Dip Leg Plug Formation Evaluation and Abatement Testing in Support of the H-Canyon Accelerated Basin De-inventory

M. L. Restivo

T. S. Rudisill

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REVIEWS AND APPROVALS

AUTHORS:

_____
M. L. Restivo, Separation Sciences and Engineering

_____
T. S. Rudisill, Actinide Materials and Separations Science and Technology

TECHNICAL REVIEW:

_____
M. R. Duignan, Separation Sciences and Engineering, Reviewed per E7 2.60

APPROVAL:

_____
B. J. Wiedenman, Manager, Separations Sciences and Engineering

_____
S. D. Fink, Director, Chemical Processing Sciences

_____
J. M. Duffey, Manager, Actinide Materials and Separations Sciences and Technology

_____
W. H. Clifton, Jr., H-Canyon Outside Facilities and Technical Support

_____
A. M. Hudlow, H-Canyon Engineering Manager

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EXECUTIVE SUMMARY

As requested in the Technical Task Request (TTR), the Savannah River National Laboratory (SRNL) performed testing on the current typical bubbler dip leg design used in the H-Area Canyon (HCA). The TTR has a functional classification of General Services.

Testing was initiated to ascertain if the plugging described in the TTR could be reproduced and, if so, to obtain general metrics on the resulting plugging dynamics, and evaluate possible mitigating methods, including design changes and cleaning/purging protocols. The bubblers were to be suspended in a simulant typical of H-Canyon solutions containing aluminum nitrate and silica at a temperature of 25°C using a nominal bubbler air flowrate of 7.5 SCFH. Emphasis was placed on minimizing equipment changes necessary to accomplish the mitigations, as equipment changes in the existing facility would be costly.

Testing evolved into four (4) phases, concluding that plugging could indeed be reproduced (believed to be attributed to localized evaporation and bridging of crystals in the dip legs). During testing, plug formation information was obtained, including time, pressure, and temperature traces leading to plugging were defined; solution compositions leading to plugging were determined, and various physical characteristics (visual and mechanical) of the crystallized plug were obtained.

Phase 1, with the original bubbler design, showed plugging occurred when approaching solubility limits based on aluminum nitrate concentrations, nitric acid concentrations, and temperature. Phase 2 demonstrated that the plug formation was repeatable, and pressure traces were made of the plug formation occurring. Phase 3 changed the method of data acquisition from manual recording to continuous recording to better monitor bubbler operation as the plug formed, even when below the solubility limits. Phase 4 utilized a new larger-diameter bubbler design alongside the original bubbler design to demonstrate how, in an aggressive solution to expedite plug forming dynamics (very close to solubility limits), plugging formation occurs such that the new nozzle design was less prone to plugging and should be further investigated. It is believed a larger internal diameter (ID), coupled with a length sufficient to keep liquid intrusion limited to the enlarged area of the nozzle, are possible mitigations to future plugging. Due to the three-inch internal diameter (ID) of the tank nozzle through which the instrumentation is installed, increasing the internal diameter of the dip leg would be limited to approximately 1¼ inches. Phases 3 and 4 also indicated that the specific gravity dip leg tends to plug before the liquid level dip leg.

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LIST OF ABBREVIATIONS

CLFL	Composite Lower Flammability Limit
DAS	data acquisition system
DI	de-ionized water
dP	differential pressure
EDL	Engineering Development Laboratory
ELN	electronic laboratory notebook
HCA	H Canyon
ICPES	Inductively Coupled Plasma Emission Spectroscopy
ID	internal diameter
LL	liquid level
M&TE	Measurement and Testing Equipment per 1Q manual, 12-2
OD	outer diameter
PSIG	pounds per square inch, gage
RH	Relative Humidity
RTD	resistance temperature detector
SCFH	standard cubic feet per hour (14.7 psia, 68°F, 36% RH)
SCH	pipe schedule sizes per ASME B36.10
SLPM	standard Liters per minute (14.7 psia, 68°F)
SNF	Spent Nuclear Fuel
Sp. G.	specific gravity of a solution
SRNL	Savannah River National Laboratory
SS	stainless steel
SSC	Structures, Systems and Components
TC	thermocouple
TTR	Technical Task Request
VI	virtual instrument
XRD	X-Ray Diffraction Analysis

1.0 INTRODUCTION

H-Canyon (HCA) vessels containing aluminum nitrate solutions have had issues over the years of plug formation in the dip legs, the instrumentation used to ascertain liquid specific gravity (Sp. G.) and level (LL). Typically, a solution is transferred into a specific tank with the bubbler system operating. Some tanks contain agitators for mixing the solution. The problem has been observed to occur after tanks containing the aluminum nitrate solution have been allowed to sit for an extended period. Once plugging occurs, additions may be made to dilute the solution until the solution is ready for transfer to another vessel using steam jets. The current method of clearing the plug formations is the use of an air and/or steam cleaning rig (Purge Wagon). The first attempt is to use pressurized air to clear plug formations, and failing that, use steam under pressure to clear the plug. This has resulted in approximately 311 manhours of support in 2019 and approximately 194 manhours in 2020 to clear the plugs in the dissolving system.

HCA requested the Savannah River National Laboratory (SRNL) to investigate the plugging issues in the liquid level and specific gravity measuring dip leg bubblers typically used in the HCA tanks containing Spent Nuclear Fuel (SNF) aluminum nitrate solutions (The bubblers were to be suspended in a simulant that also contains a nominal concentrate of silica and operated at a temperature of a nominal 25°C using a nominal bubbler air flowrate of 7.5 SCFH). The dip legs (1/2-inch Schedule 40 SS pipe) are part of the bubbler system, which encompasses liquid level and specific gravity instrumentation. Constant flow air is introduced at a metered rate to the dip legs, and the resulting pressure difference between the liquid level (LL) reference (bottom leg) and the Sp. G. leg (which is located 10 inches vertically above the LL reference leg discharge) provides the specific gravity of the solution as Sp. G.= ΔP / height difference (set at 10 inches). With the specific gravity measurement, the liquid level of the tank is then determined by the pressure difference between the LL dip leg and the vapor space dip leg as h (in inches) = ΔP / (Sp. G.). ΔP in these calculations is in inches of H_2O. Additionally, the liquid level air flow to the dip legs is credited in the safety basis for HCA to keep vapor space hydrogen concentrations below 25% of the Combined Lower Flammability Limit (CLFL).

SRNL approached the problem by developing a phased approach: first, determine if plug formation is achievable and repeatable in a lab setting, and what process or equipment changes could be applied to mitigate the plug formation within the constraints of modifications achievable in the HCA.

The first step started with a mockup of the current dip leg design, and preparation of a typical aluminum nitrate/nitric acid solution that would be found in the HCA vessels to see if plugging could be reproduced. Once plugging occurred, consultation with HCA led to a modified bubbler dip leg design that used HCA operational experience and lessons learned in previous SRNL testing[3] to try and minimize or eliminate this plugging. The governing installation criteria for the new dip leg design was the requirement that the legs must be able to be lowered through a 3 in. internal diameter (ID) hole (tank nozzle) remotely. This dimension was administratively reduced to a 2 1/2 in. ID hole based on HCA operational experience.

Full length of Typical Bubbler System

Vapor Leg

Sp. G. Leg
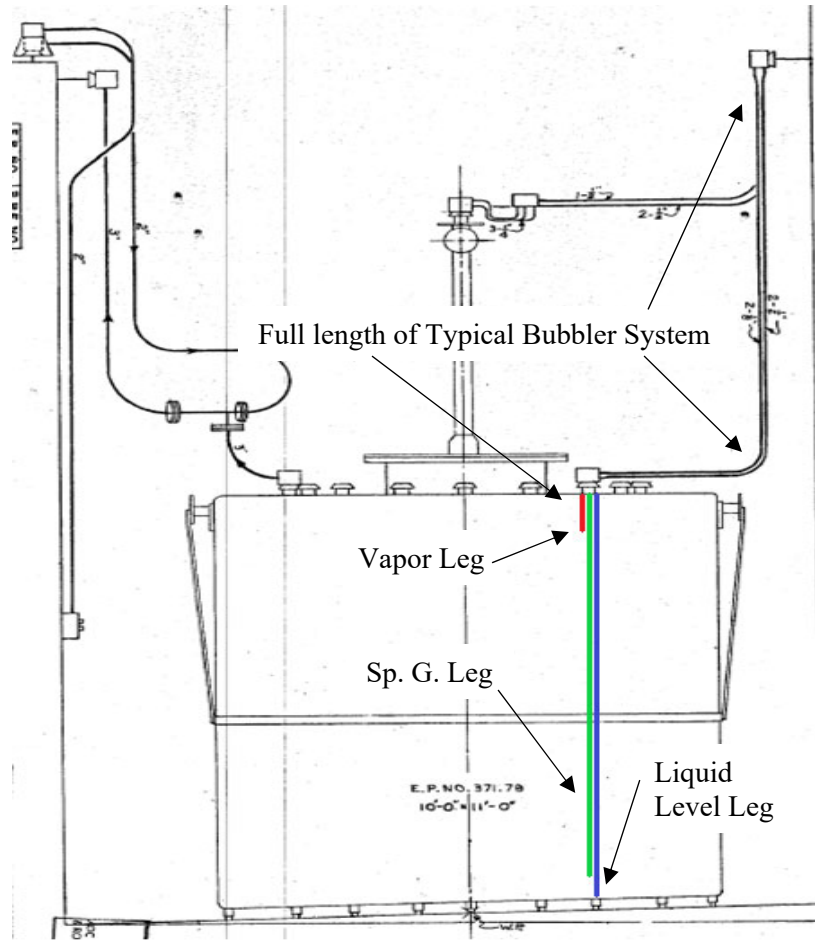
Liquid Level Leg

Figure 1-1. Typical Canyon Liquid Level Piping Arrangement.

For reference, a typical bubbler arrangement is shown above (Figure 1-1 and Figure 1-2). The red leg is the vapor space reference leg, the green leg is the Sp. G. reference leg, and the blue leg is the LL reference (bottom leg).
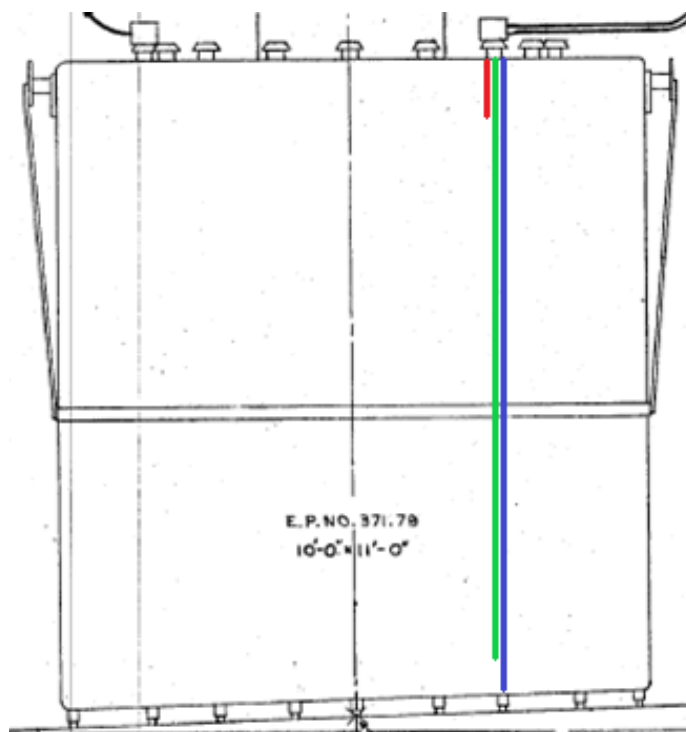
Figure 1-2. Detailed Liquid Level Piping Arrangement.

The specific dip legs used in the initial testing were based on SRS drawing W230479, Revision 1, detail 1, shown below (Figure 1-3).
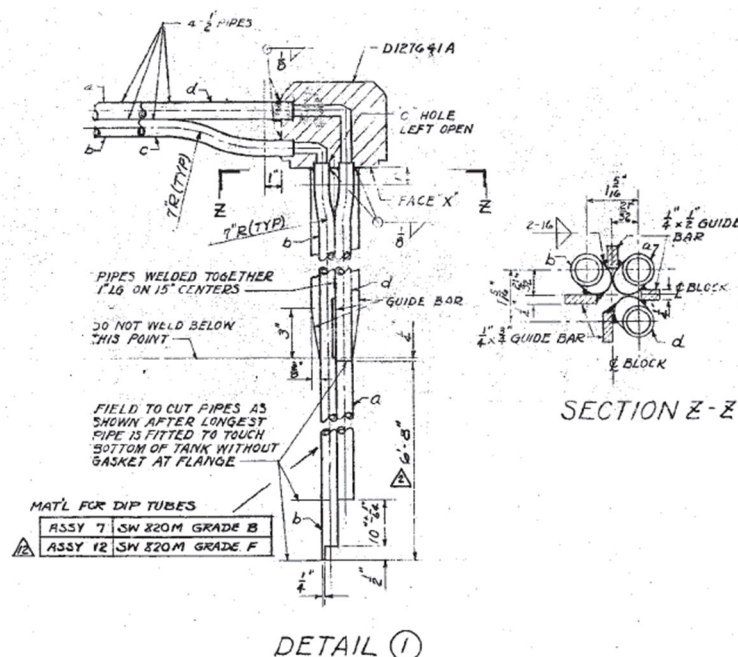
Figure 1-3. Detail of Typical Canyon Vessel Dip Leg.

