fluorescent dyed MCU solvents to the baseline Oak Ridge CSSX solvent. Based on the data, use the dyed MCU solvent that best approximates the physical properties.
- Use approximately a 1/6th linear scale of the SEFT to replicate the internal configuration for DWPF mixing.
- Determine agitator speed(s) for scaled testing based on the DWPF SEFT mixing speed.
- Perform mixing tests using the 1/6th SEFT and determine any mixing issues (entrainment/dispersion, accumulation, adhesion) through visual observations and by pulling samples to assess uniformity.

The mixing tests used MCU solvent fabricated at SRNL blended with Risk Reactor DFSB-K43 fluorescent dye. This dyed SRNL MCU solvent had equivalent physical properties important to mixing as compared to the Oak Ridge baseline solvent, blended easily with the MCU solvent, and provided an excellent visual aid.

Testing involved agitator speeds of 230 revolutions per minute (RPM) and 422 RPM. The 230 RPM value was based on power per unit volume criteria, while the 422 RPM used tip speed criteria and were scaled using the DWPF SEFT operating speed of 67 RPM. In general, both speeds satisfied the objective of MCU solvent entrainment and dispersion. The agitator speed of 230 RPM scales from 37 to 67 RPM for the DWPF SEFT, which bounds the present operating speed of the DWPF SEFT. Although mixing speed provided sufficient dispersion, problems noted included adhesion of MCU solvent to tank internals, accumulation of MCU solvent in the sparger and pump cavities, and evidence of a slight floating layer of MCU solvent above 7845 gallons that stayed in the quaternary where the baffles are 106.45° apart. To avoid surface air entrainment, SRNL recommends the DWPF SEFT tank should have a lower operating tank level limit of 34.2 inches or 2410 gallons.

When the 422 RPM test is scaled using the P/V correlation, a DWPF SEFT operating speed of 125 RPM is obtained, which is outside the SEFT operating speed. Once again, dispersion was sufficient; however, aerosol generation was observed during the pump down portion of testing. The aerosols are likely the result of either air introduction via surface entrainment and dispersion via the impeller or the vapor pressure on the back side of the impeller being less than that of the fluid. If aerosol generation is a concern for DWPF operations, SRNL recommends additional testing be performed to determine the source of aerosol generation. Additional guidance could be provided in the operation

of the DWPF SEFT to avoid this potential problem. Solvent adhering to the tank internals were also present during this test. .

Accumulation of solvent on the tank walls and/or on the internals could present an operating problem if the accumulated material dislodges and is transferred to the SRAT as a slug of organics. SRNL recommends further testing to determine if the MCU solvent will continuously buildup based on the operating cycle and conditions of the DWPF SEFT. Multiple cycles at the latest anticipated MCU solvent concentration are recommended at 230 RPM.

The results in this test are based on using a 0.001 M HNO_3, solution and MCU solvent only. If contaminates are present in the strip effluent stream or in the SEFT, the results and recommendations made for the dispersion of the MCU solvent in the DWPF SEFT vessel may no longer be valid.

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LIST OF ACRONYMS

ACTL	Aiken County Technology Laboratory
ADS	Analytical Development Section
CSSX	Caustic Side Solvent Extraction
Coliwasa	Composite Liquid Waste Sampler
DWPF	Defense Waste Process Facility
HPLC	High Performance Liquid Chromatography
MCU	Modular CSSX Unit
ORNL	Oak Ridge National Laboratory
P/V	Power Per Unit Volume
RPM	Revolutions Per Minute
SEFT	Strip Effluent Feed Tank
SRAT	Sludge Receipt and Adjustment Tank
SRNL	Savannah River National Laboratory
SVOA	Semi-Volatile Organic Analysis
SWPF	Salt Waste Processing Facility
WPT	Waste Processing Technology

1.0 INTRODUCTION AND BACKGROUND

Laboratory scale mixing tests[1] performed at the Aiken County Technology Laboratory (ACTL) showed that the mechanical mixing (e.g. stir bar) used to entrain and disperse the Modular Caustic Side Solvent Extraction Unit (MCU) optimized solvent[2] in a 0.001M HNO_3 solution was not effective in producing a uniform dispersion of the solvent in the HNO_3 solution. Some of the solvent either agglomerated (after being dispersed) or was never entrained into the mixture and was observed to float on top of the mixture. During the laboratory scale tests, the MCU solvent also collected in the dip tube (for pumping out the contents), where the less dense solvent displaced the HNO_3 solution, over time. This suggested that internal components of the mixing system could accumulate the MCU solvent.

In the Defense Waste Process Facility (DWPF), the Strip Effluent Feed Tank (SEFT) will receive this waste stream from either the MCU or the Salt Waste Processing Facility (SWPF) from the Caustic Side Solvent Extraction (CSSX) process. The contents in the SEFT are mechanically agitated with a centrally located impeller system, rotating at 68 RPM. The impeller system consists of a 3-blade hydrofoil axial flow impeller located about 2/5th of the way from the bottom to the top of the tank and a 4-blade flat blade radial flow impeller located near the bottom of the tank. Additional details of the SEFT are provided in Section 2.1.

It has been suggested that MCU solvent will not be dispersed throughout the SEFT and will also agglomerate in the transfer pump and air sparger, due to the difficulties observed in the laboratory scale mixing tests[1]. If lack of dispersion or agglomeration occurs in the SEFT, non-homogeneous feed concentrations of the MCU solvent will be fed to the Sludge Receipt and Adjustment Tank (SRAT). The current DWPF operating safety basis assumes that the organic solvent is uniformly dispersed when fed to the SRAT to maintain flammability controls.

DWPF issued a Technical Task Request[3] to the Savannah River National Laboratory (SRNL) to investigate the mixing behavior in the SEFT. The work performed in this task is covered by the Task Technical and Quality Assurance Plan written by Hansen[4]. The primary objective of this task was to investigate the degree to which a floating layer of MCU solvent can be entrained and dispersed in an aqueous phase of a 0.001M HNO_3 solution upon initiation of mechanical agitation, at various agitator speeds, and during pump down. In order to achieve this objective, the following tasks were completed and will be discussed in more detail in the following sections;

- Modify the existing 1/6th scale SRAT mixing tank at ACTL for SEFT operations.
- Determine the upper and lower 1/6th scale mixing speeds.
- Use MCU solvent fabricated at SRNL (without the BobCalixC6) for testing. Determine if visual dyes can be used with the MCU solvent without impacting the solvent physical properties.
- Perform baseline water runs to determine if there are any operational issues with water alone.
- Perform mixing tests at two different mixing speeds containing 228 ppm MCU solvent in 0.001M HNO_3. Quantify mixing results at full tank conditions and report any mixing issues that could be related to full scale SEFT operations.

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[1] Baich, M. A., Herman, C. C., Eibling, R. E., Williams, M. F., and Smith, F. G., "*Sludge Batch 4 Simulant Flowsheet Studies with ARP and MCU: Impact of MCU Organics*", WSRC-TR-2004-00230, Revision 0, July 2005

[2] MCU solvent was composed of 0.007*M* calix arene-bis (t-octyl benzo-crown-6), known as BOBCalixC6 or extractant; 0.75 *M* 1-(2,2,3,3-tetrafluoropropoxy)-3-(4-*sec*-butylphenoxy)-2-propanol, known as Cs-7SB or modifier; and 0.003 *M* tri-n-octylamine (TOA) or supprssor in the Isopar®L or diluent.

[3] Norato, M. A., "Technical Task Request: Investigation of the Dispersion of Entrained CSSX Solvent in the SEFT (U)", HLW/DWPF/TTR-2005-0017, Rev. 0, June 28, 2005

[4] Hansen, E. K., "*Task Technical and Quality Assurance Plan - Investigation of the Dispersion of Entrained CSSX Solvent in the SEFT*", WSRC-RP-2005-01689, Rev. 0, June 28, 2005

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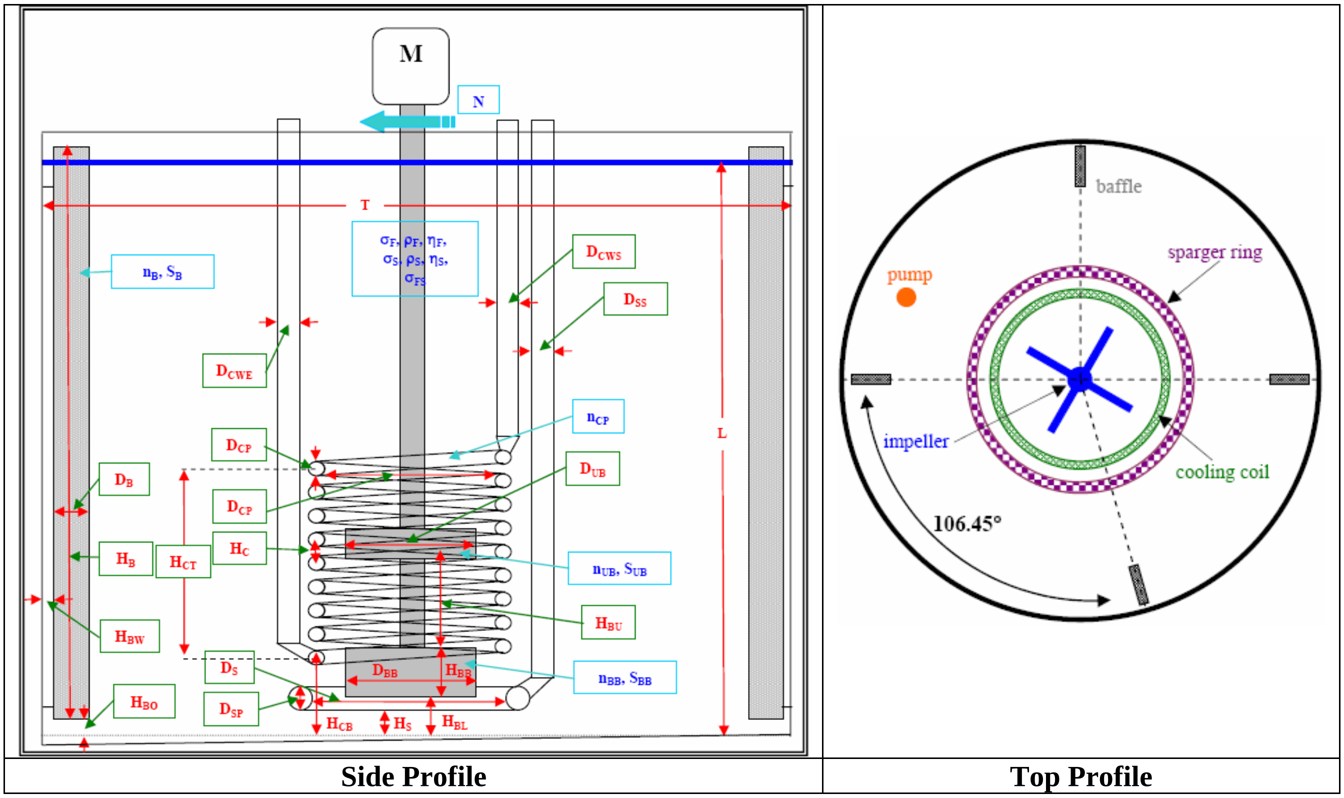
2.0 APPROACH

2.1 Fabricate 1/6th Scale SEFT

The primary processing mixing vessels used in DWPF have the same tank shell (diameter, slope tank bottom, mercury sump, etc.), but have different internal components (agitator assembly, baffles, cooling/heating coils, pumps, level detection, etc) and operating parameters (agitator speed, heating/cooling, air flow, etc.). An existing 1 to 6.21 (to be called 1/6th) linearly scaled[5] SRAT mixing vessel, used in previous testing[6, 7] at ACTL, was selected for modification. This same ratio was used for the SEFT internal components. The vessel was modified to the SEFT configuration by removing the existing coil assembly and fabricating/installing a single coil assembly, baffles, transfer pump, and sparger. The internal components of the 1/6th SEFT were scaled linearly from the full scale SEFT. The layout of the SEFT is shown in Figure 2-1 and the top section profile shows the tank broken into 4 different sections, where one quaternary having baffles 106.45° apart and the other quaternaries with baffles 90° or less apart. The dimensions and operation conditions from the reference drawing of the full scale SEFT and the dimensions for the actual 1/6th SEFT are specified in Table 2-1.



Side Profile | Top Profile

Figure 2-1 SEFT Internal Components and Configuration

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[5] Linearly scaled is defined as linearly scaling a single dimension (length) from one scale to another scale.