2.0 EXPERIMENTAL EQUIPMENT

2.1 Piping and Instrument Diagram

The testing (Phases 1 and 2) began with local (manually read) instruments, including differential pressure gauges (0-100 in. H2O), and rotameters (0-10 SCFH). The differential pressure gauges used the high side for pressure input, and the low side was open to atmosphere, resulting in a gauge pressure reading. This configuration is shown below in Figure 2-1. The test rig was modified with electronic instruments after initial testing to better evaluate plugging dynamics. This included the replacement of the rotameters with mass flow controllers, the pressure gauges with pressure transducers, the local readout RTD (Resistance Temperature Detector) with thermocouples, and the addition of a computer with a Data Acquisition System (DAS) continuously reading and recording the electronic instrumentation (Phases 3 and 4). This enhanced configuration is shown in Figure 2-2. HCA directed SRNL to flow air at 7.5 SCFH (3.5 SLPM), as this was typical for the Canyon Vessels.
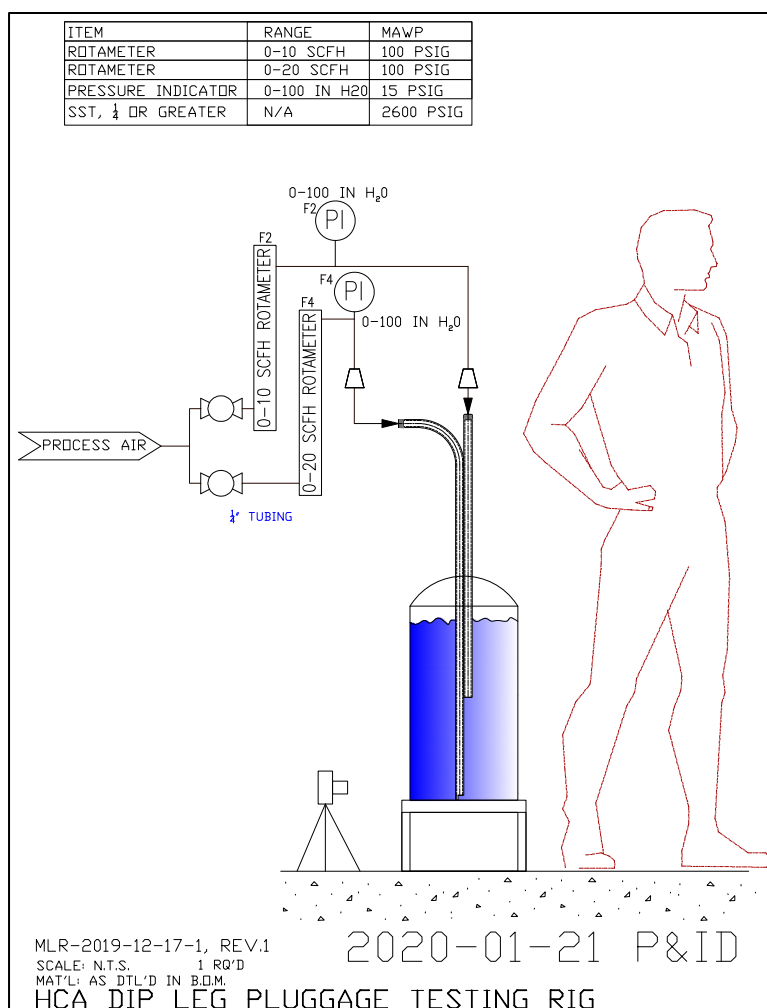
Figure 2-1. Local Instruments Piping and Instrument Diagram.
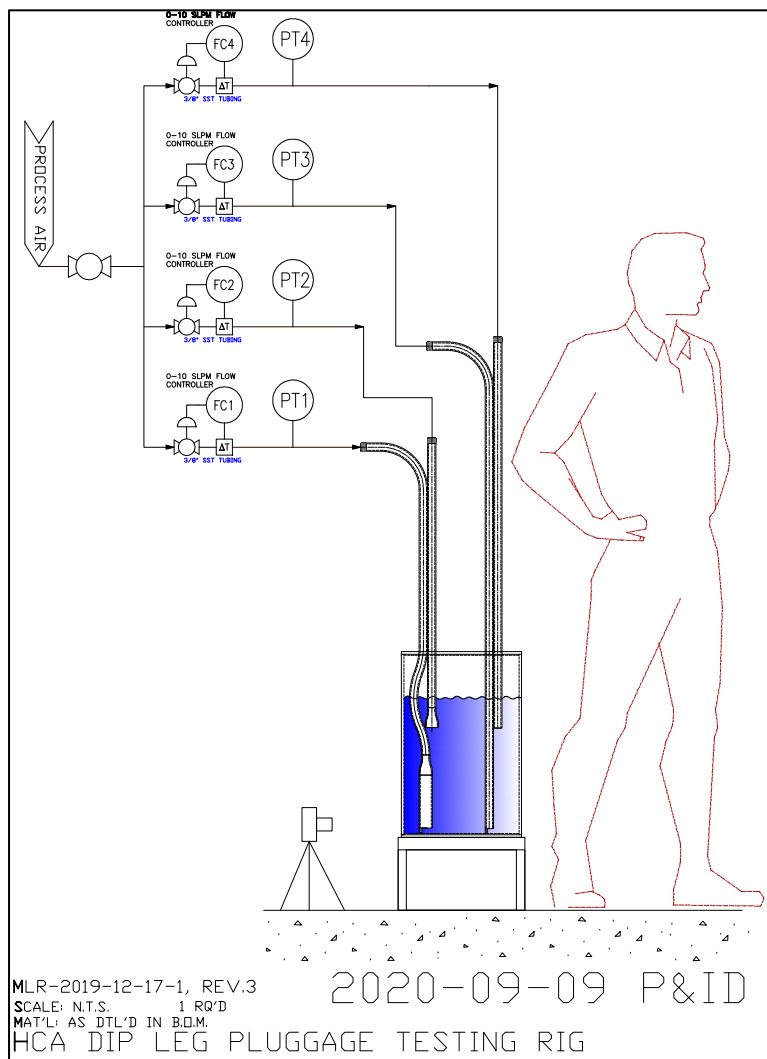
Figure 2-2. Electronic Instruments Piping and Instrument Diagram.

2.2 Mechanical Modifications, Design, and Machine Shop Drawings

Before recommending any major changes, HCA Engineering requested SRNL test the existing bubbler design, which is a bundle of three small tubes welded together. In an initial attempt to increase the internal diameter of standard dip legs (1/2 in. Sch. 40 SS Pipe), several fabrication processes were attempted, all cold worked. The intent of this mechanical expansion process was to determine the amount of expansion achievable on dip leg piping using manual tools available in the field. A reducing diameter mandrel was oiled, and placed into a ½ in. Sch. 40 pipe, which was then placed into an arbor (left photo in Figure 2-3). The arbor was then used to press the mandrel into the workpiece. The maximum internal diameter (ID) achievable through this method was 0.69 in. ID. An attempt was made on an aluminum ½ in. Sch. 40 pipe, and the maximum diameter achievable before fracture was 0.84 in. internal diameter (right photo in Figure 2-3). These values are both above the nominal ½ in. Sch. 40 SS pipe ID of 0.622 in. These attempts were made to ascertain the feasibility of field modifications achievable on existing dip legs. The final results indicate that modifications to existing equipment are not an option.
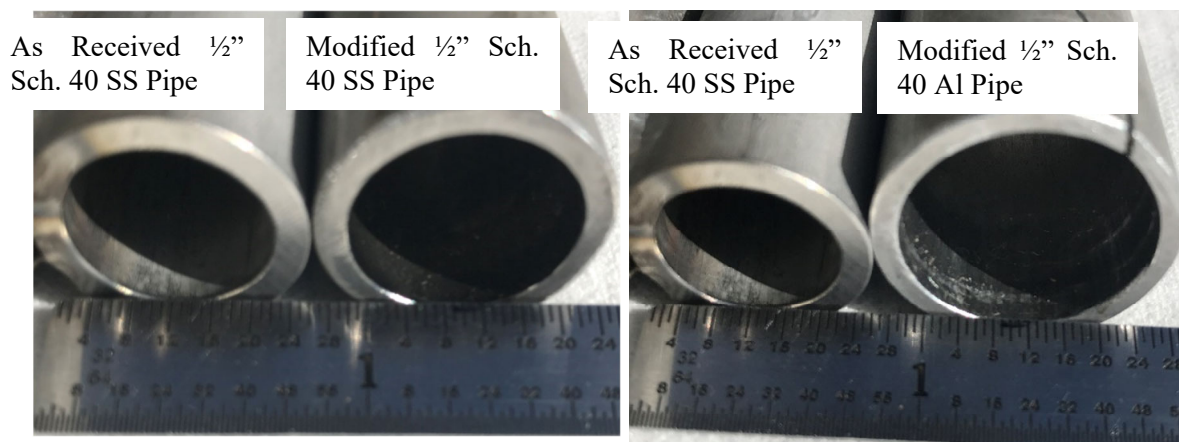
Figure 2-3. Mandrel Expansion Efforts on ½ in. Sch. 40 SS Pipe and Aluminum Pipe.

This cold-working tube-enlargement investigation led to the decision to design a new nozzle head. The new nozzle design model is shown in Figure 2-4. It is based on a standard welded ½ in. x 1 in. Sch. 40 welded reducer for ease of manufacture. The LL dip leg new nozzle has the addition of 5 ½ in. of 1 in. Sch. 40 pipe welded to the welded reducer. The Sp. G. dip leg new nozzle only has the ½ in. x 1 in. welded reducer. The original test rig design and the new design are shown side by side below in Figure 2-5.

The new nozzle design was constrained by the need to insert the full dip leg jumper (all three nozzles, vapor, Sp. G., and LL which are welded together along the seam interface) into a tank nozzle which is 3" in internal diameter. SRNL had to consider how to modify the dip leg nozzles such that the bottom two nozzles, the LL and Sp. G. dip leg nozzles, could still be installed into the 3 in. ID nozzle. This dimension was administratively controlled to 2 ½ in. ID by HCA Engineering based on past experience. This resulted in the new nozzle design geometry shown in Figure 2-4.

Figure 2-4. New Designed Nozzle Model.
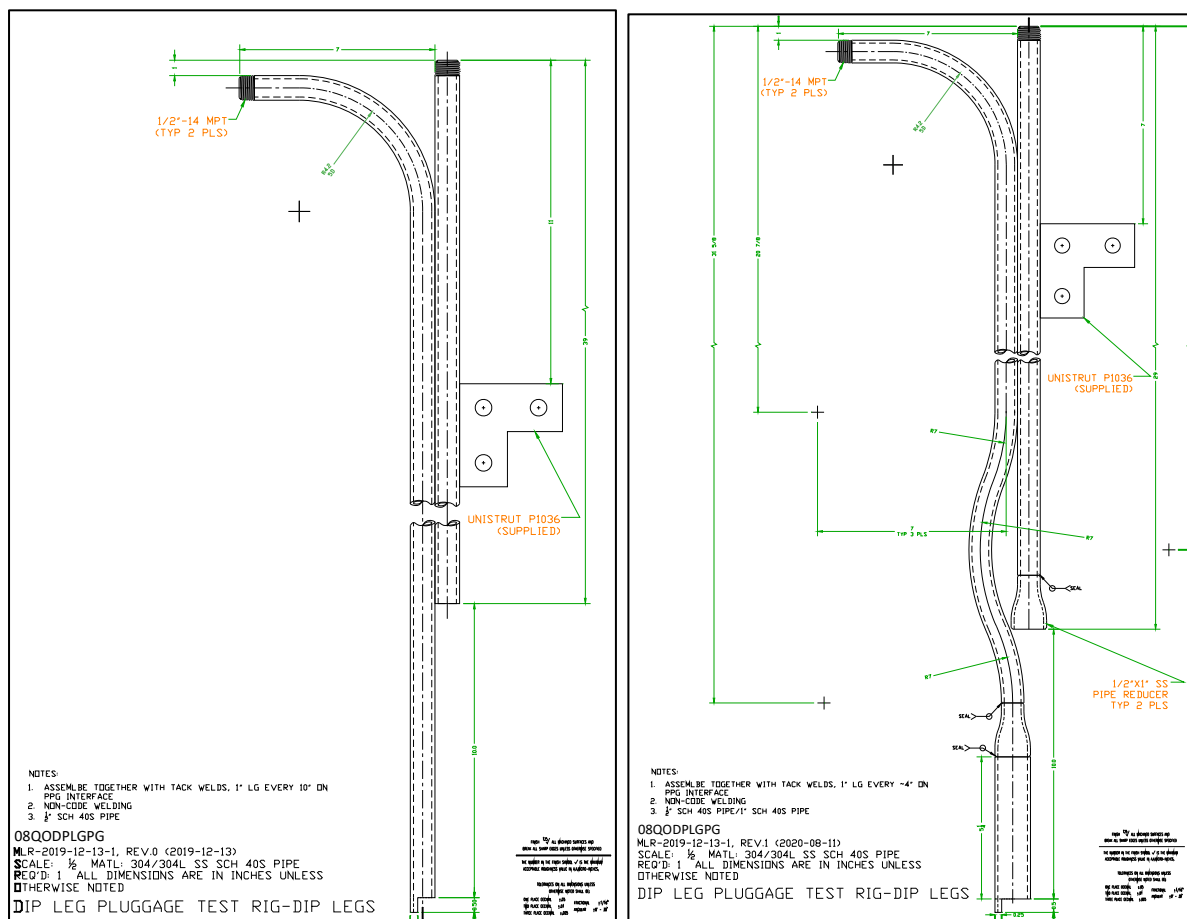
Figure 2-5. Machine Shop Drawings.

2.3 <u>Instruments</u>

A collection of local instruments was identified and installed on the first test rig (local instruments only). The instrument list is shown below in Table 2-1. This initial test rig instrumentation configuration, which required manual recordings, eventually proved insufficient in determining the dynamics of plug formation, and these instruments were replaced with remote instruments with a computer data acquisition system (Phases 3 and 4).

Table 2-1. Phase 1 (Local Instruments) Instrument List.

M&TE #	Instrument Name	MFG	Model	Range	Tolerance
38540	F2 Rotameter	Dwyer	RMB 50 SSV	1-10 SCFH	+/-3% FS
38544	F4 Rotameter	Dwyer	RMB 51 SSV	1-20 SCFH	+/-3% FS
N/A	F2 Differential Pressure Gauge	Dwyer	2000	0-100 in. H_2O	+/-1% FS
N/A	F2 Differential Pressure Gauge	Dwyer	2000	0-100 in. H_2O	+/-1% FS
N/A	RTD	Cole Parmer / Digi-sense RTD	93400-00	-200-850 °C	+/-0.2°C

With the use of non-M&TE equipment, it is good practice to perform a verification comparison of readings over the expected range using calibrated instruments. This was performed for both the rotameters, differential (dP) pressure gauges (used as pressure gauges), and RTD. Using M&TE 44352/44350 gas flow meter, a comparison

was made to verify the flow readings of both rotameters at 3.3 SLPM (7.0 SCFH). The results were all within the manufacturer's tolerance for the rotameters. Additionally, the dP gauges were compared to the readings of the calibrated EDL dead weight tester, M&TE 41913. The RTD was compared to the readings of M&TE 30184 over a range of temperatures close to room temperature, and all RTD indications were all within 1°C of the standard applied.

Once it was determined that plugging was better evaluated with continuous reading instruments, 4 Rosemount pressure transducers, 4 MKS mass flow controllers, and one thermocouple were added to the rig to replace the local instruments. This instrument list is shown below in Table 2-2.

Table 2-2. Phase 2(Rosemounts™ and TC only), 3, and 4 Electronic Instrument List.

M&TE #	Instrument Name	MFG	Model	Range	Tolerance
N/A	Type K Thermocouple	Omega	CASS-116U-12	0-200 °C	+/-2.2°C
36228	FC1	MKS	FC5K-14	0-5 SLPM	+/-2 %FS
47660	PT1	Rosemount	3051CD03A22A1AB4	0-10 PSIG	+/-0.5% FS
36224	FC2	MKS	FC5K-14	0-5 SLPM	+/-2 %FS
39116	PT2	Rosemount	3051CD03A22A1AB4	0-10 PSIG	+/-0.5% FS
33036	FC3	MKS	FC5K-14	0-5 SLPM	+/-2 %FS
46702	PT3	Rosemount	3051CD03A22A1AB4	0-10 PSIG	+/-0.5% FS
36220	FC4	MKS	FC5K-14	0-5 SLPM	+/-2 %FS
46700	PT4	Rosemount	3051CD03A22A1AB4	0-10 PSIG	+/-0.5% FS

2.4 Data Acquisition System

A software package, Labview™, was used for the signal processing. A virtual instrument (VI) was programmed, with the front panel shown below in Figure 2-6, which shows an example of the test in operation. The VI received all the transducer signals, appropriately converted them to engineered units and displayed them. The data was also recorded to a tab-delimited file when requested.

The following is a description of the signal processing hardware used.

Computer
Dell™ OptiPlex 390 running Windows™ XP
Signal Processing Hardware
National Instruments™ Hardware
cDAQ-9174, signal processing chassis for C-Series Signal Modules
NI-9213, C-Series Temperature Input Module
NI-9205, C-Series Voltage Input Module
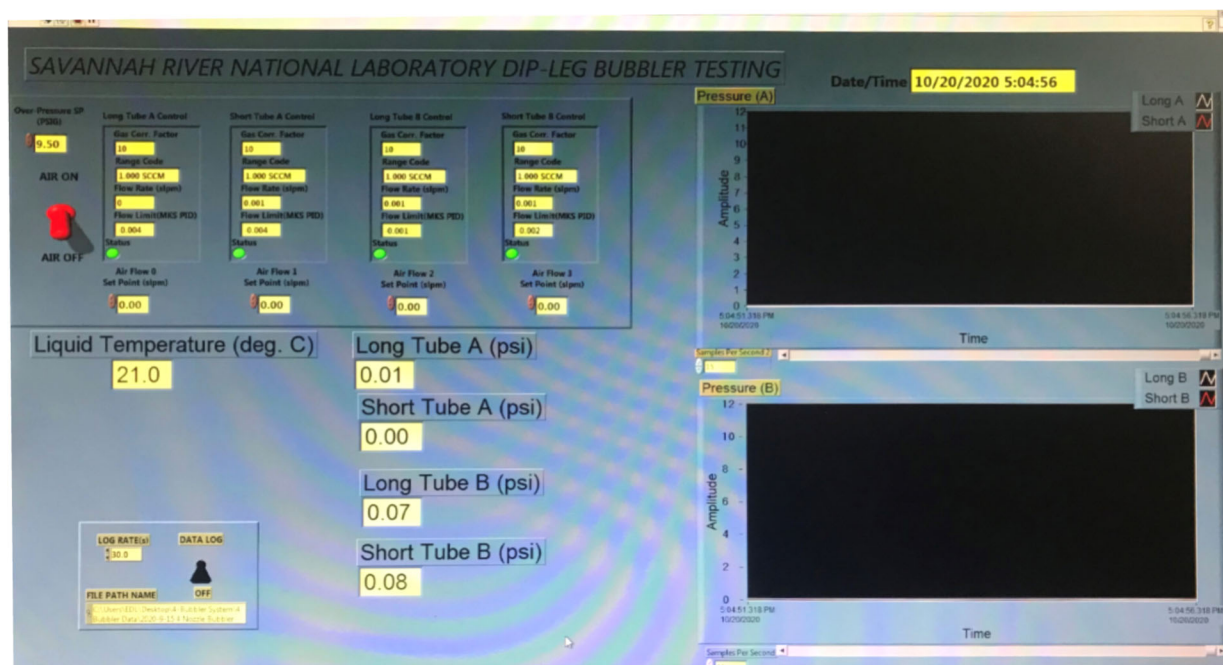
Figure 2-6. Data Acquisition Screen Shot.

2.5 Chemicals

To begin testing, a chemical feed solution was created per the recipe described in the TTR. The specific requirement was 0.2 M to 1.9 M aluminum nitrate, ½ M nitric acid, and a small amount of silica at a temperature of 25°C (this would be adjusted later as testing developed based on input from the customer). All feed solutions began as a modification to nominal 2 M aluminum nitrate solution stored in EDL, with modifications over time as directed by the customer. The intent of the feed solution was to be a simulant similar to solutions in the ABD affected vessels in the HCA. Complete make up details are given in section 3.1. The analytical results of this base solution were obtained from sampling performed in 2019 and is listed as sample No. SRTT15015 in the EDL chemical inventory (Appendix B). This sample number will be used to refer to the test solution, which was the basic feed for all solutions used in testing.

2.5.1 Phase 1 Starting Solution Composition

Using the solution described above, the Phase 1 feed tank solution was prepared using the 2020-5-27 R&D Directions (Appendix C) for Chemical Mixing and Loading. The salient features of the directions are listed below.

- Take 13.2 kg of SRTT15015 solution
- Add 380 mL of 70 wt% Nitric Acid
- Add 7.2 mL of HS-30 Colloidal Silica (30 wt%)
- Mix and add to feed tank

Analytical results for all feeds are given below in Table 2-3.

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2.5.2 Phase 2 Starting Solution (Electronic Pressure Measurements, no flow controller)

This was the same solution as Phase 1. See Appendix D for details of the cleaning and simulant addition.

2.5.3 Phase 3 Starting Solution (Electronic Pressure and Temperature, local flow measurements)

Phase 3 used the final solution of Phase 2 feed (see Appendix E), with the following volume additions:

- Obtained 1 liter of initial feed solution SRTT15015
- Add 38 mL of 70 wt% Nitric Acid
- Add 0.72 mL of HS-30 Colloidal Silica (30 wt%)
- Mix and add to feed tank

2.5.4 Phase 4 Starting Solution (Electronic Pressure, Temperature, and flow measurements /4 Nozzles)

Phase 4 used a new solution independent of prior solutions. Per 2020-9-8 R&D Directions (Appendix F) for Chemical Mixing and Loading (large tank), the following steps were taken to create this solution.

- Obtained 39.6 kg of initial feed solution SRTT15015
- Add 2.25 L of 70 wt% Nitric Acid
- Add 26.8 g of HS-30 Colloidal Silica (30 wt%)
- Add 4,234 g of Al(NO$_3$)$_3$·9H$_2$O
- Mix and add to feed tank

The Solution Matrix, including final solution concentrations and a description of when the sample were taken of all the solutions is given below in Table 2-3.

Table 2-3. Solution Matrix with plug formation information.

Solution	Date/Time	AD ID ID Number	Al(NO3)3 M	HNO3* M	Si g/L**	Description
Initial Target per TAR			2	0.5	0.05-0.25	
Stored Aluminum Solution	5/27/2020	16369	1.99	N/A	ND	
Phase 1 starting solution	5/27/2020	16370	2.059	0.508	0.206	Initial Feed Tank Solution Prior To Testing
Phase 1 Partial Plugging	6/22/2020/8:58 am					Sample Not Taken
	Partial Plugging of SG Leg. Occurred over weekend. Allowed to partially dissolve before startup					
Phase 2 Plugging	6/26/2020/2:30 am	17875	2.419	0.415	0.0461	Solution at time of 6/26 Plugging
						Lvl Leg plugged first, followed by SG leg.
						4 1/2 hours to almost fully plugged
Phase 3 Feed	Per R&D Directions		1.850	0.5	0.0461	
Phase 3 Plugging	7/30/2020/8:12 am	18214	2.411	0.500	0.207	Solution at time of 7/28-7/29 pm Plugging
						SG Leg on 7/28, Lvl Leg on 7/29
						Took approx. 2 hours to almost fully plug
Revised Target per Customer Input			2	1.1	0.05-0.25	
Feed Solution for Phase 4	9/15/2020/8:40 am	18736	2.21	1.1	0.0539	
Original Design SG Leg Plugging	9/16/2020/7:36 am	18835	2.34	0.962	0.0528	
New Design SG Leg Plugging	9/18/2020/7:00 am	18836	2.28	0.96	0.0522	
Original Design Liquid Level Reference Plugging	9/18/2020/2:31 pm	18837	2.29	0.96	0.0454	
New Design Liquid Level Reference Plugging	9/21/2020/7:14 am	18838	2.22	0.94	0.0398	

*-HNO3 Target for phases 1, 2 and 3 was 0.5 M. The target for Phase 4 was 1.1.
**-Silicon concentration varied during testing.

The silicon concentration was targeted for 0.05g/L to 0.25 g/L. Samples were taken as dip samples from an unagitated tank, with no filtration applied. Silicon is sometimes difficult to analyze in salt solutions for two main reasons:

1) Silicon compounds have varying degrees of solubility. Sometimes they do not dissolve cleanly, precipitate later, or form colloids.
2) The analytical method, Inductively Coupled Plasma Emission Spectroscopy (ICPES), sometimes can give a low bias to the Si result without a special sample preparation.

It is likely that the Si in solution formed colloids or fine solids over time, which reduced the Si in solution that the analytical method would capture.

2.6 Time Lapse Photography

A time lapse setup was installed to obtain evidence of plug forming. This configuration is shown in Figure 2-7.

Figure 2-7. Time Lapse Photography Setup.

The original intention of the time lapse photography was to allow documentation of gradual plug formation with photos taken external to the dip legs. The belief was that plug formations would be sufficiently observable outside of the dip legs to glean information on their plug dynamics. As would be shown later, plug formations do extend beyond the exterior of the dip legs (See Figure 3-5). However, because of the varying opacity of the bubbling solution and that the plug material was found deep within the bubblers, time lapse photography proved insufficient to document the plug forming dynamics.

2.7 Video Inspection Technique

Video inspections were performed after testing to check plug formation upstream of the nozzle exits. A 3/8 in. diameter endoscope camera was snaked from upstream of the plug, and slowly inserted down the dip legs to document plug formation. This proved to be valuable in the determination of plug forming dynamics. Heights above the discharge were correlated with imaging to determine the characteristics of plug forming towards the discharge of each leg.

2.8 R&D directions (stored in SRNL ELN Experiment O9117-00066-11)

Phase 1-2020-5-27 R&D Directions for Chemical Mixing and Loading 1 (Appendix C)
Phase 2-2020-6-29 R&D Directions for Bubbler Removal and Sampling (Appendix D)
Phase 3-2020-7-20 R&D Directions for Chemical Mixing and Loading 2 (Appendix E)
Phase 4-2020-9-8 R&D Directions for Chemical Mixing and Loading (large tank) (Appendix F)

2.9 Quality Assurance

As required in the TTR[1], a Functional Classification of General Services applies to this work. Equipment with a General Service functional classification comprises the analytical measurement systems used to collect data for this testing. Standards used to calibrate these systems were purchased at Level 2 with a certificate of analysis. Chemicals and reagents used in testing and sample preparation were purchased at levels 2 or 3.

Requirements for performing reviews of technical reports and the extent of review are established in Manual Procedure E7 2.60, Section 5.2. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in form WSRC-IM-2002-00011, Rev. 2.

All work performed in this report were commensurate with the General Services designation.

3.0 RESULTS AND DISCUSSION

The results of the Phase 1 through Phase 4 dip leg testing are provided in the following sections. A test matrix detailing the research approach is shown below (Table 3-1).

Table 3-1. Test Matrix.

Test Matrix					Instruments	
Phase	Purpose	Nozzles	Temperature	Flowrate, SCFH	Local	Electronic
1	Can plugging be induced, and under what approximate conditions	2 Original Design	Nominal 20 °C	Nominal 7.5 SCFH	Pressure, Temperature, and Flow	N/A
2	Can plugging be duplicated, with continuous pressure traces	2 Original Design	Nominal 20 °C	Nominal 7.5 SCFH	Temperature and Flow	Pressure
3	Details of plug formation with pressure and temperature, and flow locally recorded	2 Original Design	Nominal 20 °C	Nominal 7.5 SCFH	Flow	Pressure and Temperature
4	Compare new design to old design in an identical solution	2 Original Design, 2 New Design	Nominal 20 °C	Nominal 7.5 SCFH	N/A	Pressure, Temperature, and Flow

3.1 Simulant Development and Solubility Discussion

The solution of interest is the result of the HCA dissolution of spent fuel elements. The aluminum-clad uranium alloy fuel dissolved in the HCA is mostly aluminum. The uranium is a minor component of the fuel. The objective when dissolving fuel is to maximize the concentration of uranium in the solution within the constraints of nuclear safety. However, the amount of aluminum which must be dissolved controls the dissolution. The solubility limit of Al in the nitric acid solution is reached before the criticality safety limit is reached for the concentration of U in solution. HCA practice is to dissolve to about 1.7 M Al which is below the solubility limit for Al in the 1-1.5 M HNO_3 which is left at the end of the dissolution. The concentration of uranium would likely be around 3-5 g/L. This mode of operation was the basis for the chemical makeup of the simulant used in testing.

Aluminum nitrate (Al(NO$_3$)$_3$•9H$_2$O) forms colorless, rhombic crystals. It is deliquescent and melts in its waters of hydration at 73 °C and decomposes at 135 °C. The aqueous solution is weakly acidic because of the hydrolysis of the aluminum ion. Its solubility in water and nitric acid at different temperatures is shown in Figure 3-1[2].

The resulting plugging from this testing will throughout this report be attributed to aluminum nitrate crystallization, although the solution also had silicon in the form of a colloidal silica. The silicon (as aluminosilicates) has the potential to be some part of the solidified crystals. X-ray Diffraction Analysis (XRD) was performed on a sample of the solids, but this analysis was unable to detect any aluminosilicates. The silicon addition was targeted as 0.05 to 0.25 g/L, and consequently the solution (and hence the crystal formations) are mostly aluminum nitrate, if not completely aluminum nitrate.
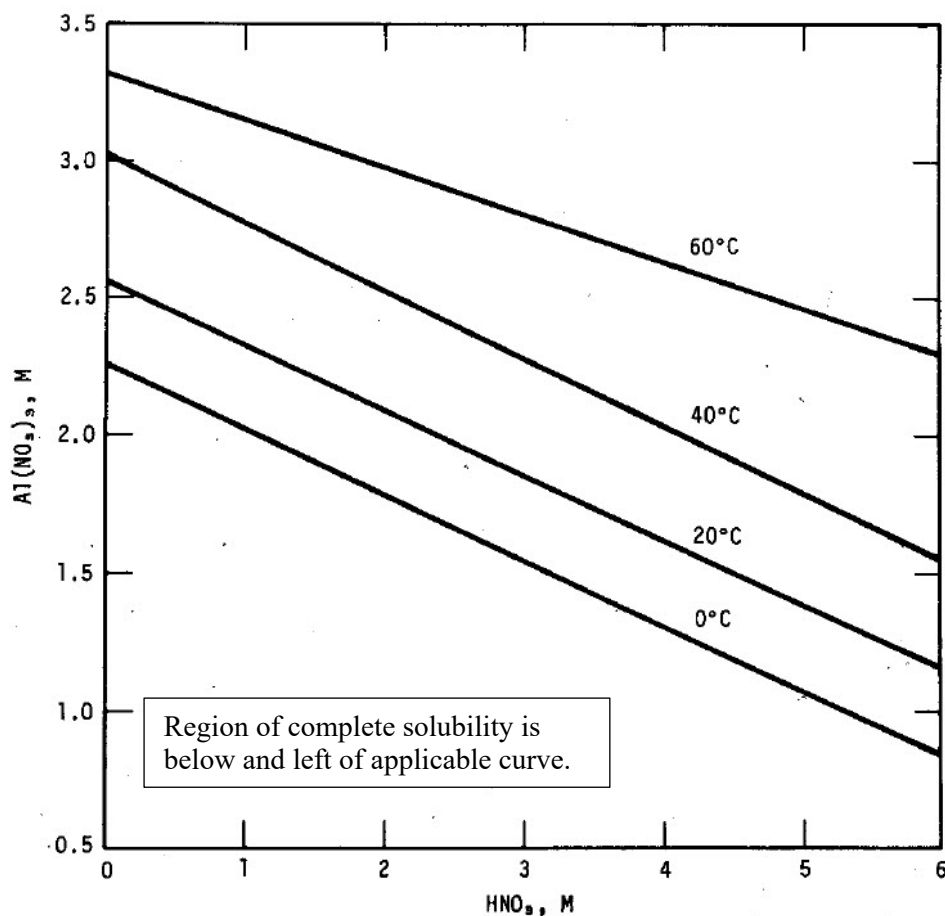
Figure 3-1. Solubility of Aluminum Nitrate in Nitric Acid Solutions[2].