[6] Marinik, A. R. and Stone, M. E., "*Evaluation of Mixing in the Slurry Mix Evaporator and Melter Feed Tank (U)*", WSRC-TR-2004-00436, Revision 0, August 2004

[7] Stone, M. E. and Marinik, A. R., "*Small Scale Mixing Tests for the DWPF Chemical Process Cell Vessels (U)*", WSCR-TR-2004-00074, Revision 0, March 2004

Table 2-1 SEFT Dimensions, Impellers, and Operating Conditions of the SEFT

Variable	Description	Full Scale	1/6th	Variable	Description	Full Scale	1/6th
N	Rotation speed	68 RPM	Upper 422 RPM	T	Inside Tank Diameter	144 inches	23.2 inches
			Lower 230 RPM	n_{B}	Number of baffles – not all 90 degrees apart	4	4
L	Height of liquid	Up to 136 inches	Up to 21.9 inches	D_{B}	Baffle Width	12 inches	1.93 inches
S_{B}	Baffle Shape	Rectangular	Rectangular	T_{B}	Baffle thickness (not shown)	1 inches	0.125 inches
H_{B}	Baffle Height	150 inches	24.2 inches	H_{BO}	Baffle bottom wall off-set	3.5 inches	0.56 inches
H_{BW}	Baffle side wall off-set	2 inches	0.32 inches	D_{CWS}	Coil coiling water supply pipe O.D.	3.5 inches	**0.551 inches**
D_{CWE}	Coil coiling water exit pipe O.D.	3.5 inches	**0.551 inches**	D_{CP}	Cooling coil inside diameter	50.125 inches	8.1 inches
D_{CP}	Cooling coil pipe outside diameter	2.375 inches	**0.375 inches**	H_{CT}	Total height of coils	70 inches	11.3 inches
H_{C}	Distance between cooling coil centerlines	3.5 inches	0.56 inches	n_{CP}	Number of coils	21	21
H_{CB}	Distance bottom coil from bottom of tank	14 inches	2.3 inches	D_{S}	Sparger inside diameter	62.5 inches	10.1 inches
D_{SP}	Sparger Outside Pipe Diameter	3.5 inches	**0.551 inches**	D_{SS}	Sparger air supply pipe O. D.	3.5 inches	**0.551 inches**
H_{S}	Distance sparger ring from bottom of tank	5 inches	0.81 inches	n_{BB}	Number of blades on bottom impeller	4	4
S_{BB}	Shape of bottom impeller	flat blade	flat blade	H_{BB}	Height of bottom impeller	9.75 inches	1.57 inches
D_{BB}	Diameter of bottom impeller	36 inches	5.8 inches	T_{BB}	Thickness of bottom blade	Not provided	0.125 inches
H_{BL}	Distance between bottom of bottom impeller to bottom of tank.	7 inches	1.1 inches	n_{UB}	Number of blades on upper impeller	3	3
S_{UB}	Shape of upper impeller	Hydrofoil	Hydrofoil	H_{BU}	Distance between bottom and upper (1/3 up impeller)	43 inches	6.9 inches
D_{UB}	Diameter of upper impeller	36 inches	5.8 inches	V_{max}	Maximum tank volume	9,600 gallons	40.1 gallons
P_{down}	Pump down rate	12 GPM	-	V_{min}	Minimum tank volume	2,000 gallons	8.4 gallons

Reference Drawings:	AXC-21204, 44314, 1	General Arrangement 144 RCT Process Cell (Spare)
	AXC-21204, 44314, 2	Vessel Orientations 144 RCT Process Cell
	W752280	Philadelphia Mixer Outline Dimension Sheet
	AXC-21204, 44307, 3	Nozzles / Studs / Dowel Pin Locations
	AXC-21204, 44307, 4	Shell and Baffle Details 144 RCT Process Cell
	AXC-21204, 44307, 37	Cooling Coil and Air Sparger 144 RCT Process Cell
	AXC-21204, 44307, 38	Supports for Cooling Coil 144 RCT Process Cell
	E-24122	Transfer Pump

The bolded items in Table 2-1 are items that were not linearly scaled using the 6.21 ratio, and the potential impact from not being linearly scaled is provided in Table 2-2. Information in Table 2-2 also provides details on the number and size of the sparger holes and the transfer pump suction line. None of the identified items seem to be show stoppers with respect to this mixing task. The cross-sectional openings of the sparger and transfer pump have the largest potential impact, but they are secondary objectives (sampling, holdup) in this task.

Table 2-2 Internal Components Not to 1/6th Scale

Component	Units	Full Scale	1/6th Scale		Potential Impact
			1 to 6.21 ratio	Actual	
Sparger Pipe Diameter	Inches	3.5	0.564	0.551	Diameter is slightly smaller. • Less flow impedance to bottom impeller discharge. • Baffling is slightly less effective.
Sparger Ring – Sparger Hole Diameter	Inches	0.1875	0.03125	0.0752	Diameter is larger. • Size of the hole impacts the ability for the solvent droplets to flow into the opening and be captured. • Sizing may not be appropriate for solvent capture.
Sparger Ringer – Sparger Holes	-	64	-	16	Less holes. • Number of holes will provide a lower quantity of captured solvent, due to less cross-sectional area available. • Sizing may not be appropriate for solvent capture.
Cooling Coil – Pipe Diameter	Inches	2.375	0.3825	0.3750	Diameter is slightly smaller. • Less flow impedance to bottom impeller discharge. • Slightly more flow through coils for a given ΔP. • Baffling is slightly less effective.
Pump – Priming Line Outside Diameter	Inches	1.660	0.267	0.25	Diameter is slightly smaller. • Baffling is slightly less effective.
Pump – Priming Line Inside Diameter	Inches	1.38	0.22	0.18	Diameter is slightly smaller. • None, unless suction line fills during testing.
Pump – Priming Line Suction Inlet	Inches	1.0625	0.171	0.171	Diameter is scaled properly. • Size of the hole impacts the ability for the solvent droplet to flow into the opening and be captured. • Sizing may not be appropriate for solvent capture.
Baffle Thickness	Inches	1	0.161	0.125	Thickness is slightly smaller. • Minimal impact on baffling effects.

2.2 Method Used to Determine Upper and Lower 1/6th SEFT Mixing Speeds

As described in section 2.1, the 1/6th SEFT is linearly geometrically similar to the full scale SEFT. Geometric similarity (fixed or distorted) is obtained from dimensional analysis and is typically a prerequisite for other types of similarities of interest, such as kinematic (all velocities in two different scales have a common constant ratio) and dynamic (relates to force ratios – Reynolds number, Froude number, etc.,) similarities[8,9,10,11,12]. Geometric similarity is also typically assumed when correlating actual data from bench or pilot scale testing, though not necessarily.

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[8] Oldshue, J. Y., "*Fluid Mixing Technology*", Chemical Engineering McGraw-Hill Publication Company, 1st edition, 1983

[9] Skelland, A. H., "*Non-Newtonian Flow and Heat Transfer*", John Wiley & Sons, 1st edition, 1967

[10] Uhl, V. W. and Gray, J. B., "*Mixing Theory and Practice – Volume 1*", Academic Press, Inc., 1st edition, 1966

[11] Tatterson, G. B., "*Scaleup and Design of Industrial Mixing Proceses*", McGraw-Hill Inc., 1st edition, 1994

[12] Dickey, D. S., "*Dimensional Analysis, Similarity and Scale-Up*", Process Mixing – Chemical and Biochemical Applications: Part II, AIChE Symposium Series, No. 293, Vol. 89, pp 143 – 150

After establishing the parameters above, the agitator speed was determined using equation [1]. In most conditions, testing is initially performed on the bench or pilot scale or both to verify the scalability prior to scaling to the full scale process. Scaling from bench/pilot results to the full scale process is known as scale-up. In this study, the reverse is occurring, where the pilot scale agitator speed will be determined from the full scale operating parameters, other-wise known as scale-down. The scale-up exponent in equation [1] is used for various conditions of mixing that are described in Table 2-3. These conditions of mixing are derived from dimensional analyses or from physical testing. The scale-up parameters listed in Table 2-3 are not all inclusive. Section 3.1 contains details about the parameters discussed in Table 2-3 and calculations to support the mixing speeds and other calculated operating parameters. The P/V and tip speed parameters are typically used when dealing with the dispersions of two immiscible fluids.

$$N_2 = N_1\left(\frac{D_1}{D2}\right)^n \qquad [1]$$

Where: N_i = Shaft rotational speed (RPM)
D_i = Impeller diameter (inches)
i = scale (bench, pilot, production)
n = scale-up exponent

Table 2-3 Scale-Up Correlations

Parameter	Function	Scale-up Exponent
Circulation time	$\frac{1}{N}$	n = 0
Froude Number	$\frac{DN^2}{g}$	n = ½
Original DWPF agitator scale-up*	$DN^{1/0.6386}$	n = 0.6386
Power/volume	$\frac{N^3D^5}{D^3}$	n = $^2/_3$
Solids suspension	$ND^{3/4}$	n = ¾
Tip Speed	ND	n = 1
Minimum Impeller Speed for Liquid-Liquid dispersion in baffled vessels – physical testing	$ND^{1.1}$	n = 1.1
Weber Number	$\frac{\rho N^2D^3}{\sigma}$	n = $^3/_2$
Reynolds Number	$\frac{\rho ND^2}{\mu}$	n = 2

* Correlation is very similar to power per unit volume. Test data from a log-log chart[6] were used to determine this exponent.

2.3 MCU Solvent and Dye Use

The MCU solvent used in this task was prepared by the Waste Processing Technology (WPT) section at SRNL. Of the four components used to make the MCU solvent, the BobCalixC6 was excluded due to cost and availability. The exclusion of BobCalixC6 is expected to have minimal impact on the physical properties important in this task, specifically to the surface and interfacial surface tensions. Two MCU solvent batches were made and their batch composition is shown in Table 2-4[13]. The physical properties of the MCU solvent were compared to the baseline MCU solvent made and characterized by Oak Ridge National Laboratory (ORNL)[14]. The physical properties measured were density (MCU solvent, 0.001M HNO_3 solution), surface tension (MCU solvent, 0.001M HNO_3 solution), interfacial surface tension (between MCU and 0.001M HNO_3 solution), and viscosity. The chemical composition of the MCU solvents have been analyzed and documented by Adu-Wusu[13]. The physical and chemical measurements performed in this task will be discussed in more detail in sections 2.3.1 through 2.3.6.

Table 2-4 Composition of MCU Used In Mixing Task

Chemical	Target mass (g)	Actual Batch Makeup (g)	
		MCU Solvent Batch 1	MCU Solvent Batch 2
Modifier	480.3	480.31	480.33
tri-n-octylamine	2.01	2.01	2.01
Isopar® L	1121	1120.99	1120.94
TOTAL	1603.31	1603.22	1603.28

To assist in observing the mixing, powdered florescent dyes which are soluble in solvent based mediums, such as the MCU solvent, were selected and tested. The amount of dye added to each MCU solvent sample was approximately 0.31 wt% of the MCU solvent mass used. The dyed MCU solvents were processed through a 0.25 micron filter to remove any insoluble dye particles. The resulting filtered dyed MCU solvents' physical properties were measured and compared to the non-dyed MCU solvent used in this task. A dye was considered suitable if the dyed MCU solvent's physical properties were comparable to the un-dyed MCU solvent and the dye provided a good visual aid. The florescent dyes tested in this task were obtained from Risk Reactor[15] and are listed in Table 2-5.

Table 2-5 Risk Reactor Florescent Dyes Tested With MCU Solvent

Dye Name	Visible Color	Florescent color (380 nm frequency)
DFSB-CO Clear Blue Dye	Clear	Blue
DFSB-C7 Clear Red Dye	Clear	Red
DFSB-K43 Fluorescent Dye	Green/yellow	Green/yellow

The following sections provide a description of the instrumentation and methods used to perform the physical and chemical analyses.

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[13] Adu-Wusu, K., Crump, S. L, and White, T. L., "*Preparation of Caustic Side Solvent Extraction (CSSX) Solvent with no BOBCalixC6 for Strip Effluent Feed Tank (SEFT) Testing – Component Amounts and Analytical Results*", SRNL-WTP-2005-00118, September 19, 2005

[14] Delmau, L. H., et. al, "*Caustic-Side Solvent Extraction: Chemical and Physical Properties of the Optimized Solvent*", ORNL/TM-2002/190, October 2002

[15] Risk Reactor, 21544 Newland Street, Huntington Beach, CA 92646, www.riskreactor.com

2.3.1 Density

The density was measured using a specific density cup (Figure 2-3), which has a known volume[16] at a given temperature. The cup has a volume of 8.32 cm^3 at 25°C. The mass of the cup/cap are weighed first. Prior to performing the measurement, both the cup/cap and samples were placed in a convection oven at 25°C. A sample is then placed into the cup, almost completely filling the cup. The cap (tapered inside) is then placed on top of the cup and excess fluid is forced through a hole located in the center of the cap as the cap is pressed into the cup. The excess fluid is cleaned from the top of the cap and the cup/cap/sample weighed. The density of the sample is calculated using equation [2]. The sample is measured twice and the results averaged.

$$\rho = \frac{M_{sample\_cup\_cap} - M_{cup\_cap}}{V_{cup}} \text{ (g/cm}^3\text{)} \qquad [2]$$

De-ionized (DI) water samples were analyzed at 25°C prior to proceeding with the solvent samples.

Figure 2-2 Specific Density Cup

Attempts were made to use the Anton Paar DMA 4500 density analyzer, which is typically used at ACTL to measure fluid densities. First the sample is loaded into a syringe and then injected into the capillary section of the instrument. The MCU solvent, for some reason could not be injected into the instrument, using this method.

2.3.2 Viscosity

The viscosity was measured using a Haake RS600 rheometer, which measures both the speed and torque of the spinning bob and converts this speed and torque into shear rate and shear stress respectively. The viscosity is then obtained using equation [3]. The measuring system used was a cone/plate configuration. The cone (truncated nose) is 60 mm in diameter with a 0.5 degree angle and is made of titanium. The cone and plate are first loaded onto the rheometer. The rheometer determines the zero position, which is the point at which the cone and plate make contact, and then moves the cone away from the plate. The sample is then loaded onto the plate and the cone goes to a predetermined position (based on the cone geometry) and the excess fluid is removed. Since the fluids in this task are Newtonian, a flow curve was not necessary. Instead, the cone was quickly

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[16] Volume quantified at 25°C per ANSI Z540-1, "Calibration Laboratories and Measuring and Test Equipment - General Requirements-Replaces Mil-Std-45662".

accelerated to a shear rate of 2000 sec^{-1} and maintained at that shear rate for 30 seconds. The viscosity data, taken every second, was averaged between 5 to 25 seconds during this 30 second measurement. The sample was run twice and the average of the two runs reported. All measurements were obtained at 25°C.

$$\mu = \frac{\tau}{\dot{\gamma}} \quad [3]$$

Where: τ = calculated shear stress (Pa)
$\dot{\gamma}$ = calculated shear rate (sec^{-1})
μ = calculated viscosity (Pa·sec), 1 Pa·sec = 1000 centipoise (cP)

The rheometer was verified operational using the 60 mm, 0.5 degree cone/plate configuration and S3 Newtonian oil standard prior to making any of the measurements. DI water runs were also performed.

2.3.3 Surface Tension

The surface tension is that between a fluid and air. The surface tension was measured using two different methods.

The first method was the capillary rise in a narrow tube[17], where the liquid will rise in a capillary tube until the gravitational force is exactly equal to the wetting force. A 0.5 mm inside diameter capillary with 10 cm of measurable height was used and installed onto a stand, vertically. A sample was then placed into a cup and the cup was raised, where the surface of the fluid in the cup is level with the zero reference point on the capillary. A priming bulb was then used to pull the sample into the capillary tube to the 10 cm mark, so as to wet the internals. After wetting was complete, the sample was pushed below the zero reference point and allowed to come to equilibrium as shown in Figure 2-3. This equilibrium height was recorded and the procedure repeated at least two more times. The average height was calculated and used to determine the surface tension using equation [4], which assumes it has a well-wetted hemispherical meniscus (the meniscus weight is neglected).

$$\sigma_{Surface\_capillary} = \frac{D \cdot h \cdot \Delta\rho \cdot g}{4} \quad [4]$$

Where: D = Inside diameter of capillary tube (cm)
h = Height of liquid in capillary tube (cm)
g = Gravitational constant (981 cm/s^2 or 9.81 m/s^2)
$\Delta\rho$ = Difference in density between the fluid and air (g/mL)
$\sigma_{Surface\_capillary}$ = Surface tension (dynes/cm)

Prior to making the surface tension measurements, the samples were maintained at 25°C in a convection oven. In between samples, the capillary was cleaned using very dilute soapy water, rinsed with DI water and dried using air from compressed air bottles.

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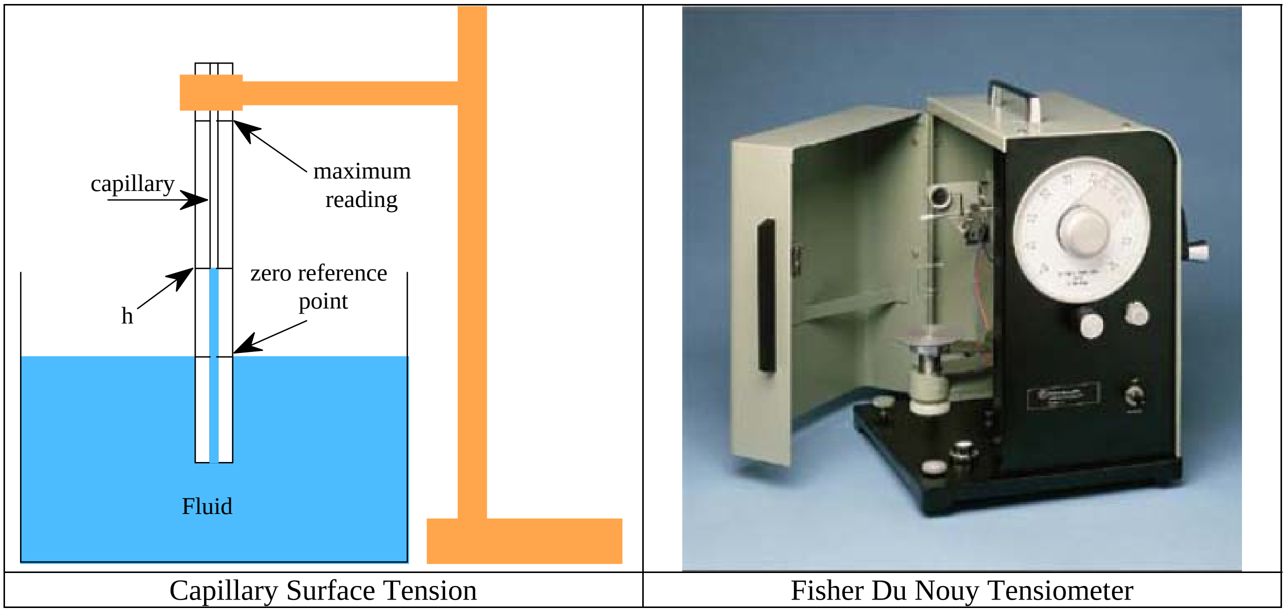
[17] Shugar, G. J. and Ballinger, J. T., "Chemical Technicians' Ready Reference Handbook", 1st Edition, McGraw-Hill, Inc., 1996



Capillary Surface Tension | Fisher Du Nouy Tensiometer

Figure 2-3 Surface and Interfacial Surface Tension Instrumentation

The second surface tension method used the Fisher Surface Tensiomat, Model 21 (Figure 2-3), which is a Du Nouy tensiometer. Much of the experimental procedure used was adapted from ASTM D1331[18] and ASTM D971[19]. A 100 ml glass beaker (diameter of 47.2 mm) was utilized. The glass beaker was rinsed with ethanol and pat dried with a handi-wipe, rinsed with tap water, and rinsed with DI water. This was followed by another ethanol rinse, removal of the excess ethanol using a handi-wipe, and drying of the beaker with compressed bottle air. The platinum ring (diameter of ring = 5.93 cm and diameter of ring wire = 0.05 cm) was cleaned in the same manner as that of the beaker, but after the 2nd ethanol rinse the ring was dried using compressed bottle air and then flamed (using a butane lighter). The ring was then inspected and cleaned of any soot present. The ring was then attached to the Tensiomat and the sample was placed into the beaker. The beaker was then raised until the ring was adequately submerged. The beaker was then slowly lowered and the maximum uncorrected surface tension (dyne/cm) required to pull the ring out of the solution was recorded. The measurement step was repeated a total of four times and the average uncorrected surface tension calculated. This data was then corrected[18,19] for the actual surface tension using equation [5]. F is the correction factor for the wire and is calculated using equation [6].

$$\sigma_{surface\_ring} = F \cdot \sigma_{uncorrected\_surface\_ring} \qquad [5]$$

$$F = 0.7250 + \sqrt{\frac{0.01452 \cdot \sigma_{uncorrected\_surface\_ring}}{C^2 \cdot \left(\rho_H - \rho_L\right)} + 0.04534 - 1.679\frac{r}{R}} \qquad [6]$$

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[18] ASTM D1331-89, "*Standard Test Method for Surface and Interfacial Tension of Solutions of Surface Active Agents*", 2001

[19] ASTM D971-99a, "*Standard Test Method for Interfacial Tension of Oil Against Water by the Ring Method*", 2004

Where: $\sigma_{surface\_ring}$ = Surface tension or interfacial surface tension (dynes/cm)
$\sigma_{uncorrected\_surface\_ring}$ = Uncorrected surface tension or uncorrected interfacial surface tension (dynes/cm)
F = Correction factor (unitless)
C = Circumference of the ring (cm)
ρ_L = Density of lighter fluid (g/mL)
ρ_H = Density of denser fluid (g/mL)
r = Radius of wire of ring (cm)
R = Radius of ring (cm)

Prior to analyzing the samples, the unit was functionally checked using DI water at 25°C.

2.3.4 Interfacial Surface Tension

The interfacial surface tension between two fluids, MCU solvent and 0.001M HNO_3 solution, were measured using the Fisher Tensiomat. The same procedure described in section 2.3.3 was used in preparing the ring and beaker. For this measurement, the denser fluid was added to the beaker and the beaker was raised to completely immerse the ring below the denser fluid surface. The less dense fluid was then carefully added to the surface of the denser fluid, to prevent any mixing. The beaker was then lowered and the maximum uncorrected interfacial surface tension was recorded as the ring moved through the interface between the two fluids. Duplicate measurements were performed and the ring and beaker were cleaned between each measurement. The average uncorrected interfacial surface tension was calculated and used to calculate the interfacial surface tension using equations [5] and [6].

2.3.5 Solvent Concentration Using Cs-7SB (Modifier) Analysis

The Cs-7SB (modifier) in the MCU solvent was analyzed using high performance liquid chromatography (HPLC) by the Analytical Developmental Section (ADS) at SRNL. The method of analysis for the modifier was determined on a normal-phase cyano column using 96% hexane and 4% isopropanol as the mobile phase. The conditions for the Modifier analysis are summarized in Table 2-6.

The quantity of Cs-7SB was reported in mg/L. The solvent concentration was then determined using equation [7], where the masses of the solvent and Cs-7SB were obtained from Table 2-4. The parts per million (ppm) of the MCU solvent in the 0.001M HNO_3 solution was calculated by dividing the solvent concentration by the density of the 0.001M HNO_3 solution.

$$C_{solvent,Cs-7SB} = C_{Cs-7SB} \cdot \frac{M_{solvent}}{M_{Cs-7SB}} \qquad [7]$$

Where: $M_{solvent}$ = Mass of MCU solvent made (grams)
$M_{Cs\text{-}7SB}$ = Mass of Cs-7SB used to make MCU solvent (grams)
$C_{Cs\text{-}7SB}$ = Concentration of CS-7SB in HNO_3 solution using HPLC analysis (mg/L)
$C_{solvent,\ Cs\text{-}7SB}$ = Concentration of MCU solvent in HNO_3solution (mg/L)

Table 2-6 Isocratic for Cs-7SB (Modifier)

Method	Conditions
Solvent system	Hexane/Isopropanol
t_o to t_1 = 8.0 min	96%/4%
Normal Phase Cyano Column	Agilent Technologies Zorbax CN
	4.6 x 150 mm, 5 mm pore size
Oven temperature	Ambient
Flow-rate	1 mL
Ultraviolet Wave Length	230 nm
Injection Volume	5 mL
Retention time for Extractant	6.3 min
Linear calibration curve	
Modifier	12 mg/L to 240 mg/L, r^2 = 0.999
R.S.D. (%) (n=7)	0.73

2.3.6 Solvent Concentration Using Isopar® L Analysis

The Isopar®L in the MCU solvent was analyzed by ADS using semi-volatile organic analysis (SVOA).

The quantity of Isopar®L was reported by ADS in ppm. The solvent concentration for each sample location was determined using equation [7], where the masses of the solvent and Isopar®L were obtained from Table 2-4.

$$C_{solvent,IsoparL} = C_{IsoparL} \cdot \frac{M_{solvent}}{M_{IsoparL}} \qquad [7]$$

Where: $M_{solvent}$ = Mass of MCU solvent made (grams)
$M_{IsoparL}$ = Mass of Isopar®L used to make MCU solvent (grams)
$C_{IsoparL}$ = Concentration of Isopar®L in HNO_3 solution by SVOA (ppm)
$C_{solvent,IsoparL}$ = Concentration of MCU solvent in HNO_3 solution (ppm)

2.4 Baseline Water Run Using 1/6th SRAT

Baseline water runs were performed at the two different agitator speeds using the 1/6th SRAT mixing configuration to determine if any operational or mixing problems needed to be corrected for the 1/6th SEFT. This was a purely visual test and provided guidance for the 1/6th SEFT test.