3.2 Phase 1 to determine if Plugging can be initiated (with local instruments only)

Phase 1 testing (Figure 3-2) was an initial attempt to replicate plugging. Local instruments, including differential pressure gauges (low side open to atmosphere) and rotameters were used to measure flow, and rounds were performed periodically to maintain 7.5 SCFH. Phase 1 and all additional phases were performed at nominal room temperatures as agreed upon with the customer.

Figure 3-2. Phase 1 Test Rig.

The original tank in the photo on the left was replaced with the tank shown in the photo on the right based on material incompatibility and freeboard concerns (Figure 3-2). A feed solution was prepared and added to the rig.
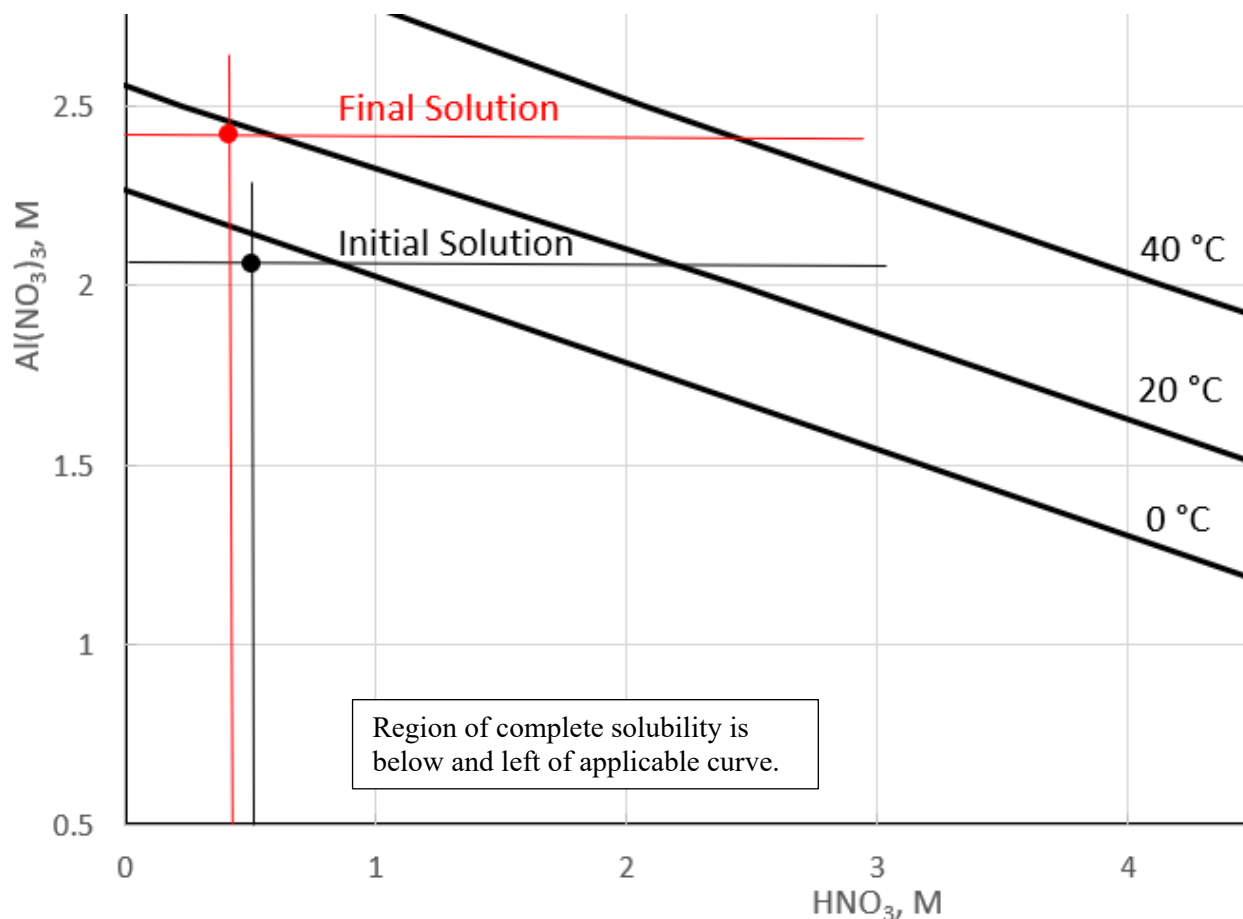
Figure 3-3. Phase 1 Feed Solution and Final Solution[2].

Once the test started, Deionized Water (DI water) was added periodically to compensate for evaporation losses from bubbling operations. This practice was continued from the start of the test on 5/27/2020 to 6/12/2020, with no indications of plugging. Consultations with HCA Engineering led to the decision to stop adding makeup water, which would cause an increase in the molarity of aluminum nitrate overtime and promote plugging. Plugging was initially determined to occur on 6/22/2020, ten days after the suspension of makeup water addition. The final solution concentrations when plugging occurred is shown in Figure 3-3. It should be noted that the analytical uncertainty of the acid analysis is approximately 10%.

The Phase 1 data indicates that the flows were maintained at 7 SCFH and the indicated pressure readings were 18-20 in. H_2O for the bottom leg (liquid level reference) and 6-8 in. H_2O for the Sp. G. reference leg (10 in. above liquid level reference leg). On Friday, 6-19-2020, the Sp. G. reference leg pressure gauge was indicating greater than 100 in. H_2O and was replaced with a 0-30 psig pressure gauge. The test was continued. Sometime between Friday afternoon, 6-19-2020, and Monday, 6-25-2020, a partial plug was determined to occur in the Sp. G. Reference leg. The pressure on the Sp. G. leg on 6-25-2020 was 5 psig (139 in H_2O).

With the plugging phenomenon observed, the rig was shut down for the addition of Rosemount™ pressure transducers, and a thermocouple was added to measure bulk liquid temperature. These changes allowed the continuous monitoring of conditions leading to plug formation. During the downtime for rig modifications, the partial plugging of the Sp. G. reference leg had sufficient time to partially or fully dissolve. This behavior is indicated by the startup of Phase 2 pressure traces, in which the back pressure previously observed has returned to the baseline of approximately 0.4 psig (11 in H_2O).

3.3 Phase 2 to repeat plug formation (with Pressure Transducers and Rotameters-Level reference leg plugging followed by Sp. G reference leg plugging again)

Phase 2 testing was a modification of Phase 1 testing, with the addition of calibrated Rosemount™ pressure transducers to measure the pressure and a thermocouple to measure bulk liquid temperature. Additionally, a data acquisition system was programmed and installed on a computer to take readings automatically.

Because plug formation was occurring during both Phase 1 and 2 testing (as the pressure spikes for Phase 2 shown below in Figure 3-4), the solubility chart for Phase 2 testing (lasting less than 15 hours until full plugging) is the same Figure 3-3.
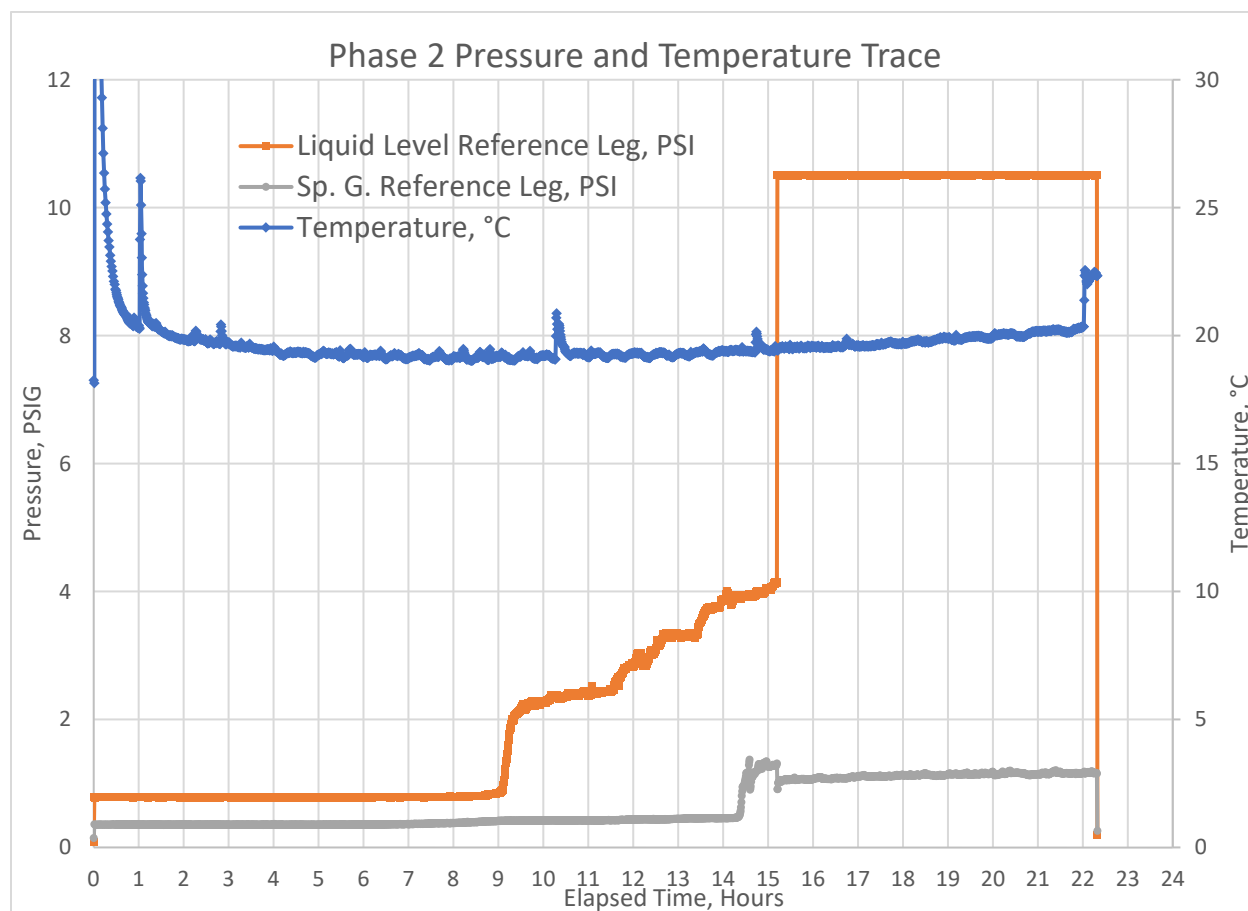
Figure 3-4. Phase 2 Pressure and Temperature Trace.

The large jump in the pressure readings for the liquid level reference leg (Figure 3-4) occurred because that leg had plugged overnight, and the corresponding flow rate had dropped to almost zero. When the flow rate was re-established to 7 SCFH, the pressure reading went above 10.5 psig as indicated. The 10.5 psig indication was the full range of the Rosemount, and so any pressures above 10.5 psig are recorded as 10.5 psig. The Sp. G. leg was beginning to re-form a plug, but testing was halted before the Sp. G. dip leg was given time to fully plug. The test was stopped when the liquid level reference leg became fully plugged.

During the plugging, photos were taken to document the plugging dynamics. Figure 3-5 is a picture of the formed plug on the bottom of the liquid level reference leg while still operating. Air is emanating from the hollow spindles, which appear to be randomly formed "air tunneling" crystals of the aluminum nitrate. This shows the ability of aluminum nitrate crystals to bridge out into open spaces under testing conditions. The picture shows three major hollow spindles bubbling air.
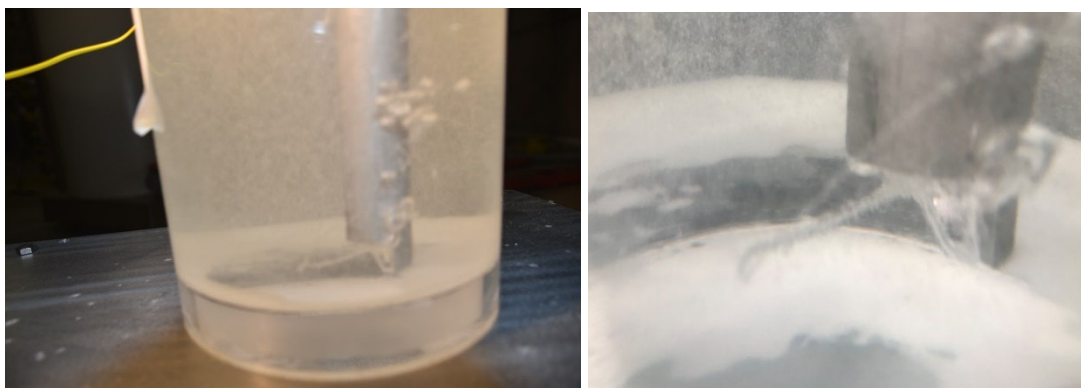
Figure 3-5. Liquid Level Reference Leg Plugging while Operating.

After Phase 2 plugging, the test was stopped, and the dip legs removed for inspection.