2.5 Solvent Runs Using 1/6th SEFT

Two 1/6th scale mixing tests were performed, targeting 228 ppm of MCU solvent in a 0.001M HNO_3 solution. The mixing tank was initially filled with DI water and a 50 wt% HNO_3 solution was added and mixed to make the 0.001M HNO_3 solution. The molarity of the HNO_3 solution was verified by measuring the pH and verifying the pH was between 2.9 to 3.1. After the agitator was started, approximately 228 ppm of MCU solvent was added to the surface of the agitated solution. During both mixing tests, samples were pulled starting 30 minutes after the MCU solvent was added to determine the tank homogeneity. The same sample locations were used for both mixing tests. Samples were pulled at four axial positions for a fixed radial position while the tank was being agitated at the targeted mixing speed. The samples were pulled at 5, 10, 15 and 18 inches from the bottom of the tank using a different glass composite liquid waste sampler (Coliwasa) for each sample location. The contents in the Coliwasa were transferred to a glass bottle (cap with Teflon seals) and the mass transferred recorded. The sampling chamber of the Coliwasa was then washed two to three times using a 0.001M HNO_3 solution to remove any MCU solvent in the sampling chamber. The walls of the sample chamber were inspected using an ultraviolet flashlight to verify the washing was effective in removing the MCU solvent. The wash solutions were added to the glass bottle containing the sample. When washing was complete, the mass of the glass sample bottle was weighed. During pump down, the initial contents were collected from the scaled transfer pump. This sample was obtained to determine how fast the MCU solvent was building up in the transfer pump suction priming line. The pulled samples, as well as a sample of the MCU solvent were analyzed for modifier and Isopar®L. After the samples were pulled, the pump down test started and continued until mixing was an issue or the minimum tank level had been reached. During these tests, an ultraviolet light was initially used, which provided a better means of visually recording the mixing. As the contents were pumped down, spot lighting was used.

The analytical results for the modifier and Isopar®L for the tank samples were those of diluted samples (containing 0.001M HNO_3 wash solution to clean the MCU solvent from the Coliwasa). Once the results were calculated as ppm as described in sections 2.3.5 and 2.3.6, they were corrected to determine the actual ppm in the mixing tank using equation [8]. This calculation slightly overestimates the solvent concentration, since the mass of the original sample only contained the material that was initially transferred to the sample bottle. The very slight quantities (and not quantifiable) of material that were flushed with the wash water would have made the mass of the original sample slightly larger.

$$C_{solvent,corrected,j} = C_{solvent,j} \cdot \frac{M_{sample+wash}}{M_{Sample}} \qquad [8]$$

Where: $M_{sample+wash}$ = Mass of sample and wash water (grams)
M_{Sample} = Mass of sample (grams)
j = Modifer (Cs-7SB) or Isopar®L
$C_{solvent,j}$ = Concentration of MCU solvent calculated in section 2.3.5 or 2.3.6 (ppm)
$C_{solvent,corrected,j}$ = Concentration of MCU solvent in mixing tank (ppm)

The rate at which the MCU solvent was concentrating in the transfer pump suction priming line was determined using equations [9] and [10]. Note that these pump samples contained more sample that what is contained in the pump suction priming line. These samples were pulled using a peristaltic pump with the intended purpose of over-sampling. Due to this fact, equation [10] assumes that during the sampling, the excess sample has the same average concentration of the tank contents. Sampling biasing was not investigated in this task. Tank agitator speed (which results in local cross face velocity), MCU droplet size, and pump suction velocity (rate of sampling) all impact sample biasing. These same quantities also impact the Coliwasas used for tank sampling as described above.

$$\overline{C}_j = \sum_{n=1}^{4} C_{solvent,corrected,j,n} \qquad [9]$$

where: $\overline{C}_j$ = Average MCU concentration in mixing tank (ppm)
n = Sample position in tank (5”, 10”, 15” and 18” positions)

$$\dot{C}_{pump} = \frac{\left(C_{Pump} - \bar{C}_j\right) \cdot M_{sample} + \bar{C}_j \cdot \rho_{0.001MHNO_3} \cdot V_{pump}}{\rho_{0.001MHNO_3} \cdot V_{pump} \cdot t} \qquad [10]$$

where: $\dot{C}_{pump}$ = Buildup rate of MCU solvent in pump suction priming line (ppm/min)
C_{pump} = MCU solvent concentration (ppm)
V_{pump} = Volume of fluid in pump suction priming line at full tank conditions (mL)
$\rho_{0.001MHNO3}$ = Density of 0.001M HNO_3 solution (g/mL)
t = Time sample was pulled after the test started (minutes)

3.0 CALCULATIONS AND TEST RESULTS

3.1 Calculation: Agitator Speed Determinations for 1/6th SEFT Mixing Tests

The solids suspension parameter (based on Zwietering correlation)in Table 2-3 is not applicable to this application since the solids (in this case the optimized solvent) are lighter than the continuous phase and are not required to be suspended from the bottom of the tank. The other parameters stated in Table 2-3 will be discussed in more detail below.

3.1.1 Circulation Time

The flow produced by an impeller is given by equation [11]. The flow number for geometrically similar impellers/processes is the same for a given Reynolds number. For turbulent flow (Re > 10,000), the flow number is essentially constant. The recirculation time is the tank volume divided by the flow produced by the impeller, equation [12].

$$Q = N_Q N D^3 \qquad [11]$$

$$\theta = \frac{V}{N_Q N D^3} \qquad [12]$$

where: Q = flow (m^3/sec)
N_Q = flow number (unitless)
N = rotational speed (revolution/second)
D = Impeller diameter (m)
θ = circulation time (sec)
V = volume of tank contents (m^3)

For geometrically similar mixing vessels, equation [12] reduces to the function shown in Table 2-3. For calculation purposes, the flow number is assumed to be 3.72 (from footnote 10, pg. 206, equation 59c) for a 4 flat blade impeller with baffles, turbulent flow (Re > 10,000), 4 baffles that are 0.1 of the tank diameter, flat bottom tank, and the turbine located with equal amount of fluid above and below the impeller. In reality, the actual flow number would be smaller than this due to the physical location[8] of the impellers and flow impedances of the SEFT internals. The calculation to determine the circulation time (at 9,600 gallons = 36.34 m^3) is provided for the full scale SEFT and the calculation of the agitator speed for the 1/6th SEFT. When the recirculation times are the same, the agitator speeds are also the same on the two scales.

$$\theta_1 = \frac{36.34 m^3}{3.72 \cdot 68 \frac{rev}{\min} \cdot \frac{\min}{60 \sec} \cdot \left(0.9144 m\right)^3} = 11.3 \sec \qquad [13]$$

$$N_2 = N_1 = 68 \ \ RPM \qquad [14]$$

The other parameters in Table 2-3 have been calculated for the recirculation time and the results are summarized in Table 3-1.

3.1.2 Froude Number

The Froude number is the function shown in Table 2-3. The Froude number, equation [15], represents the ratio of inertial to gravitational forces. The application of the Froude number is typically used for unbaffled agitated tanks. There have been studies which investigate vortex formation in baffled tanks[20], which incorporates the Froude number. The Froude number for the full scale SEFT and the agitator speed for the 1/6th SEFT are calculated below.

$$N_{Fr} = \frac{N^2 D}{g} = \frac{\left(68 \frac{rev}{\min} \cdot \frac{\min}{60 \sec}\right)^2 \cdot 0.9144 m}{9.81 \frac{m}{\sec^2}} = 0.120 \qquad [15]$$

$$N_2 = N_1 \left(\frac{D_1}{D_2}\right)^{1/2} = 68 \ RPM \left(6.21\right)^{1/2} = 169 \ RPM \qquad [16]$$

The other parameters in Table 2-3 have been calculated for the Froude number and the results are summarized in Table 3-1.

3.1.3 Power/Volume

The power produced by an impeller is given by equation [17], if the flow is turbulent. The power number for geometrically similar impellers/processes in turbulent flow (Re > 10,000) conditions is constant. The power per unit volume is the power divided by the tank volume and is shown in equation [18]. The power per unit volume is valid when the objective is to achieve equal interfacial area per unit volume in a liquid-liquid mixture on two different scales. Power per unit volume is also the basis for interfacial mass transfer/droplet size conditions and has been commonly used for reproducing dispersion qualities[8,9,11,21,22]. Etchels[22] and Skelland[23] state that the use of this criterion is likely to be conservative if only dispersion homogeneity is desired. The scale-up exponent used by Ekato[6] to size the agitators used in DWPF was determined to be 0.6386, which is very close to the power/volume exponent and would have yielded a slightly lower agitator speed. Hence, no calculation was performed for the Ekato correlation.

$$P = \rho N_P N^3 D^5 \qquad [17]$$

$$\frac{P}{V} = \frac{\rho N_P N^3 D^5}{V} \qquad [18]$$

where: P = Power (watts)
ρ = Density of the fluid (kg/m3)
N_P = Power number (unitless)

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[20] Clark, M. W. and Vermeulen, T., “Incipient Vortex Formation in Baffled Agitated Vessels”, AIChE, pp 420-422, May 1964

[21] Etchells III, A. W, Hemrajani, R. R., Koestler, D. J., Paul, E. L., “*The Many Faces of Mixing*”, Chemical Engineering, pp. 92 – 94, March 1992

[22] Uhl, V. W. and Gray, J. B., “*Mixing Theory and Practice – Volume 1I*”, Academic Press, Inc., 1st edition, 1966

[23] Skelland, A. H. P. and Seksaria, R., “Minimum Impeller Speeds for Liquid-Liquid Dispersion in Baffled Vessels”, Industrial Engineering Chemical Process Design Development, Vol. 17, No.1, pg. 56-61, 1978

For geometrically similar mixing vessels, equation [17] reduces to the function shown in Table 2-3. For calculation purposes, the power number is assumed to be 5.75 (from footnote 8, pg. 66, Table 3-2) for the bottom flat blade impeller. The contribution of the hydrofoil is at most 10% of that of the bottom blade under full tank conditions and will be ignored in these calculations. As with the flow number, the power number is an over-estimate of the actual power number. Corrections, such as free flow, impeller location, and liquid above the impeller, all impact the power number by reducing its value. A calculation to determine the power per unit volume (at 9,600 gallons) is provided for the full scale SEFT and the calculation of the agitator speed for the 1/6th SEFT.

$$\frac{P}{V}=\frac{\rho N_P N^3 D^5}{V}=\frac{1000\frac{kg}{m^3}\cdot 5.75\cdot\left(68\frac{rev}{\min}\cdot\frac{\min}{60\sec}\right)^3\cdot\left(0.9144m\right)^5}{36.34m^3}=\frac{147\ W}{m^3} \quad [19]$$

$$N_2=N_1\left(\frac{D_1}{D_2}\right)^{2/3}=68\ RPM\left(6.21\right)^{2/3}=230\ RPM \quad [20]$$

The other parameters in Table 2-3 have been calculated for the power per unit volume and the results are summarized in Table 3-1.

3.1.4 Tip Speed

The tip speed is the product of the impeller diameter and shaft speed (function shown in Table 2-3) multiplied by π. Tip speed provides equal liquid velocity at the discharge of the impeller and has been commonly recognized as equal agitation intensity for liquid blending (from footnote 21, pg. 144 and reference 22, pg. 30-31). The tip speed for the full scale SEFT and the agitator speed for the 1/6th SEFT are calculated below.

$$S=68\frac{rev}{\min}\cdot\frac{\min}{60\sec}\cdot\pi\cdot 0.9144m=3.26\frac{m}{\sec} \quad [21]$$

$$N_2=N_1\left(\frac{D_1}{D_2}\right)=68\ RPM\left(6.21\right)=422\ RPM \quad [22]$$

The other parameters in Table 2-3 have been calculated for the tip speed and the results are summarized in Table 3-1.

3.1.5 Minimum Impeller Speed for Liquid-Liquid Dispersion in Baffled Vessels

A correlation to determine the minimum agitator speed required for liquid-liquid dispersion in baffled vessels is provided by Skelland[23] and is shown as equation [23]. Skelland's definition of minimum mixing speed was that of a well mixed or completely dispersed state where only a small relatively non-stationary, liquid pockets, remained unmixed in the bulk dispersion. In this study, two immiscible liquids in equal volumetric proportions were used.

$$\frac{N\cdot D^{1/2}}{g^{1/2}}=C_1\left(\frac{T}{D}\right)^{\alpha_1}\left(\frac{\mu_c}{\mu_d}\right)^{1/9}\left(\frac{\Delta\rho}{\rho_c}\right)^{1/4}\left(\frac{\sigma}{D^2\rho_c g}\right)^{0.3} \quad [23]$$

where: T = tank diameter (m)
μ_c = continuous phase viscosity (Pa·sec)
μ_d = dispersed phase viscosity (Pa·sec)
$\Delta\rho$ = positive density difference between continuous and dispersed phase (kg/m^3)
ρ_c = density of continuous phase (kg/m^3)
σ = interfacial surface tension (N/m)

For geometrically similar mixing vessels and constant physical properties, equation [23] reduces to the function shown in Table 2-3. The rotational speed for the 1/6th scale SEFT is calculated below.

$$N_2 = N_1\left(\frac{D_1}{D_2}\right)^{1.1} = 68\ \ RPM\left(6.21\right)^{1.1} = 507\ \ RPM \qquad [24]$$

The other parameters in Table 2-3 have been calculated for the condition of minimum impeller speed for liquid-liquid dispersion in baffled vessels and the results are summarized in Table 3-1.