Plugging on the Sp. G. reference leg while operating was also documented through pictures as shown in Figure 3-6.
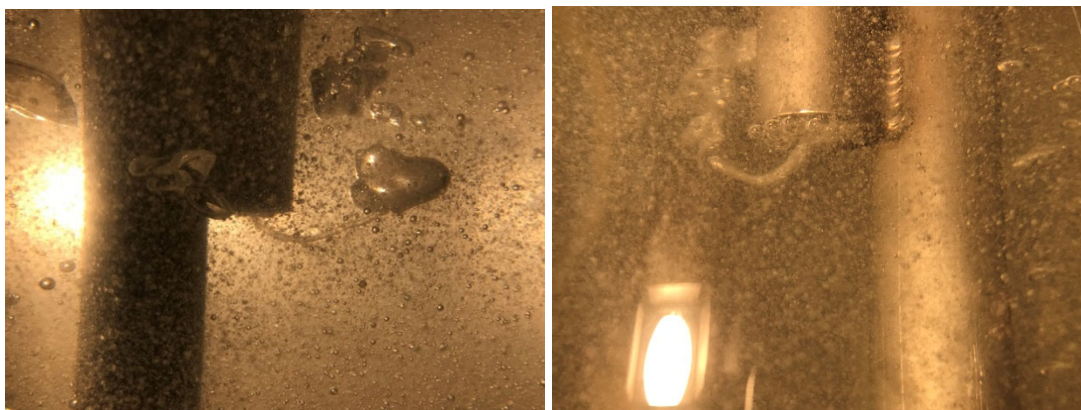
Figure 3-6. Sp. G. Reference Leg Plugging while Operating.

As the solids built up in the bottom of the dip legs, bubbling was continuously observed. The air continued to "tunnel" through areas of crystallized aluminum nitrate, creating pathways for air flow.

Once the dip legs were removed, close up photos were taken as shown in Figure 3-7 and Figure 3-8.

Unique Spindle formation of aluminum nitrate solids in Sp. G. Leg

Sp G plug just prior to mechanical removal (Spindle redissolved)

Figure 3-7. Sp. G. Reference Leg Plugging Closeups.

| Spindle formation prior to leg removal (LL) | Spindle formation redissolved prior to cleaning (LL) |

| End view of liquid level plug prior to cleaning | Plug height with ruler for scale (LL) |

Figure 3-8. Liquid Level Reference Leg Plugging Closeups.

The photos indicate that the short period of time from removal from service (air flow stopped) and dip leg removal from the tank allowed sufficient time for the spindle tunnels to re-dissolve in the bulk liquid, while the main plug remained.

A photo was taken of the liquid level reference leg bottom plug while the leg was in the tank after the tank was emptied (Figure 3-9). The photo shows remnants of the spidering/spindling of aluminum nitrate crystals prior to final dissolution when the leg was removed from the empty tank.
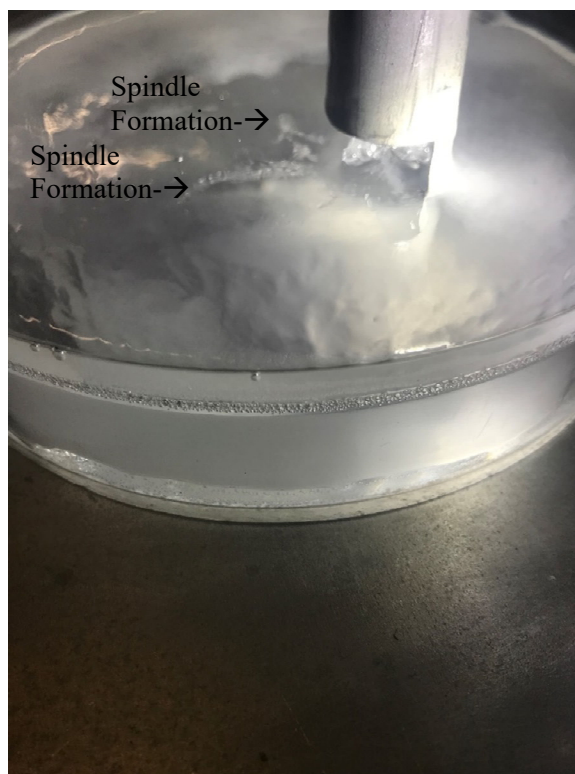
Figure 3-9. Liquid Level Reference Leg Spidering Photo.

Finally, both legs showed the creation of solid aluminum nitrate up to ¾ in. thick (across full ½ in. sch. 40 ID), with additional deposits of crystallized aluminum nitrate up to 3 inches axially from their respective exits as shown in Figure 3-10.

Figure 3-10. Spiraling Crystallization of Aluminum Nitrate in Liquid Level Reference Leg after removing the full diameter end plug.

The plug removal required significant effort. The bottom of the plug formed a hockey puck-shaped solid plug, approximately ½ in. thick that had the consistency of rock candy. Removal efforts required some impact loading on a cold chisel. After repeating impacts, the bottom plugs broke free and revealed the spiraling shown in Figure 3-10. This spiraling required significant scraping to remove. Once the plug was removed, the dip legs were cleaned with Alcanox® and hot water. Pictures of the cleaned state are shown in Figure 3-11.
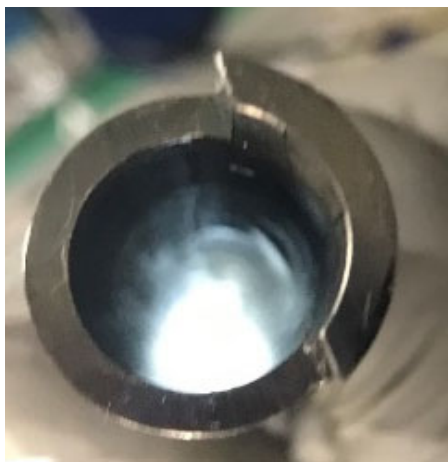
Figure 3-11. Post Cleaning Liquid Level Reference Leg.

3.4 Phase 3 (with Pressure Transducers and rotameters)

With the addition of the DAS and an increased use of electronic instrumentation, pressure, temperature, and flow data could now be obtained around the clock as plug formation occurred. Because of feed solution losses (evaporation) during testing, Phase 3 testing began with the addition of the existing solution from Phase 2 testing as shown in Figure 3-12.
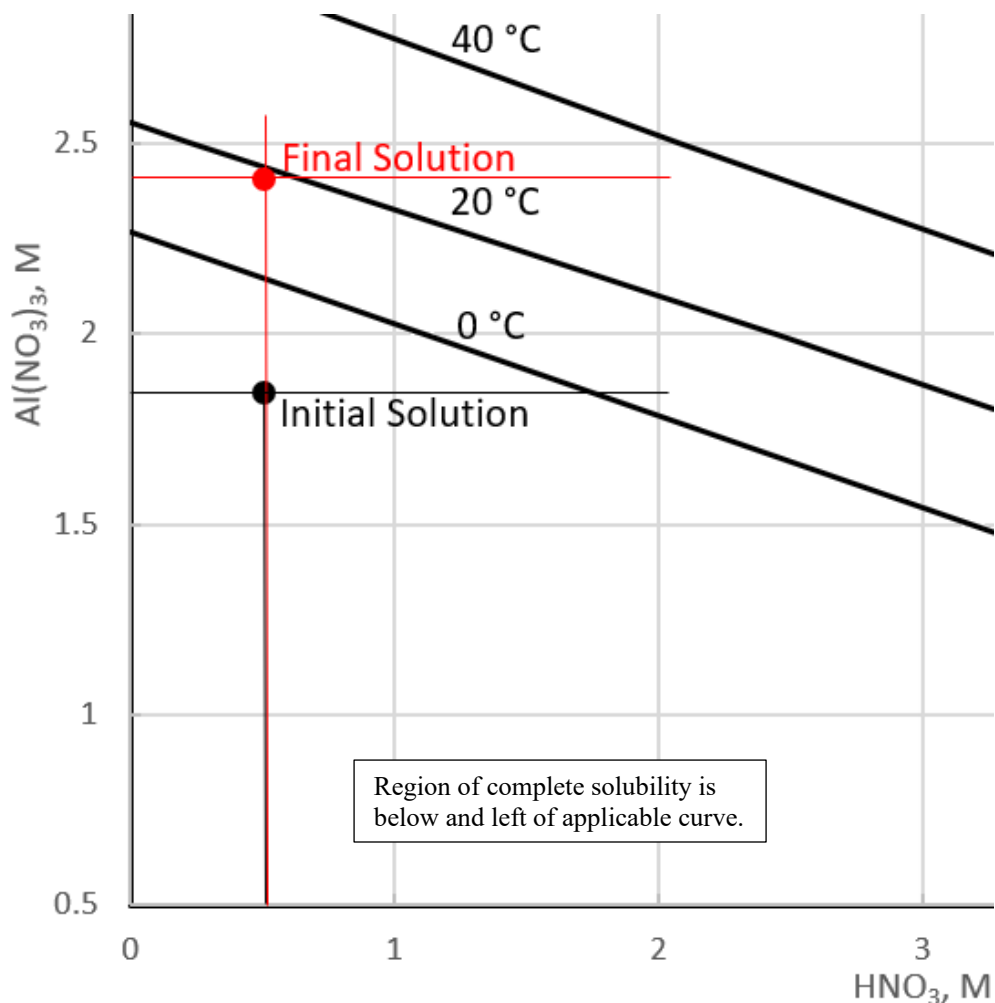
Figure 3-12. Phase 3 Feed Solution and Final Solution.
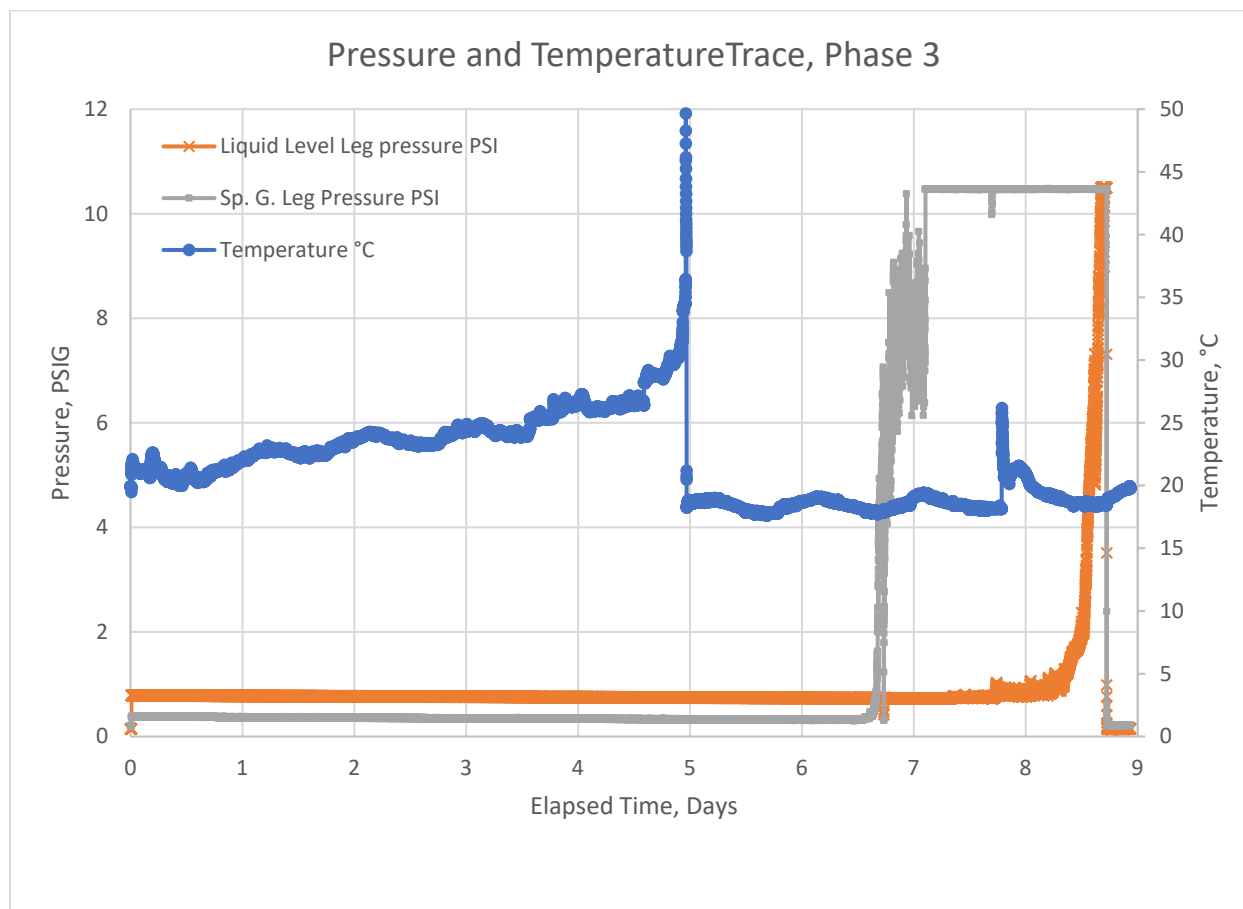
Figure 3-13. Pressure and Temperature Trace, Phase 3.

The Sp. G. leg plugged first, with the plugging occurring within 45 minutes from the first increase in pressure indication at the nozzle. The observed trend suggests that the Sp. G. leg tends to form a plug prior to the LL dip tube (see following paragraph for discussion). The liquid level dip leg (bottom leg) plugged approximately 35 hours later, with the first indications of increase in pressure 24 hours after the Sp. G. leg plugged. The liquid level plug formed more slowly, taking approximately 10 hours for an identical increase in differential pressure as demonstrated by the Sp. G. leg plugging dynamics (3/4 hours versus 10 hours to plug, see Figure 3-13). As the liquid level leg was forming a plug, there was an almost full day of slight pressure increases indicating the pending full plug to form. No pictures were taken, nor video inspections performed during Phase 3 testing. The intent of Phase 3 testing was to keep testing ongoing during the concurrent redesign of the nozzles proceeded forward, and possibly observe any new phenomenon associated with plugging.

From Phase 3 test results it became apparent that the Sp. G. dip leg tends to plug before the LL dip leg in the same solution. This conclusion was confirmed from limited observations of previous testing. Phase 1 indicated Sp. G. leg plugging occurring first. Phase 2 resulted in the LL dip leg plugging first, followed by the Sp. G. dip leg. However, this may be explained by the fact that after Phase 1 testing, both dip legs were allowed to sit in solution for a period of days without any formal cleaning, and the plug formations in both legs fully or partially dissolved over time, leading to possible preferential plug formation of one leg over the other prior to starting Phase 2 testing. Phases 3 and 4 testing would provide a more definitive answer to the order of plugging as will be discussed in the Phase 4 testing results discussion.

The thermocouple readings were observed to be drifting on day 4, and indicated instrument failure; therefore, the thermocouple was replaced at the beginning of the 5th day. This is shown in the precipitous drop off in readings on day 5 (Figure 3-13).