3.1.6 Weber Number

The Weber number for mixing applications is shown in Table 2-3. The Weber number is the inertial force divided by the surface tension force and is used in bubble/drop formation. The application of using the Weber number by itself for scaling in mixing is limited and is expected to yield a non-conservative mixing condition when scaling-up for dispersion. The Weber number for the full scale SEFT and the agitator speed for the 1/6th SEFT are calculated below. The interfacial surface tension between the optimized solvent and 0.001M nitric acid solution is approximately 15 dynes/cm.

$$N_{We} = \frac{\rho N^2 D^3}{\sigma} = \frac{1000\frac{kg}{m^3}\cdot\left(68\frac{rev}{\min}\cdot\frac{\min}{60\sec}\right)^2\cdot\left(0.9144m\right)^3}{15\frac{dyne}{cm}\cdot\frac{10^{-5}N}{dyne}\cdot\frac{100cm}{m}} = 65,469 \qquad [25]$$

$$N_2 = N_1\left(\frac{D_1}{D_2}\right)^{3/2} = 68\ \ RPM\left(6.21\right)^{3/2} = 1052\ \ RPM \qquad [26]$$

The other parameters in Table 2-3 have been calculated for the Weber number condition and the results are summarized in Table 3-1.

3.1.7 Reynolds Number

The Reynolds number is the function shown in Table 2-3. The Reynolds number is the inertial force divided by the viscous force. The application of the Reynolds number has little practical use in scale-up and has been shown to be very non-conservative. The Reynolds number for the full scale SEFT and the agitator speed for the 1/6th SEFT are calculated below. The Reynolds number does provide the condition of flow (laminar, transitional, or turbulent).

$$N_{\mathrm{Re}} = \frac{\rho N D^2}{\mu} = \frac{1000\frac{kg}{m^3}\cdot 68\frac{rev}{\min}\cdot\frac{\min}{60\sec}\cdot\left(0.9144m\right)^2}{0.001\ \ Pa\cdot\sec} = 947,611 \qquad [27]$$

$$N_2 = N_1 \left(\frac{D_1}{D_2} \right)^2 = 68 \ RPM \left(6.21\right)^2 = 2622 \ RPM \qquad [28]$$

The other parameters in Table 2-3 have been calculated for the Reynolds number and the results are summarized in Table 3-1.

3.1.8 Summary of Mixing Speeds

The results, using the different scaling parameters, are shown in Table 3-1. Maintaining the same circulation time results in the same agitator speed, but the power requirements are not adequate for dispersion. The Weber and Reynolds number correlations yield unreasonable power requirements for the 1/6th scaled system, where the Reynolds number power requirements are larger than that of the full scale tank. The Froude number was provided as information only, since vortex formation is not expected and, if present, the basic Froude number may not be applicable to this application.

The two sizing parameters, which are typically used for liquid-liquid dispersions, are the power per unit volume and tip speed parameters. The power requirements from these sizing parameters are reasonable at the 1/6th scale. Additionally, the actual agitators were sized by Ekato, where the resulting scale-up exponent is very close to that of the power per unit volume. Use of the Ekato scale-up exponent would have yielded a rotational speed slightly less than that of the power per unit volume. The circulation times for the 1/6th scale SEFT are between 4 to 6 times faster than that of the full scale SEFT using the P/V and tip speed scaling parameters. The correlation used to determine the minimum agitator speed for liquid to liquid dispersion of a baffled tank is very similar to that of the tip speed correlation, but yielding a slightly higher rotational speed and about twice the power consumption. The bolded items in Table 3-1 are the results for the P/V and tip speed parameters.

Table 3-1 Scale-down Results for the 1/6th SEFT

Parameter	Full Scale SEFT	1/6th SEFT						
		Circ. Time	Froude	**P/V**	**Tip Speed**	Minimum L to L*	Weber	Reynolds
Rotational Speed (RPM)	68	68	169	**230**	**422**	507	1052	2622
Circulation Time (Sec)	11.3	11.3	4.5	**3.3**	**1.8**	1.5	0.7	0.3
Froude Number	0.12	0.02	0.12	**0.22**	**0.74**	1.07	4.62	28.67
Power (W)	5351	0.58	8.97	**22**	**139**	240	2147	33226
Power (HP)	7.2	0.001	0.012	**0.030**	**0.19**	0.32	2.9	44.5
Power Per Unit Volume (W/m^3)	147	3.8	59	**147**	**914**	1581	14149	218943
Tip Speed (m/s)	3.26	0.52	1.31	**1.77**	**3.26**	3.91	8.11	20.22
Weber Number (unitless)	65469	273	1698	**3121**	**10543**	15191	65469	406532
Reynolds Number (unitless)	947611	24576	61240	**83026**	**152605**	183179	380276	947611

* Minimum Impeller Speed for Liquid-Liquid Dispersion in Baffled Vessels

Note that the reverse in agitator speed determinations would occur when sizing from a pilot scale to a full scale process. The Reynolds number would yield the lowest agitator speed and the circulation time would yield the greatest speed.

3.2 Results: MCU Solvent and Dyed MCU Solvent

The MCU solvent made by WPT and dyed MCU solvents were physically analyzed by the Stabilization Science Research Group and chemically analyzed by ADS[13]. The chemical analyses[13] of the MCU solvents were within ±10% for Cs-7SB and Isopar®L and ±13% for trioctyl-amine of the batched chemicals and are considered chemically acceptable MCU solvents.

The K43 and CO florescent dyes were soluble in the MCU solvent. The C7 (red dye) was not soluble in the MCU solvent. The characterized physical properties of the MCU solvent and dyed MCU solvents are shown in Table 3-2. Table 3-2 also contains the physical data of the Oak Ridge CSSX optimized[14] solvent in the columns labeled Oak Ridge. The data shows that the MCU solvents made by WTP are physically very similar to that of the Oak Ridge CSSX optimized solvent. The largest difference was the surface tension of the 0.001M HNO_3 solution (6.32×10^{-5} grams of HNO_3 per gram of H_2O), where Oak Ridge measured 36.4 dynes/cm and SRNL measured 69 dynes/cm using two different methods. The physical properties (including interfacial surface tension) of the SRNL solvents (including dyed solvents) are within 10% of the Oak Ridge solvent and are considered acceptable for this mixing task. The slightly higher interfacial surface tension of the SRNL solvents will lead to slightly larger droplets in a mechanically agitated mixing vessel. The bolded MCU solvent in Table 3-2 was selected for use in the mixing test since the dye also provided a color that was visible with the naked eye.

Table 3-2 Measured Physical Properties

Sample I.D.	Density (g/mL)		Surface Tension (dynes/cm)			Interfacial Surface Tension (dynes/cm)		Viscosity (cP)	
			Capillary	Du Nouy Method		Solvent and 0.001M HNO_3			
	SRNL	Oak Ridge	SRNL	SRNL	Oak Ridge	SRNL	Oak Ridge	SRNL	Oak Ridge
DI water	0.9982	n/m	69.9	70.0	71 - 73	n/m	n/m	0.886	0.89011*
0.001 M HNO3	0.9982	0.9974	69.2	69.4	36.4	n/m	n/m	n/m	n/m
MCU Solvent Batch 1	0.8498	0.8516	23.8	23.8	24.1	16.0	14.9	3.20	3.51
MCU Solvent Batch 2	0.8495	0.8516	23.8	23.6	24.1	15.8	14.9	3.21	3.51
MCU Solvent Batch 1 + K43	**0.8495**	**n/m**	**24.0**	**23.1**	**n/m**	**16.1**	**n/m**	**3.25**	**n/m**
MCU Solvent Batch 1 + CO	0.8499	n/m	24.1	23.4	n/m	15.8	n/m	3.23	n/m

n/m = not measured, * Viscosity of water at 25ºC from NIST

3.3 Water Mixing Tests on 1/6th SRAT Results

Water mixing tests using the 1/6th SRAT were performed at 230 RPM and 422 RPM and snap shots of various levels of mixing are shown in Figure 3-1. At 230 RPM, air entrainment was obvious only at the lowest tank level and the air that was entrained yielded large bubbles. At 422 RPM, air entrainment was obvious for much of the mixing, other than when the liquid level cleared the top of the coil assembly. At the lowest mixing level at 422 RPM, the generated bubbles were much smaller than the bubbles generated at 230 RPM. During this phase of testing, there was indication of aerosol generation.

Based on these water runs and the fact that the SEFT has more baffling than the SRAT, the issue of air entrainment at 422 RPM was deemed not an issue and the mixing speeds of 230 RPM and 422 RPM were pursued for the 1/6th SEFT.

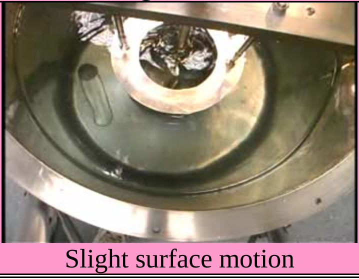
Condition	230 RPM	422 RPM
1/6th scale 8.35 gallon Full Scale 2000 gallons	 Air entrainment – large bubbles	 Massive air entrainment
1/6th scale 14.70 gallon Full Scale 3520 gallons	 Defined vortex	 Air entrainment
1/6th scale 21.05 gallon Full Scale 5040 gallons	 Good mixing	 Slight air entrainment
1/6th scale 27.40 gallon Full Scale 6560 gallons	 Slight vortex	 Vortex, sight air entrainment
1/6th scale 40.10 gallon Full Scale 9600 gallons	 Slight surface motion	 Defined vortex

Figure 3-1 Water Mixing Results of 1/6th SRAT

3.4 MCU Solvent Mixing 1/6th SRAT Results

Two tests were performed using dyed MCU solvent. The first one occurred at 230 RPM and the second one at 422 RPM.

3.4.1 230 RPM MCU Solvent Mixing Testing

In the 230 RPM test, 151,381 grams of DI water were added to the tank and then 13.718 grams of 69.7 wt% HNO_3 solution were added to the agitated tank, resulting in a solution of 0.001M HNO_3 having a measured pH of 2.99. The dyed MCU solvent, 34.582 grams, was then added to the top of the tank and allowed to mix for 30 minutes prior to sampling. Mixing occurred at room temperature, which was approximately 22°C. During the 30 minutes of agitation, large droplets of MCU solvent were observed to be pulled (entrained) from the top surface (via vortices/eddies) down through the top impeller, and the droplets dispersed into smaller droplets by the bottom flat blade impeller and/or by making physical contact with the internals. It was very evident that MCU solvent was being dispersed because the color of the solution become darker and darker, indicating, as the mixing progressed, more and more solvent was being dispersed. This can be observed in Figure 3-2.

Figure 3-2 230 RPM Mixing, First 30 Minutes

The samples that were pulled after 30 minutes of mixing were analyzed for the modifier (Cs-7SB) and Isopar®L. Of the two methods used to analyze for MCU solvent concentration, the modifier results are more consistent with the visual observations and are those used to determine the state of mixing. These results were then used to calculate the ppm of solvent in the mixing tank and the results are shown in

Table 3-3. The quantity of solvent added to the system was 34.58 grams and the amount accounted for in the average tank sample using the modifier results is 26.58 grams. The difference is most likely due to the holdup of MCU solvent in the pump and sparger ring, MCU solvent adhering to the exposed surface of the tank internals, MCU solvent floating on the surface, and sampling location and technique. The MCU solvent floating on top of the surface stayed in the quaternary where the baffles were 106.45° apart, where portions of the surface were continuously entrained (via droplets), and droplets seemed to agglomerate in the area of the floating solvent. The 30 minutes of mixing may not have been adequate in obtaining a steady state condition.