3.5 <u>Phase 4 (Spiked solution with Pressure Transducers and Mass Flow Controllers)</u>

With the addition of two newly re-designed nozzles with larger openings and the cleaned original design nozzles (see Figure 2-2), the original feed tank had to be removed and replaced with a procured acrylic tank. The tank design is provided in Appendix A. The new feed solution was mixed, installed in the new feed tank with all four (4) nozzles, and Phase 4 testing commenced (see Figure 3-14 below).
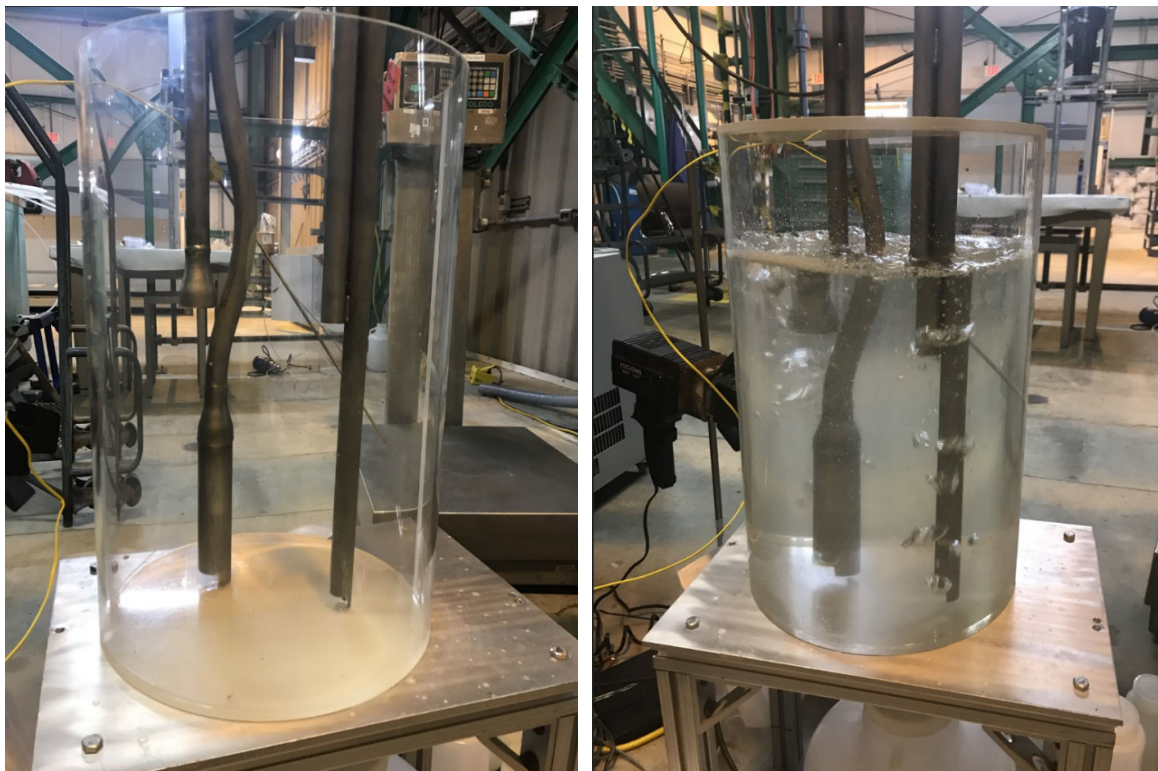
Figure 3-14. Phase 4 Test Set Up.

Feed solution and the final solutions at each of the four plugging times are shown in Figure 3-15 .
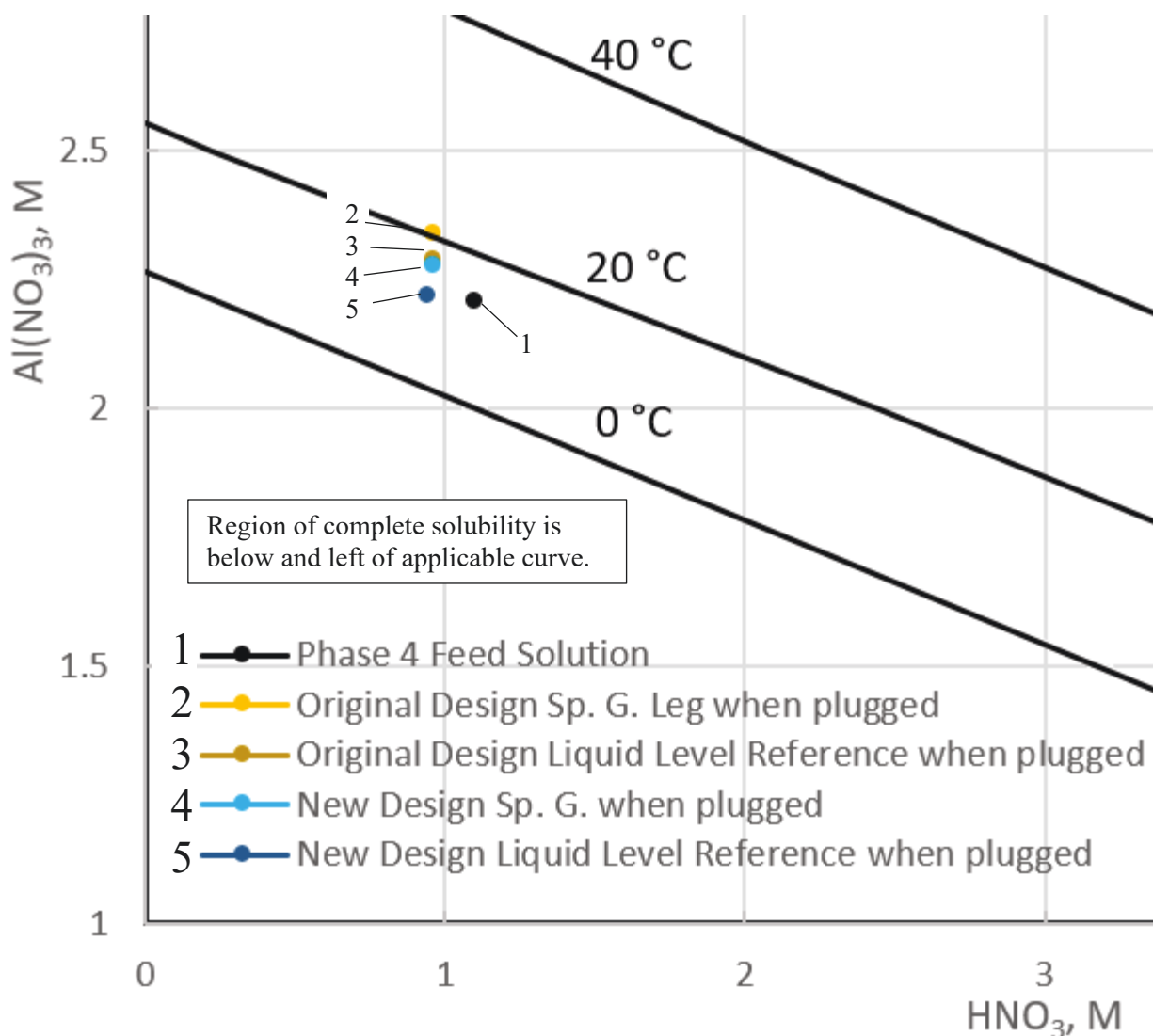
Figure 3-15 . Phase 4 Feed Solution and Final Solutions.

As shown, the feed was purposely prepared to be closer to the solubility limits of the solution to expedite plug forming. As each leg plugged, a sample of the solution was taken and analyzed. Because testing was performed at or below 20 °C, the solution can be seen below solubility limits, the 20°C line being clearly demarcated. The temperature of testing was below the requirements of the TTR but was agreed upon by the customer. The variations in aluminum nitrate molarity fall within analytical tolerances.

The temperature and pressure traces for Phase 4 testing are shown in Figure 3-16.
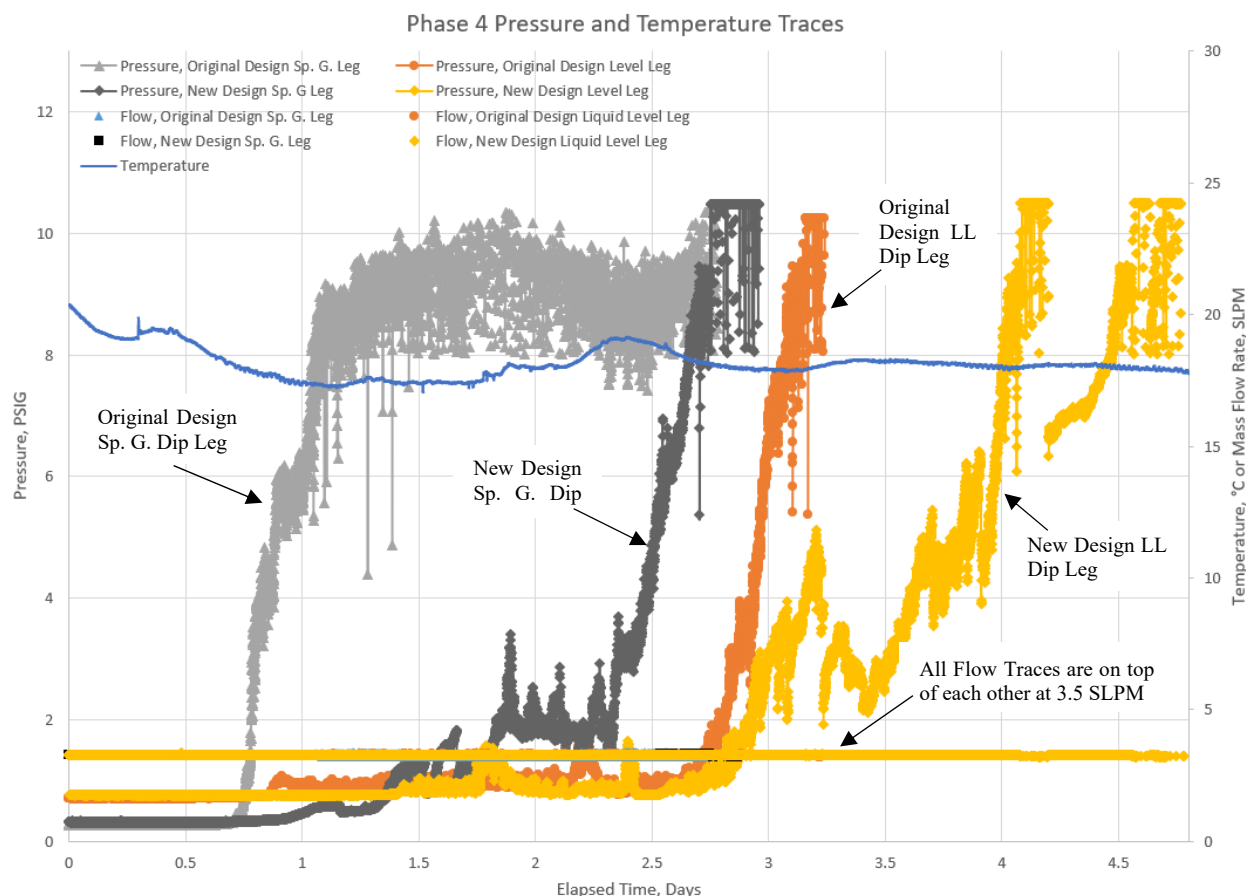
Figure 3-16. Phase 4 Pressure and Temperature Trace.

During discussions on the parameters for Phase 4 testing, it was suggested that periodic flow interruptions be performed to attenuate possible plug formation. The intention was to maximize flow rates at the beginning and end of shifts for each leg for several minutes. This specific criterion was not implemented, but a similar criterion was implemented into the logic of the DAS. A periodic blowdown[3] has been previously reported as a possible successful strategy for removing a plug, including air alone or air and water. Reported water pressure was as high as 90 psig required to dislodge a plug[3]. This blowdown was not always successful.

A different blowdown strategy was implemented into the DAS logic. The DAS was programmed to stop flow when 9 psig was achieved, and time was allowed to vent the specific dip leg through the normal leg piping.

This blowdown strategy was an attempt to clear plugging after the majority of the plug had formed. HCA Engineering had suggested a daily purge of the dip legs by flowing 20 scfh air through the dip legs in the morning and the evening for approximately 1-minute duration each purge. HCA Engineering indicated that 20 scfh is the maximum amount of flow the HCA facility can accommodate to facilitate these purges. These purges were to be done starting concurrent with the test start, and to run the length of the test. Unfortunately, this strategy did not prevent plug formations and will be discussed more fully in the conclusions.

Because plugs were not dislodged significant fluctuations of pressure occurred once plugging caused a greater than 9 psig pressure. The 9 psig pressure was chosen because it was just below the full range of the pressure transducers. The period of these fluctuations was approximately 1 minute. Data were removed to eliminate fluctuation-

associated data spikes so that the pressure traces in Figure 3-16 could be seen better. This allowed the overall pressure trends to be more clearly displayed.

As indicated in Figure 3-16, the Sp. G. dip legs plugged earlier than their similar design mate. That is, the original design Sp. G. leg plugged before the new design Sp. G. leg, and the original design LL dip leg plugged before the new design LL dip leg. Although there are limited data sets, it appears that the Sp. G. legs are likely to form plugs earlier than their mated LL dip leg. The reason for this is not known exactly, but a possible reason is that the Sp. G. dip legs are exposed to a lower hydraulic pressure (higher off the bottom of the tank), and consequently, larger bubbles are being formed due to the lower pressure. These larger bubbles may be driving a higher evaporation rate, and consequently a faster crystallization rate. Additionally, the Sp. G. dip leg is exposed to additional air that is emanating from the LL dip leg located almost directly below it.

3.6 Time Lapse Photography Results

The time lapse photography proved little benefit overall to documenting the plug formation over time (See section 2.6). Two photos are shown in Figure 3-17 to demonstrate information typically obtained from the photographs. The photograph on the left was taken at 3:16 a.m. on 6/26/2020 of the Sp. G. reference leg as plugging was beginning to be indicated through pressure readings. The photo on the right is at 1:16 p.m. on the same day and was the first photo of sufficient clarity to visualize the nozzle discharge.
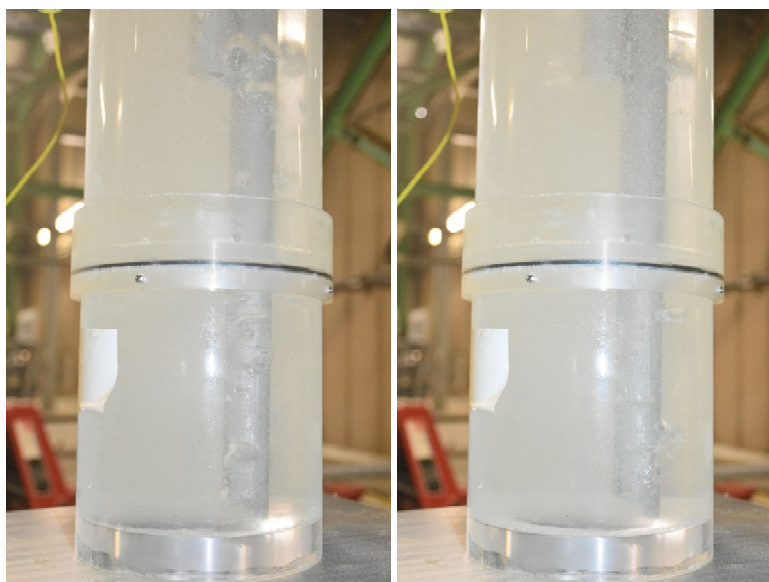
Figure 3-17. Typical Time Lapse Photography Results.