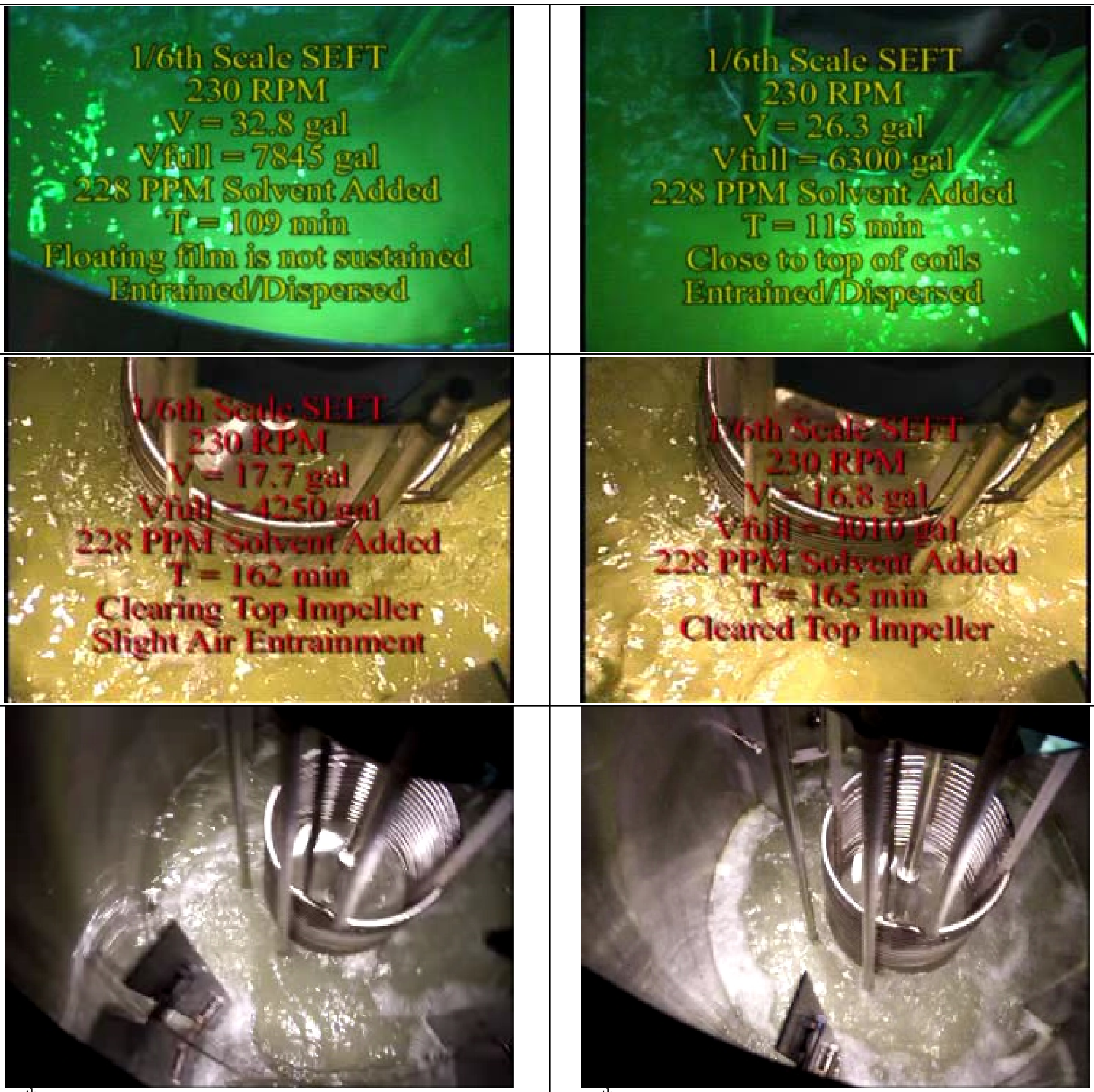
The pump sample was pulled 48 minutes into the mixing test. Table 3-3 contains the concentration and the buildup rate of MCU solvent in the pump. The concentration in this sample was greater than the concentration for any given tanks sample, using the modifier results. The rate at which the MCU solvent accumulated in the pump suction line was 12.7 ppm/min.

Table 3-3 MCU Solvent Concentration in Mixing Tank and Pump at 230RPM

Description	Units	Mixing System	Using the following results	
			Modifier	Isopar®L
Mass of solvent added to tank	Grams	34.58	n/a	n/a
ppm based on mass of solvent added	ppm	228	n/a	n/a
18 inches from bottom of tank	ppm	n/a	193	68
15 inches from bottom of tank	ppm	n/a	178	53
10 inches from bottom of tank	ppm	n/a	157	125
5 inches from bottom of tank	ppm	n/a	174	96
Average of 4 measurements	ppm	n/a	176	86
Mass of Solvent in solution	Grams	n/a	26.58	12.95
Pump Sample	ppm	n/a	207	43
Buildup rate of MCU solvent in pump	ppm/min	n/a	12.7	n/c

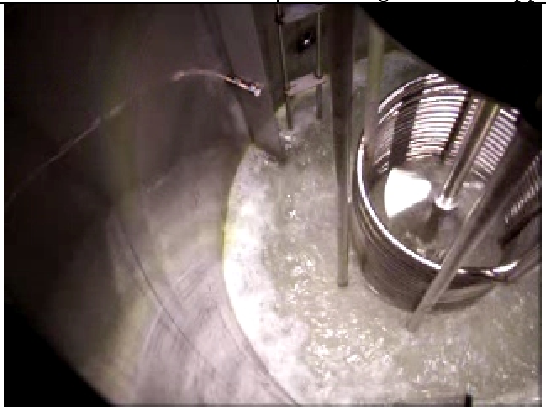
n/a = not applicable, n/c = not calculated

During the pump down test, the tank contents were removed via the installed scaled pump connection using a peristaltic pump. Key pump down conditions are shown in Figure 3-3. The first significant point was the complete entrainment of the MCU solvent, which occurred at a tank volume of 32.8 gallons (approximately 17.9 inches of liquid level). The next point was when the surface approached the top of the cooling coil assembly, which showed no mixing problems were encountered. As the surface went through the upper impeller, some bubbles were generated, but they were not stable and did not plate out on to exposed surfaces. At 10.1 gallons (5.5 inches of liquid level), the initial signs of air entrainment were observed, but the bubbles were not stable. When the contents were pumped down to 9.11 gallons (five inches of liquid level), air was being activity entrained, resulting in the generation of bubbles and is shown in Figure 3-3. As the contents of the tank were pumped down past this point, larger quantities of air were entrained. The entrained air seemed to strip the MCU solvent out of solution and plate out the MCU solvent onto the exposed surfaces at this level. The surface tension of the MCU solvent, which is much less than that of the 0.001M HNO_3 solution, could be the reason why solvent is stripped from the solution via aeration. The depletion of the solvent in the solution can be seen in the last three pictures shown in Figure 3-3, as the contents are pumped down and entraining air, there is clear evidence of a green film/foam (MCU solvent) gathering on the sides of the tank. These bubbles were not stable, in the sense that there was never a stable foam layer.



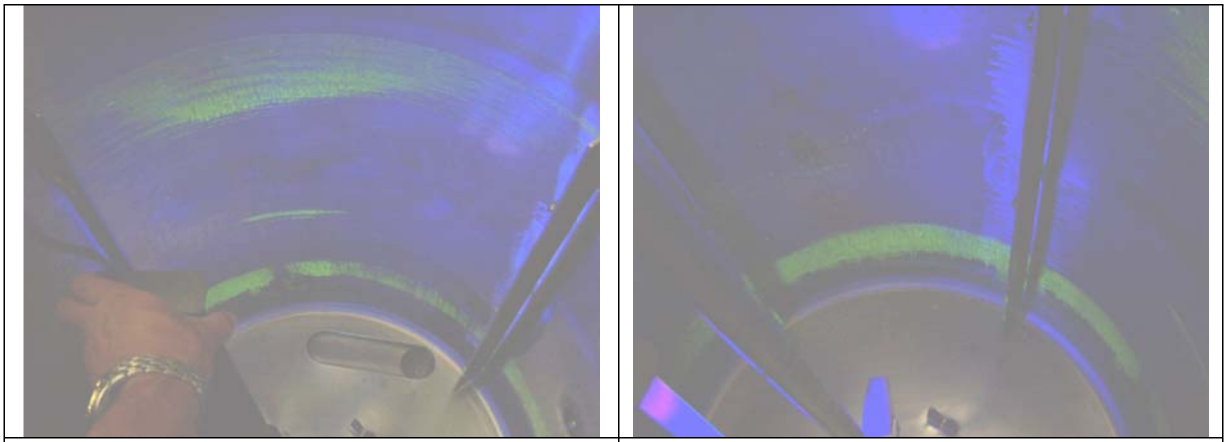
1/6th SEFT, 230 RPM, V = 9.11 gallons, V_{full} = 2180 gallons, 228 ppm MCU solvent added

1/6th SEFT, 230 RPM, V = 8.15 gallons, V_{full} = 1950 gallons, 228 ppm MCU solvent added



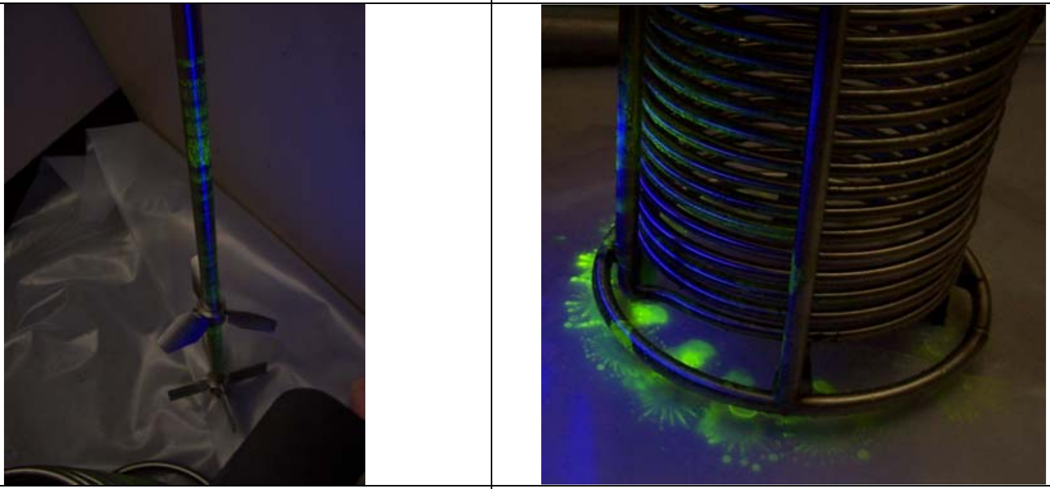
1/6th SEFT, 230 RPM, V = 7.20 gallons, V_{full} = 1720 gallons, 228 ppm MCU solvent added

Figure 3-3 Key Pump Down Results During the 230 RPM Test



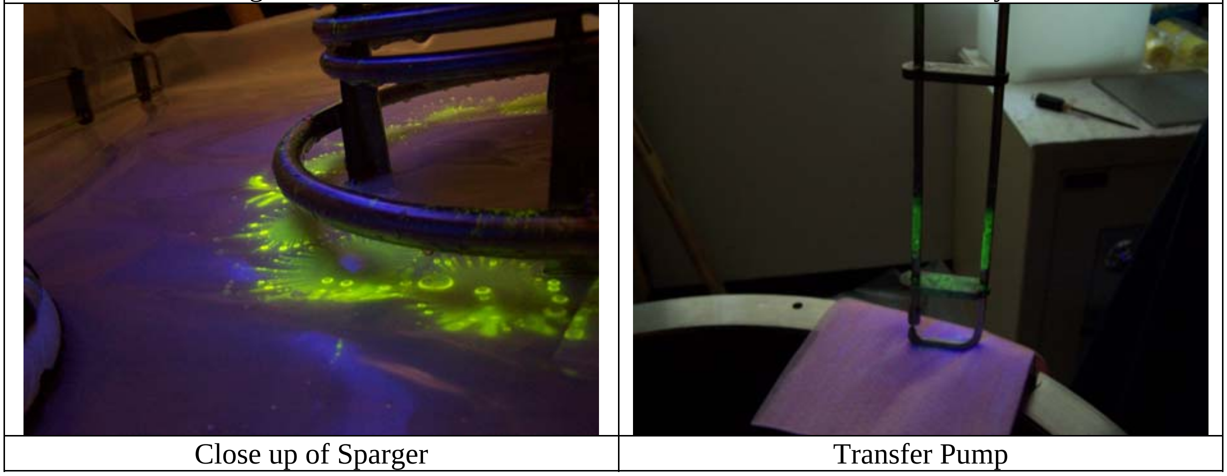
Tank Wall Between 106.45° Baffles

Tank Wall - bottom of tank



Agitator Shaft

Coil Assembly



Close up of Sparger

Transfer Pump

Figure 3-4 Internal Post Inspection of Wetted Parts and Tank Surface at 230 RPM

After the pump down test was completed, the contents of the tank were emptied and pictures of the internals taken. The pictures in Figure 3-4 clearly show the MCU solvent adhering to the surfaces. The tank wall pictures show that the MCU solvent collected between the 106.45° baffle, where the top layer is that at full tank conditions and the lower layer is from air entrainment during mixing at the lower tank levels. The picture also shows that the sparger system will accumulate MCU solvent. The sparger was blown down with air prior to taking these pictures. Note the defined MCU solvent layer on the pump. This layer is due to the air entrained during mixing at the lower tank levels and is consistent in height with the layer on the side of the tank walls. The wetted parts of the tank and internal components were cleaned using a soapy rung out rag and inspected using the UV light immediately after the visual inspection was completed.

3.4.1.1 DWPF SEFT Minimum Tank Volume

During the 230 RPM, 1/6th SEFT testing, air started to be entrained around 10.1 gallons or 5.5 inches of liquid level. Per personal communications with Dr. Art Etchels, DuPont Mixing Consultant, given the geometric similarity used in this task, linear scaling for the onset of air entrainment in the DWPF SEFT from the 1/6th SEFT is appropriate. Hence, the minimum tank level would be 34.2 inches or approximately 2400 gallons.

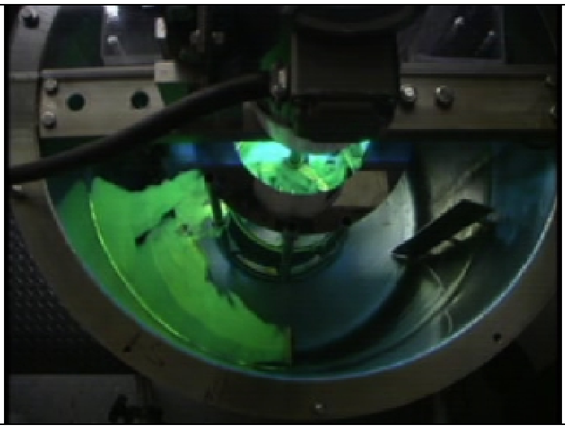
3.4.2 422 RPM MCU Solvent Mixing Testing

In the 422 RPM test, 151,382 grams of DI water were added to the tank and then 13.718 grams of 69.7 wt% HNO_3 solution were added to the agitated tank, resulting in a solution of 0.001M HNO_3 having a measured pH of 2.94. Mixing occurred at room temperature, which was approximately 22°C. Prior to starting this test, the agitator was run at 87 RPM when the solvent was added to see what would happen if the mixing times on both scales were approximately the same. This was the lowest speed achievable by the variable speed drive unit, not 67 RPM, which would have been equal mixing times. During this part of the test, 35.59 gram of dyed MCU solvent was added to the top of the tank. No solvent entrainment occurred, which was expected since the flow patterns in the tank did not generate the vortices necessary for entraining the MCU solvent. This can be seen in the first snap shot in Figure 3-5.

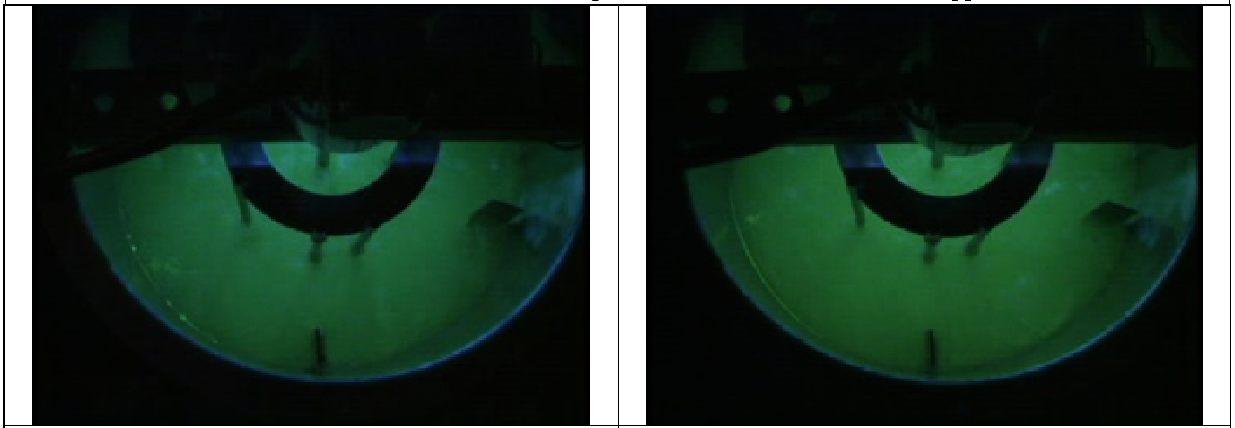
The agitator speed was then set to 422 RPM and started. Almost immediately, the solvent was entrained and dispersed throughout the mixing tank, and the results after one minute of mixing can be seen in Figure 3-5. The MCU solvent droplets in this test were visually much smaller and numerous as compared to the 230 RPM test. The contents were then allowed to mix for 30 minutes prior to sampling. After 30 minutes of agitation, the contents seemed to be slightly greener in color as compared to the one minute of mixing as seen in Figure 3-5. There is no explanation for this slight color difference. No air entrainment was evident during this time of mixing. Some sight buildup of solvent was seen on the side of the tank, on the wall between the 106.45° baffles.

The samples that were pulled after 30 minutes of mixing were analyzed for the modifier (Cs-7SB) and Isopar®L. Once again, of the two methods used to analyze for MCU solvent concentration, the modifier results were more consistent with the visual observations and were those used to determine the state of mixing. These results were then used to calculate the ppm of solvent in the mixing tank and the results are shown in Table 3-4. The quantity of solvent added to the system was 35.59 grams and the amount recovered using the modifier results was 37.91 grams. The difference is most likely due to the sampling location and technique, since the quantity of material added to the tank is less than what was calculated.

The pump sample was pulled 45 minutes into the mixing test. Table 3-4 contains the concentration of the solvent in the pump sample, which was less than that of the tank average. Due to this fact, the buildup rate cannot be determined. Note that because of the very high fluid velocities near the pump suction and small MCU droplets sizes as compared to the 230 RPM test, the inlet into the pump may not be as preferential in entraining droplets relative to the process condition.



87 RPM, Full Tank T= 0 min, V = 40.09 gallons, V_{full} = 9600 Gallons, 235 ppm Solvent



422 RPM, T= 1 min, V = 40.09 gallons, V_{full} = 9600 Gallons, 235 ppm Solvent

422 RPM, T= 30 min, V = 40.09 gallons, V_{full} = 9600 Gallons, 235 ppm Solvent

Figure 3-5 422 RPM Mixing, First 30 Minutes

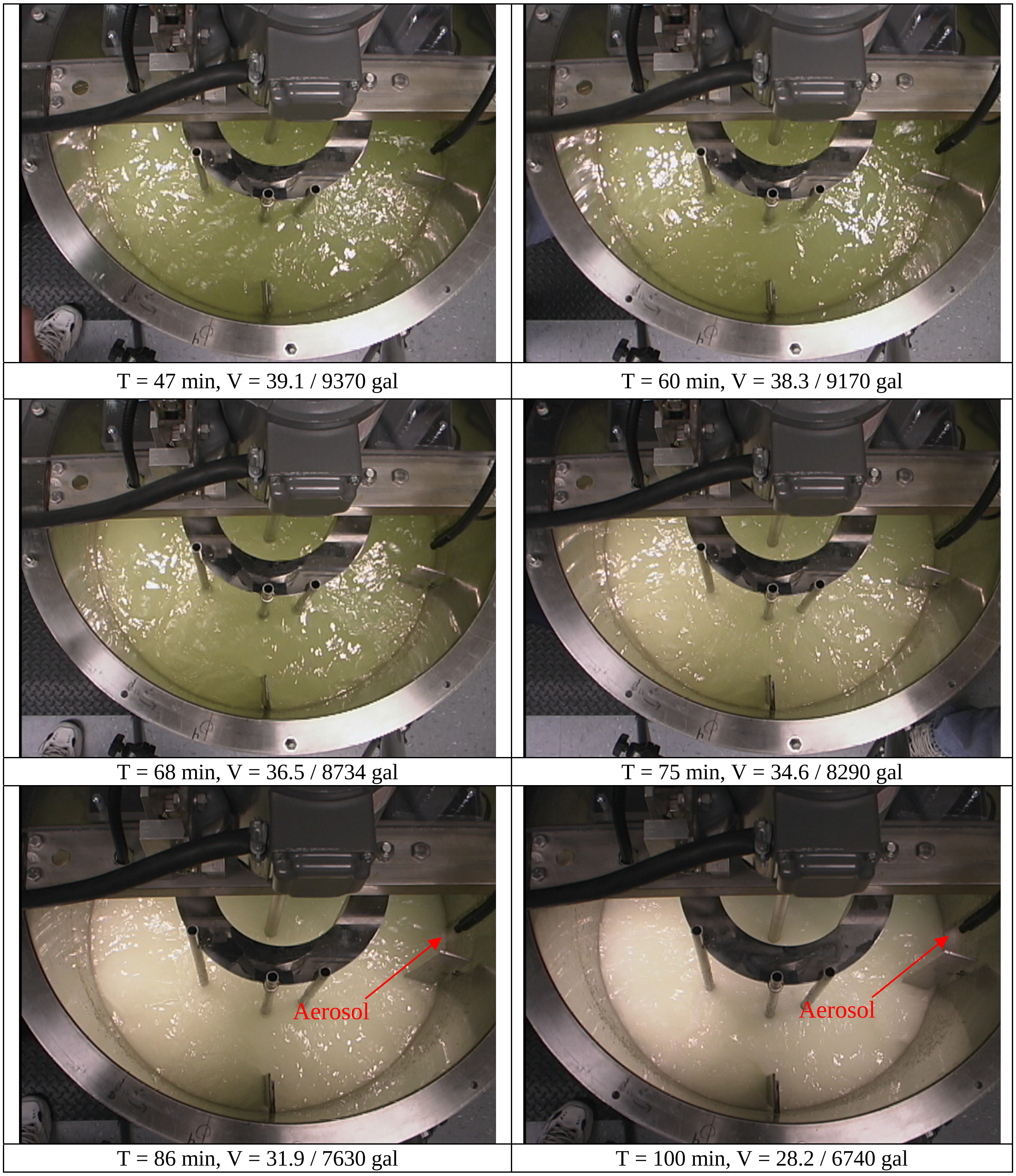
Table 3-4 MCU Solvent Concentration in Mixing Tank and Pump at 422 RPM

Description	Units	Mixing System	Using the following results	
			Modifier	Isopar®L
Mass of solvent added to tank	Grams	35.59	n/a	n/a
ppm based on mass of solvent added	ppm	235	n/a	n/a
18 inches from bottom of tank	ppm	n/a	286	240
15 inches from bottom of tank	ppm	n/a	258	942
10 inches from bottom of tank	ppm	n/a	236	637
5 inches from bottom of tank	ppm	n/a	221	691
Average of 4 measurements	ppm	n/a	250	627
Mass of Solvent in solution	Grams	n/a	37.91	95.12
Pump Sample	ppm	n/a	201	531
Buildup rate of MCU solvent in pump	ppm/min	n/a	n/c	n/c

n/a = not applicable, n/c = not calculated

During the pump down test, the tank contents were removed via the installed scaled pump piping using a peristaltic pump. Key pump down conditions are shown in Figure 3-6 and Figure 3-7. In Figure 3-6, it becomes obvious that at T = 68 minutes, some aeration was occurring, since the solution is slightly less green in color when compared to T = 60 minutes. As the contents are continuously pumped down, more and more air is being entrained. At around T = 79 minutes, aerosol generation was visually observed via the optical lamp used to light up the tank. After T = 86 minutes, the quantity of air entrained had turned the solution whitish. At T = 100 minutes, the mixing task was stopped due to high (not quantified) aerosol generation rate that was impacting personnel safety. Approximately two to three minutes after the mixing had stopped, the surface was recorded and small micro bubbles were observed popping at the surface as shown in Figure 3-7. The tank volume at this condition was 28.2 gallons and the level in the tank was above the top impeller.

The mixing performance below this level is unknown, due to the generation of the aerosols.



T = 47 min, V = 39.1 / 9370 gal

T = 60 min, V = 38.3 / 9170 gal

T = 68 min, V = 36.5 / 8734 gal

T = 75 min, V = 34.6 / 8290 gal

T = 86 min, V = 31.9 / 7630 gal

T = 100 min, V = 28.2 / 6740 gal

Figure 3-6 Key Pump Down Results During the 422 RPM Test

Aerosol Observed From Optical Lamp T = 79 min

Micro Air Bubbles Bursting At Tank Surface

Figure 3-7 422 RPM Mixing, Aerosol Observations

3.4.2.1 Methods of Aerosol Generation at 422 RPM

The aerosol particles (bubbles) that were generated can potentially come from two different sources. One is from the free turbulent surface. The other is from cavitation in the low pressure region of the impeller.

Air is entrained from the turbulent surface (Figure 3-8) of the agitated vessel, where vortices trap the air and draw it into the high shear mixing zone of the bottom impeller. The air is then sheared with the solution, creating micro bubbles in the trailing vortices leaving the impeller. The MCU solvent seems to prefer the interface between the 0.001M HNO_3 solution and air since testing with only water at 422 and 600 RPM did not generate aerosols. A portion of these bubbles pop at the surface, which generates the aerosols. During the water runs performed using the SRAT internals, there was never any indication of aerosol generation at 422 RPM and at tests performed at 600 RPM. The 600 RPM tests are not documented in this report, since the intent was to observe mixing based on 230 and 422 RPM. During pump down, there were no observed vortices as shown in Figure 3-8. Unstable vortices, which are quickly generated and collapsed due to the very turbulent surface motion, could be considered a source. A transparent mixing tank would be required to observe surface entrainment.