3.7 Video Results

Video inspections were performed after Phase 2 and Phase 4 testing. An endoscope with a 3/8 in. diameter camera was extended from the upstream side of the dip legs, and video was made as the camera extended down towards the plug.

3.7.1 Phase 3 Video Results

Both legs were very similar in the presentation of plug formation from the upstream side of the plugs. Several photos (Figure 3-18 through Figure 3-21) show how the crystallization process progressed. It should be noted that the liquid level for this phase of testing was approximately 4 in. above the bottom of the Sp. G. reference leg. Additionally, the testing commenced with the dip legs submersed prior to the introduction of air purging, and consequently the internal surfaces were exposed to solution prior to testing. This is atypical from plant operations, where the solution is transferred into the tank while the bubbling system is operating. The video inspection of the LL dip leg is shown in Figure 3-22. The main plug formation in the LL dip leg had re-dissolved by the time of the video inspection.

Figure 3-18. Sp. G. Reference Leg, 8 in. from Leg Discharge.

Figure 3-19. Sp. G. Reference Leg, 4 in. from Leg Discharge.

Figure 3-20. Sp. G. Reference Leg, 2 in. from Leg Discharge.

Figure 3-21. Sp. G. Reference Leg, Closeup of Backside of Plug.

Figure 3-22. LL Dip Leg, 2 in. from Nozzle End.

3.7.2 Phase 4 Video Results

During phase 4 testing, because each dip leg pair (Sp. G. and LL dip legs) were welded together to be as prototypical as possible, when a dip leg became plugged, it could not be immediately removed from the solution for endoscope inspection until its mated dip leg also became plugged. Because of this, when a dip leg became plugged an effort was made to prevent the plug from dissolving by capping the top of the plugged tube. This allowed later removal of the assembly for endoscope inspection. Unfortunately, capping did not prevent the liquid in the tank from leading to some dissolution of the plug formation in the capped tube.

Below in Figure 3-23 through Figure 3-27 are screen shots taken from the endoscope video inspection with descriptions.

Figure 3-23. Original Design Sp. G. Reference Leg, 8 in. from Discharge.
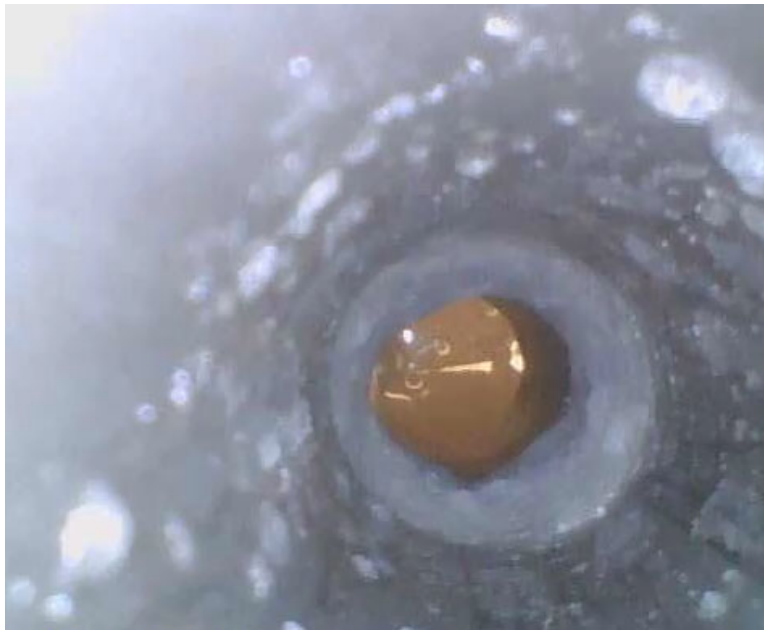
Figure 3-24. Original Design Sp. G. Reference Leg, 2 in. from Discharge.

Figure 3-25. New Design Sp. G. Reference Leg, 8 in. from Discharge.
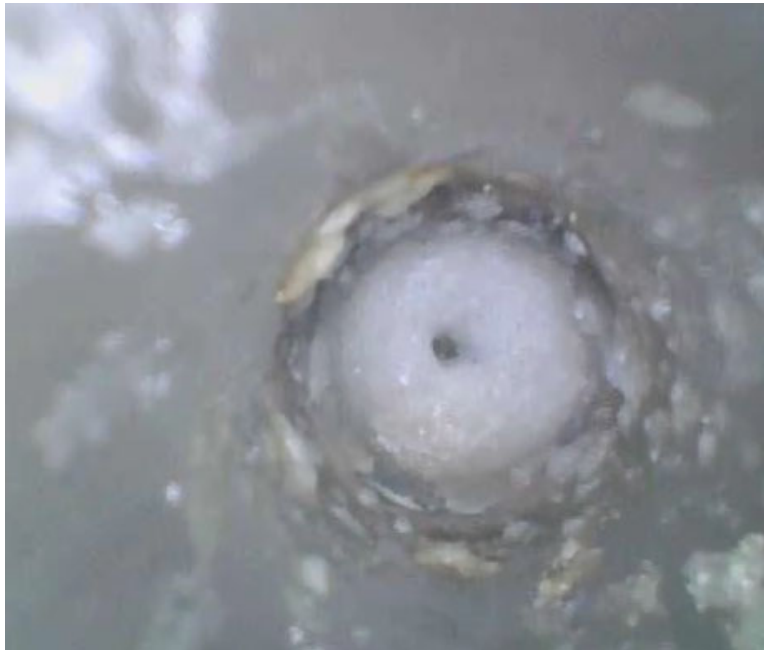
Figure 3-26. New Design Sp. G. Reference Leg, 3 in. from Discharge.

Figure 3-27. New Design Sp. G. Reference Leg, Closeup.

It is important to note that the plugging formed at the narrow section of the new Sp. G. reference leg (within ½ in. of beginning of expansion), with the expanded section completely clear of any indications of plugging. This behavior is shown in Figure 3-28. This section was completely flooded, suggesting that a large diameter tube the full length of liquid height may be a possible solution.

Figure 3-28. Bottom of New Design Sp. G. Reference Leg.

4.0 Conclusions

It appears that localized evaporation is leading to crystal formations at the liquid/gas interface along the ID of the dip leg discharge nozzles, near the bottom. The crystals appear to form on the interior surfaces, and bridge across the internal diameter, bridging across the ID until eventually fully plugging the dip legs.

Phase 1 testing indicated that the plugging occurs in areas close to the solubility limit as shown in Figure 3-1. This leads to the understanding that the farther away from the solubility limit, the less chance of encountering plug formation. Phase 1 testing additionally showed that long periods below the solubility limit did not provide localized evaporation sufficient for crystallization of aluminum nitrate in the dip legs. For example, Phase 1 testing involved a feed solution that was 2.059 M $Al(NO_3)_3$ and 0.508 M HNO_3 at the beginning, and a final solution containing 2.419 M $Al(NO_3)_3$ and 0.415 M HNO_3 (See Figure 3-3). The rig was run for three (3) weeks at the initial solution concentration, with water added to make up for evaporation, and no plugging was detected. Once the addition of water was stopped, allowing the increase in aluminum nitrate molarity, plugging occurred within ten (10) days, with a quick rise in pressure readings in the final three (3) days of operation. Phase 1 also demonstrated that the Sp. G. leg tends to plug first.

Phase 2 had the opposite results with the LL leg plugging first but as discussed in Section 3.4, it may be that the dip legs for Phase 2 testing were in different states of plug formation prior to the start of Phase 2 testing. This phase was a continuation of Phase 1 testing also demonstrated that the solubility limit does not need to be exceeded, only approached for plugging to occur. It appears that the localized evaporation at the interface is a catalyst to the plug formation, starting at the interior surfaces of the dip legs, and slowly bridging across the full diameter. This bridging-type behavior potential was demonstrated by the spindle formations documented in Figure 3-6, Figure 3-7, and Figure 3-8

Phase 3 testing, with the addition of pressure transducers and a thermocouple and the feed solution starting at a condition below the solubility limit, demonstrated plugging in 8 days, again with the Sp. G. leg plugging first, followed by the liquid level reference leg several days later. Once the plugging process could be determined to be occurring through pressure increases, the Sp. G. dip leg became fully plugged in 119 minutes, and the LL dip leg becoming fully plugged in 240 minutes.

Phase 4 testing was set up to accelerate plug formation based on the time required to achieve plugging seen in previous testing. The solution was prepared very close to the solubility limit for the temperature of testing. Consequently, plug formation time was compressed to days (compared to weeks in Phase 1 testing). Phase 4 testing plugging occurred anywhere from ¾ day to 4 days, depending on the specific dip leg.

In this aggressive plugging condition, the Sp. G. legs plugged first irrespective of their respective design, be it original design or new design. Of interest is that while the original design Sp. G. leg plugged in ¾ of a day, the new design liquid level nozzle took 4 days to plug, with a significant 1 ½ day run-up from a clear bubbler to full plugging. The new design appears to show improved behavior with respect to the original design, particularly the new design liquid level nozzle, which had a full 5 ¼ in. length of 1.049 in. nominal clear ID. This compares to the original design of 0.622 in. nominal clear ID. Of particular interest is that the new design Sp. G. reference leg showed plugging at the reduction location from 1.049 in. nominal ID to 0.622 in. nominal ID (see Figure 3-28), with very minor deposition of localized crystals in the expanded section interior diameter. The aluminum nitrate appears to be crystallizing on the surface of the nozzle first, and consequently bridging across the nozzle. The increased flow area appears to play a significant role in giving the crystallizing solids more time to dissolve which should inhibit bridging and eventual plugging. The data suggests that if the dip legs were a full 1.049 in. nominal clear ID for its submerged length that a reduction in plugging over time might be achieved in future HCA operations. See section 5.1 for suggested further testing.

Finally, as discussed in Phase 4 testing results, the HCA Engineering suggested daily purging was not implemented, and should be considered for any future testing as it may have a more significant impact on mitigating plug formation.

5.0 Information and Suggestions for Further Testing

5.1 Additional Testing with Current and New Design

Continuation of the current work would be beneficial to more precisely determine the benefit of larger diameter dip legs, and specifically the decreased frequency of plugging expected. The current HCA Structures, Systems and Components (SSC's) could handle the new design nozzles, although the impact to HCA operations would be expensive to implement. This new testing would include the new nozzle design, with a maximum barrel length achievable. The maximum length is intended to extend the largest diameter dip leg as high as possible to prevent tank solution from entering the smaller diameter tube that feeds air to the bubbler. The new test would also compare the current design with the expanded nozzle design. This testing would also test various molarities of aluminum nitrate and acids to determine at what concentration of aluminum nitrate plugging begins. This information will be beneficial in planning for long term storage of the SNF solution. A suggested test matrix is shown below.

Test	Nozzles	Tank Solutions	Purpose	Air Flow Rates	Temp.
1	New and Original	4-6 solutions of varying Molarities	To provide guidance on molarity limits for HCA operations	7.5 SCFH	25 °C
2	New and Original	1.9 M Al2O3, 1 M HNO3	To ascertain the effect of flow rates on plug dynamics	Various Flow Rates up to 15 SCFH	25 °C

Test 1 would provide information on where incipient plugging occurs, possibly providing HCA Engineering with feedback on process control. Test 2 would determine if plugging is a function of flow rates of purge air, with the belief that higher flowrates might limit plugging forming dynamics.

With this additional testing, a more rigid purge protocol should also be tested to determine the benefit of frequent purging, as frequent purging would require no change in infrastructure for the HCA. Concurrently, testing with a purge protocol would be done to establish single parameter variable studies. The increased flow area of the new nozzles, coupled with the relative ease with which the solids have shown to re-dissolve during periods of testing inactivity, could also be a plug clearing dynamic that can be investigated.

Agitation was not part of this testing. It has been shown in previous studies (reference 3), that mechanical agitators located in the tank may lead to pressure fluctuations on the inlet of the dip leg nozzles, possibly accentuating the plug forming dynamics. If the testing described above provides a successful nozzle design change, additional testing on the new nozzle design should also be repeated with agitation that mimics agitation occurring in the HCA facility.

5.2 Perform Calculations to More Thoroughly Support Localized Evaporation Conclusion

To further our understanding of the dynamics involved in plug formation, it is suggested that the nozzle flow be modeled using M-Star CFD (Computational Fluid Dynamics). The M-Star program, using the physical inputs of geometry, pressure, temperature, and solution density and viscosity, can determine if, under normal operating conditions, liquid can migrate into the interior space of the nozzles during operations. This information would better support the conclusion of local evaporation being the main contributor to plug formation dynamics.

5.3 New Nested Design

As documented in reference 3, maximizing the internal diameter minimizes the tendency to bridge solids due to the larger flow area (See Figure 3-28). It should be noted that reference 3 solution was a simulant of the SRAT and SME vessels at elevated temperatures, and the mechanism of bridging may be different from the crystal formations occurring in the HCA dip legs. The canyon SSC's severely limit major design changes to the dip legs. The maximum opening through which the dip legs must be lowered by crane is 3 in. ID, and because of the nature of crane operations, this was limited to 2 ½ in. As noted in reference 3, a nested design that allowed smaller tubing to provide air to sections of larger OD nozzles provided some relief from plugging. Beginning with 2 ½ in. SCH 10 SS Pipe (2.875 in. OD) as a large diameter sheath (no additional outer piping), a design could be developed with a maximum of 1 1/2 in. SCH 40 pipe nozzles that could be located at all three locations. See Figure 5-1 below.
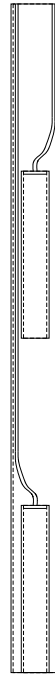
Figure 5-1. Suggested Nested Design Concept.

5.4 Additional Testing with a Porous Metal Nozzle Design

Previous testing[9] has indicated that a 10 micron sintered metal porous tube, purged daily, may provide a solution to plugging. This test would require the nesting of the porous tube, as water or air was constantly supplied in attempts to mitigate plug formation. This test, with the porous tube welded to the bottom of the bubblers, demonstrated promising results when used in conjunction with water as a wetting agent. The infrastructure support for this design would require extensive redesign of the bubblers.

5.5 Additional Testing with Humidified Air

Increasing the relative humidity of the input air could minimize the evaporation effect at the nozzle tips. However, testing results from reference 10 indicates that significant problems were encountered. For example, thermodynamic conditions can be made to saturate the air stream (moist air) or supersaturate the air stream (i.e., mostly steam), but by the time the stream reaches the bubbler exit, the conditions are such that condensation will occur, drying the stream, making it ineffective.