On the back side of a rotating flat blade impeller, there is a low pressure zone. If this pressure is lower than the vapor pressure of the fluid (either the 0.001M HNO_3 solution or MCU solvent[24]), the gas formed by cavitation is vaporized liquid (e.g. water vapor or solvent vapor) generated on the back side of the rotating impeller blades. This gas can then be entrained into the trailing vortices of the impeller, creating the micro bubbles. As the tank level is lowered, the amount of head available decreases, which further reduces the pressure in the low pressure zone. The pressure on the back of the impeller is a function of impeller tip speed, fluid density, and tank level. Given the same tip speeds in the DWPF and 1/6th SEFT, the tank level at which micro bubbles are generated would be the same in both tanks. Note that the tip speed of the DWPF SEFT is the same as that of the 422 RPM 1/6th SEFT. The initial level of air entrainment was observed at 36.5 gallons or 20 inches of tank level. Comparing this to the recommended

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[24] Hansen, C. A., Assistant Manager Waste Disposition Project, Letter to Mrs. V. G. Dickert, "Information on Caustic-Side Solvent Extraction (CSSX) Solvent Properties (Your Letter CBU-PIT-2004-00015, Dated 1/14/2004)"

minimum tank level of 34.2 inches, this micro bubble generation should not occur in the DWPF SEFT. SRNL will pursue a calculation to support this assessment.

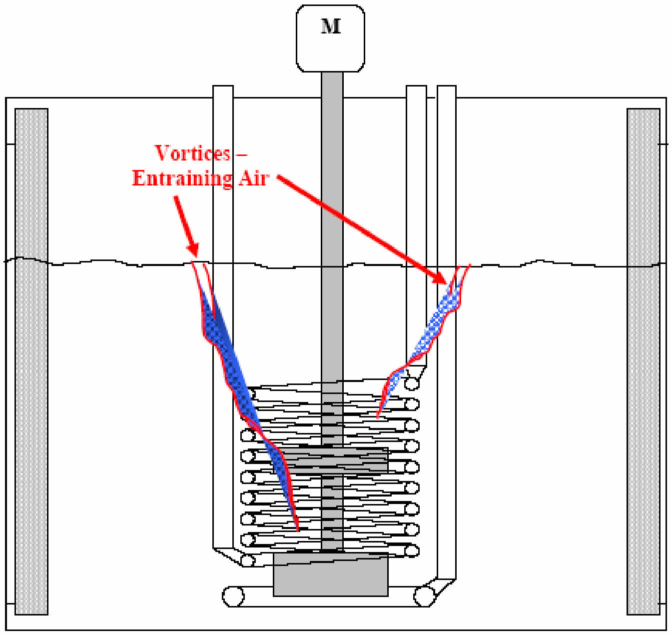

Figure 3-8 Air Entraining Vortices

3.4.3 Temperature Effects

The droplet size, given the physical dimensions of the agitator, impeller power number and agitator speed, is proportional[8,11,22] to the interfacial surface tension to the 0.6 power. The tests performed on the 1/6th scale SEFT were performed at room temperature (22°C). The present maximum temperature for SEFT operations is 45°C. The surface tension typically decreases as the temperature increase, which results in a smaller droplet size. Droplet size could be a factor in generating bubbles that result in generating aerosol particles.

Vapor pressure is another temperature dependent property. Vapor pressures increase more or less logarithmically with temperature. Higher temperatures will promote cavitation in the impellers at lower speeds. Vapor pressure data already exists for both the MCU solvent[24] and water. Vapor pressure data for the 0.001M HNO_3 solution will have to be measured, calculated or obtained from literature.

3.4.4 Scale-up

Scaling is typically performed on a bench/pilot scale to full scale. Applying this method to the test results, and using tip speed and power per unit correlations, the agitator speeds required for the full scale SEFT to have the same type of mixing are shown in Table 3-5. For instance, if one were to size the 422 RPM test results using power/volume (P/V), the resulting full scale SEFT agitator speed would be 125 RPM. If aerosol generation were solely dependent on P/V, then this would indicate that aerosols cannot be generated at the full scale, since the actual agitator speed is 68 RPM. As discussed in Section 3.1.5, liquid-liquid dispersion is also a method used for mixing immiscible fluids and the results are also shown in Table 3-5. These results indicate that a lower agitator speed would be required for good mixing of immiscible fluids.

Of the two different mixing speeds used, and based on the original sizing of the DWPF agitators, the P/V relationship, or 230 RPM test results would be more realistic of the mixing that would occur in the DWPF SEFT. The type of mixing occurring at 422 RPM was more than adequate to entrain/disperse the MCU solvent.

Table 3-5 Full Scale SEFT Agitator Speeds (RPM) Based On 1/6th SEFT Testing

Scaling Parameter	1/6th SEFT Agitator Speed	
	230 RPM	422 RPM
P/V	68	125
Tip Speed	37	68
Liquid-Liquid	31	57

4.0 CONCLUSIONS

The MCU solvents (including dye) used in this task were fabricated at SRNL and excluded the BobCalix component due to the cost. The solvents were acceptable based on comparing their physical properties with that of the baseline ORNL CSSX solvent[14]. The CO and K43 fluorescent dyes easily blended with the MCU solvent and provided excellent visual aids.

Two different agitator speeds were selected, one using the power per unit volume (P/V) criteria and the other tip speed sizing criteria. Both criterions are applicable to the dispersion of immiscible fluids. The 1/6th SEFT agitator speed was 230 revolutions per minute (RPM) using power per unit volume and 422 RPM using tip speed criterions.

In general, both speeds satisfied the objective of this task, which was the entrainment and dispersion of the MCU solvent. Of the two test speeds tested, the 230 RPM 1/6th SEFT bounds the present operating speed of the DWPF SEFT. Mixing at 230 RPM was adequate in the entrainment/dispersion of MCU solvent and scaling to the DWPF SEFT provided an agitator speed range between 37 to 67 RPM. Scaling the 422 RPM 1/6th SEFT using the P/V correlation yields a speed of 125 RPM, outside the operating speed of the DWPF SEFT. SRNL also recommends a lower operating tank level of 34.2 inches or 2410 gallons for the DWPF SEFT, based on the 230 RPM pump down test results.

The results in this test are based on using a 0.001 M HNO_3 solution and MCU solvent only. If contaminates[11,25] are present in the strip effluent stream (i.e. solids) or SEFT, the results and recommendations made for the dispersion of the MCU solvent in the DWPF SEFT vessel may no longer be valid.

Other mixing issues were noted at these two different mixing speeds and results for each test speed are summarized below.

The following observations were from the 230 RPM 1/6th SEFT mixing test:

- The calculated mixing time is 3.4 times faster for the 1/6th SEFT, which means that the solvent may have more time to agglomerate into larger droplets in the full scale SEFT.
- 77% of the added MCU solvent was entrained/dispersed when sampling at full tank conditions 30 minutes after the MCU solvent was added and the dispersed MCU solvent was both analytically and visually (droplets) homogenous. The other 23% of the MCU solvent was adhering to the internal wetted components of the tank, captured in the sparger or pump cavities, or floating on the top surface.. The 1/6th SEFT full tank condition, 40.1 gallons correlated to the DWPF SEFT condition of 9600 gallons. Given the time of mixing (approximately 30 minutes) after the MCU solvent was added to the top of the tank prior to pump down, a steady state condition with respect to the MCU solvent being entrained/dispersed may not been reached.
- Mixing in the tank was homogenous based on sampling results and visual observation. However, this excluded the very small floating layer of MCU solvent in the quaternary where the baffles are 106.45° apart, any adhesion of MCU solvent on internal wetted parts, and MCU solvent accumulating in the sparger assembly or pump suction line.

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[25] Paul, E. L., Atiemo-Obeng, V. A., Kresta, S. M., "*Handbook of Industrial Mixing – Science and Practice*", Wiley-Interscience, 1st edition, 2004

- Analytical results from the tank sample indicated fairly uniform concentrations from top to bottom. Modifier analysis is recommended over that of the Isopar®L analysis when determining the concentration of MCU solvent.
- A very small floating layer of MCU solvent was present on top of the surface until the volume reached 32.8 gallons (7845 gallons full scale). The floating layer stayed in the quaternary where the baffles are 106.45° apart. The floating layer was not observed in the other three quaternaries, where the baffles were 90° or less apart. The floating MCU solvent layer is expected to occur in the same quaternary in the DWPF SEFT.
- No mixing issues were noted as the level in the tank traversed through the upper impeller.
- Air entrainment was observed at 9.1 gallons (2180 gallons full scale). It was not clear whether the bubbles (coated with MCU solvent) were immediately plating out on the exposed surfaces of the tank.
- Entrained air seemed to phase separate the MCU solvent from the 0.001M HNO_3 solution. The large bubbles were then forced to the outside of the tank and the solvent plated out and adhered to the surface of the tank/internal components.
- At 8.15 gallons (1950 gallons full scale), rapid mixing was an issue for pump operability.
- Post inspection of the 1/6th SEFT showed that the MCU solvent adhered mostly to the upper and lower sections of the tank, which corresponded to locations where the tank maintained static tank conditions (i.e., the level of the tank was not dropping). The method of adding the MCU solvent to the top of the mixing tank may have yielded a larger quantity of adhered material to the top section of the tank.
- MCU solvent will accumulate in the sparger piping/ring. The maximum amount of solvent in the sparger piping/ring will be dependent on the tank operating conditions. There is a potential to increase the MCU solvent concentration in the tank, via the sparger piping/ring during pump down, at the lower tank levels. This condition will not exist if the sparger piping/ring is sparged using water each cycle, prior to pump down.
- Analytical results of the pump suction sample indicate that the solvent is accumulating in the pump at 12.7 ppm/min. This value can be impacted by the sample technique and the calculation method used to determine its value.

The following observations were from the 422 RPM, 1/6th SEFT mixing test;

- The calculated mixing time is 6.2 times faster for the 1/6th SEFT, which means that the solvent may have more time to agglomerate into larger droplets in the full scale SEFT.
- MCU solvent was immediately entrained/dispersed at full tank conditions, 40.1 gallons (9600 gallons full scale).
- The mixing tank was homogeneous based on visual observation.
- Variable depth sampling results seemed to be highly effected by the strong flow field. A gradient existed from the bottom to the top of the tank, where solvent concentration increased as the sample height increased.
- Modifier analysis is recommended over that of the Isopar®L analysis when determining the concentration of MCU solvent.
- The 422 RPM MCU solvent droplets were smaller than the droplets in the 230 RPM test.
- During pump down, aerosols were generated. Air could be introduced via the turbulent surface or if the vapor pressure in the impeller zone is less than that of the fluid.

5.0 RECOMMENDATIONS/PATH FORWARD

The following test plan is recommended to determine if the MCU solvent continuously builds up or reaches a steady state condition in the SEFT:

- Obtain cycle time of SEFT operations and determine how to replicate the conditions in the 1/6th scale SEFT. This includes pump down rate.
- Obtain the latest anticipated MCU solvent concentration for the DWPF SEFT stream.
- The MCU solvent will not be delivered to the top of the surface in the DWPF SEFT as was done on the 1/6th SEFT. Determine a method for the initial incorporation of solvent into 0.001M HNO_3 solution.
- Determine if 1/6th scale sample pump suction inlet needs re-sizing from linear to area and make adjustments as necessary.
- Perform multiple cycles using the 1/6th SEFT at a given agitator speed. The number of cycles will be determined with DWPF Engineering.
- Determine a better pump sampling scheme.
- Upon completion of run, perform inspection of buildup and accumulation of solvent on internal components.

The following recommendations are proposed if aerosol generation or accumulation in the SEFT or its off gas system is considered an issue that needs to be addressed by DWPF with respect to the safe operation of the SEFT:

- SRNL to perform a calculation to determine if aerosol generation can be due to the impeller speed creating a vacuum on the backside of the impeller blade, where the vapor pressure is low enough to generate vapors. Vapor pressure of 0.001M HNO_3 solution will be calculated or obtained from a reference.
- If the above calculation does not support vapor generated bubbles, then;
    - Determine the composition of the aerosol.
    - Determine flammability of the aerosol.
    - Determine physical properties of the MCU solvent at 20°C, 35°C and 45°C.
    - Perform bench scaled (1 to 2 gallons) tests at 20°C and 45°C. Determine speeds at which complete mixing occurs and when aerosol generation occurs for two different MCU solvent concentrations.
    - Perform laboratory (1/6th scaled) tests at 20°C and 45°C. Determine if the agitator, via vapor pressure, produces the aerosol.

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