6.0 References

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Appendix A. Alternate Feed Tank

Ø3.0"
THRU HOLE
TOP PIECE ONLY

TOP

1/4" THK

18½"

BOTTOM

Ø12" TYP
Ø11.5" TYP

TYPICAL DETAIL TOP
AND BOTTOM
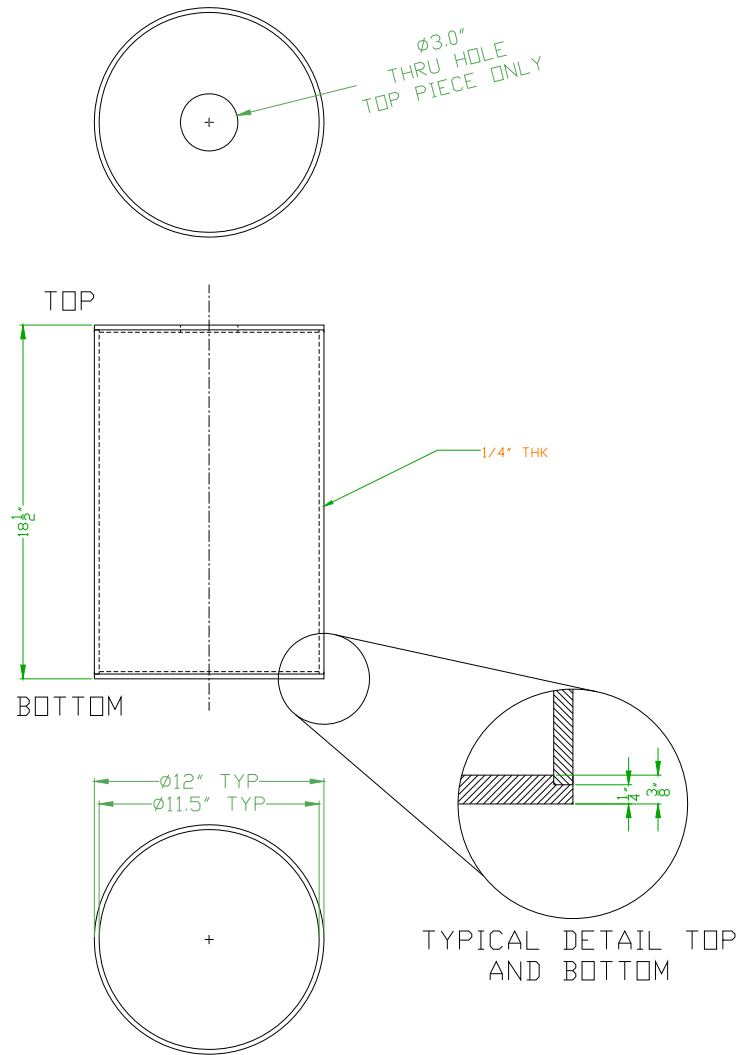
NOTES:

1. BOTTOM PIECE GLUED FOR WATER TIGHTNESS
2. TOP PIECE NEEDS LOOSE FIT FOR EASY REMOVAL

MLR-20200304-1, REV.0 (2020-2-4)
SCALE: 1/4   1 RQ'D
MAT'L: ACRYLIC
H CANYON DIP LEG TANK

Appendix B. Test Solution SRTT15015 Analytical Results

ANALYTE_NAME	RESULT	RESULT_UNITS		ANALYTE_NAME	RESULT	RESULT_UNITS
BARIUM	318	ug/L		1,1-DICHLOROETHYLENE	50	ug/L
BERYLLIUM	238	ug/L		1,1-DICHLOROETHYLENE	50	ug/L
CHROMIUM	121	ug/L		ACETONE	609	ug/L
IRON	8350	ug/L		ACETONE	609	ug/L
MAGNESIUM	26200	ug/L		1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	250	ug/L
NICKEL	161	ug/L		1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	250	ug/L
POTASSIUM	10800	ug/L		IODOMETHANE (METHYL IODIDE)	250	ug/L
TITANIUM	352	ug/L		IODOMETHANE (METHYL IODIDE)	250	ug/L
VANADIUM	146	ug/L		CARBON DISULFIDE	250	ug/L
BROMIDE	200	mg/L		CARBON DISULFIDE	250	ug/L
BROMIDE	200	mg/L		ACETONITRILE (METHYL CYANIDE)	1250	ug/L
FLUORIDE	100	mg/L		ACETONITRILE (METHYL CYANIDE)	1250	ug/L
FLUORIDE	100	mg/L		ISOBUTANOL	2500	ug/L
PH	0.41	pH		ISOBUTANOL	2500	ug/L
PH	0.41	pH		ALLYL CHLORIDE	250	ug/L
CHLORIDE	200	mg/L		ALLYL CHLORIDE	250	ug/L
CHLORIDE	200	mg/L		DICHLOROMETHANE (METHYLENE CHLORIDE)	250	ug/L
NITRATE	90800	mg/L		DICHLOROMETHANE (METHYLENE CHLORIDE)	250	ug/L
NITRATE	90800	mg/L		ACRYLONITRILE	250	ug/L
TOTAL ORGANIC CARBON	200	mg/L		ACRYLONITRILE	250	ug/L
TOTAL ORGANIC CARBON	200	mg/L		METHYL TERTIARY BUTYL ETHER (MTBE)	50	ug/L
TOTAL SUSPENDED SOLIDS	86	mg/L		METHYL TERTIARY BUTYL ETHER (MTBE)	50	ug/L
TOTAL SUSPENDED SOLIDS	86	mg/L		TRANS-1,2-DICHLOROETHYLENE	50	ug/L
TOTAL ORGANIC CARBON	200	mg/L		TRANS-1,2-DICHLOROETHYLENE	50	ug/L
TOTAL ORGANIC CARBON	200	mg/L		1,1-DICHLOROETHANE	50	ug/L
TOTAL ORGANIC CARBON	200	mg/L		1,1-DICHLOROETHANE	50	ug/L
TOTAL ORGANIC CARBON	200	mg/L		VINYL ACETATE	250	ug/L
TOTAL ORGANIC CARBON	200	mg/L		VINYL ACETATE	250	ug/L
TOTAL ORGANIC CARBON	200	mg/L		CHLOROPRENE	50	ug/L
TOTAL ORGANIC CARBON	200	mg/L		CHLOROPRENE	50	ug/L
TOTAL ORGANIC CARBON	200	mg/L		METHYL ETHYL KETONE	250	ug/L
DICHLORODIFLUOROMETHANE	50	ug/L		METHYL ETHYL KETONE	250	ug/L
DICHLORODIFLUOROMETHANE	50	ug/L		CIS-1,2-DICHLOROETHYLENE	50	ug/L
CHLOROMETHANE (METHYL CHLORIDE)	50	ug/L		CIS-1,2-DICHLOROETHYLENE	50	ug/L
CHLOROMETHANE (METHYL CHLORIDE)	50	ug/L		1,2-DICHLOROETHYLENE	100	ug/L
CHLOROETHENE (VINYL CHLORIDE)	50	ug/L		1,2-DICHLOROETHYLENE	100	ug/L
CHLOROETHENE (VINYL CHLORIDE)	50	ug/L		2,2-DICHLOROPROPANE	50	ug/L
BROMOMETHANE (METHYL BROMIDE)	50	ug/L		2,2-DICHLOROPROPANE	50	ug/L
BROMOMETHANE (METHYL BROMIDE)	50	ug/L		METHACRYLONITRILE	250	ug/L
CHLOROETHANE (ETHYL CHLORIDE)	50	ug/L		METHACRYLONITRILE	250	ug/L
CHLOROETHANE (ETHYL CHLORIDE)	50	ug/L		ETHYL ACETATE	250	ug/L
TRICHLOROFLUOROMETHANE	50	ug/L		ETHYL ACETATE	250	ug/L
TRICHLOROFLUOROMETHANE	50	ug/L		PROPIONITRILE	250	ug/L
ACROLEIN	250	ug/L		PROPIONITRILE	250	ug/L
ACROLEIN	250	ug/L		BROMOCHLOROMETHANE	50	ug/L

ANALYTE_NAME	RESULT	RESULT_UNITS		ANALYTE_NAME	RESULT	RESULT_UNITS
BROMOCHLOROMETHANE	50	ug/L		O-XYLENE	50	ug/L
CHLOROFORM	50	ug/L		XYLENES	150	ug/L
CHLOROFORM	50	ug/L		XYLENES	150	ug/L
1,1,1-TRICHLOROETHANE	50	ug/L		STYRENE	50	ug/L
1,1,1-TRICHLOROETHANE	50	ug/L		STYRENE	50	ug/L
1,1-DICHLOROPROPENE	50	ug/L		BROMOFORM (TRIBROMOMETHANE)	50	ug/L
1,1-DICHLOROPROPENE	50	ug/L		BROMOFORM (TRIBROMOMETHANE)	50	ug/L
CARBON TETRACHLORIDE	50	ug/L		CUMENE (ISOPROPYLBENZENE)	50	ug/L
CARBON TETRACHLORIDE	50	ug/L		CUMENE (ISOPROPYLBENZENE)	50	ug/L
1,2-DICHLOROETHANE (EDC)	50	ug/L		1,1,2,2-TETRACHLOROETHANE	50	ug/L
1,2-DICHLOROETHANE (EDC)	50	ug/L		1,1,2,2-TETRACHLOROETHANE	50	ug/L
BENZENE	50	ug/L		TRANS-1,4-DICHLORO-2-BUTENE	250	ug/L
BENZENE	50	ug/L		TRANS-1,4-DICHLORO-2-BUTENE	250	ug/L
TRICHLOROETHYLENE (TCE)	50	ug/L		1,2,3-TRICHLOROPROPANE	50	ug/L
TRICHLOROETHYLENE (TCE)	50	ug/L		1,2,3-TRICHLOROPROPANE	50	ug/L
1,2-DICHLOROPROPANE	50	ug/L		1,2-DIBROMO-3-CHLOROPROPANE	50	ug/L
1,2-DICHLOROPROPANE	50	ug/L		1,2-DIBROMO-3-CHLOROPROPANE	50	ug/L
DIBROMOMETHANE (METHYLENE BROMIDE)	50	ug/L		1,2,3-TRICHLOROBENZENE	50	ug/L
DIBROMOMETHANE (METHYLENE BROMIDE)	50	ug/L		1,2,3-TRICHLOROBENZENE	50	ug/L
BROMODICHLOROMETHANE	50	ug/L		METHYL ACETATE	250	ug/L
BROMODICHLOROMETHANE	50	ug/L		METHYL ACETATE	250	ug/L
2-CHLOROETHYL VINYL ETHER	250	ug/L		CYCLOHEXANE	50	ug/L
2-CHLOROETHYL VINYL ETHER	250	ug/L		CYCLOHEXANE	50	ug/L
CIS-1,3-DICHLOROPROPENE	50	ug/L		METHYLCYCLOHEXANE	50	ug/L
CIS-1,3-DICHLOROPROPENE	50	ug/L		METHYLCYCLOHEXANE	50	ug/L
METHYL ISOBUTYL KETONE	250	ug/L		1,3-DICHLOROBENZENE	50	ug/L
METHYL ISOBUTYL KETONE	250	ug/L		1,3-DICHLOROBENZENE	50	ug/L
TOLUENE	50	ug/L		1,4-DICHLOROBENZENE	50	ug/L
TOLUENE	50	ug/L		1,4-DICHLOROBENZENE	50	ug/L
TRANS-1,3-DICHLOROPROPENE	50	ug/L		1,2-DICHLOROBENZENE	50	ug/L
TRANS-1,3-DICHLOROPROPENE	50	ug/L		1,2-DICHLOROBENZENE	50	ug/L
1,1,2-TRICHLOROETHANE	50	ug/L		1,2,4-TRICHLOROBENZENE	50	ug/L
1,1,2-TRICHLOROETHANE	50	ug/L		1,2,4-TRICHLOROBENZENE	50	ug/L
1,3-DICHLOROPROPANE	50	ug/L		NAPHTHALENE	50	ug/L
1,3-DICHLOROPROPANE	50	ug/L		NAPHTHALENE	50	ug/L
2-HEXANONE	250	ug/L		1,4-DIOXANE	2500	ug/L
2-HEXANONE	250	ug/L		1,4-DIOXANE	2500	ug/L
TETRACHLOROETHYLENE (PCE)	50	ug/L		METHYL METHACRYLATE	250	ug/L
TETRACHLOROETHYLENE (PCE)	50	ug/L		METHYL METHACRYLATE	250	ug/L
DIBROMOCHLOROMETHANE	50	ug/L		CAPROLACTAM	4500000	ug/kg
DIBROMOCHLOROMETHANE	50	ug/L		P-NITROANILINE	4500000	ug/kg
1,2-DIBROMOETHANE	50	ug/L		PHENACETIN	4500000	ug/kg
1,2-DIBROMOETHANE	50	ug/L		BIS(2-CHLORO-1-METHYLETHYL)ETHER	4500000	ug/kg
CHLOROBENZENE	50	ug/L		BIS(2-ETHYLHEXYL)PHTHALATE (DEHP)	450000	ug/kg
CHLOROBENZENE	50	ug/L		BORON	5000	ug/L
1,1,1,2-TETRACHLOROETHANE	50	ug/L		METHYL METHANESULFONATE	4500000	ug/kg
1,1,1,2-TETRACHLOROETHANE	50	ug/L		ARSENIC	495	ug/L
ETHYLBENZENE	50	ug/L		SELENIUM	3000	ug/L
ETHYLBENZENE	50	ug/L		N-NITROSO-N-METHYLETHYLAMINE	4500000	ug/kg
M,P-XYLENE	100	ug/L		2,3,4,6-TETRACHLOROPHENOL	4500000	ug/kg
M,P-XYLENE	100	ug/L		HEXACHLOROETHANE	4500000	ug/kg
O-XYLENE	50	ug/L		BIS(2-CHLOROETHOXY)METHANE	4500000	ug/kg

ANALYTE_NAME	RESULT	RESULT_UNITS		ANALYTE_NAME	RESULT	RESULT_UNITS
DIBENZOFURAN	4500000	ug/kg		DIETHYL PHTHALATE	4500000	ug/kg
N-NITROSODIETHYLAMINE	4500000	ug/kg		ACETOPHENONE	4500000	ug/kg
MOLYBDENUM	1000	ug/L		1,2,4,5-TETRACHLOROBENZENE	4500000	ug/kg
2,4,5-TRICHLOROPHENOL	4500000	ug/kg		NITROBENZENE	4500000	ug/kg
2,4-DINITROTOLUENE	4500000	ug/kg		O,O,O-TRIETHYL PHOSPHOROTHIOATE	4500000	ug/kg
CHLOROBENZILATE	4500000	ug/kg		LEAD	10000	ug/L
KARL FISCHER MOISTURE	34.1	%		HEXACHLOROPROPENE	4500000	ug/kg
2-CHLOROPHENOL	4500000	ug/kg		2-ACETYLAMINOFLUORENE	4500000	ug/kg
ISOSAFROLE	4500000	ug/kg		BENZO[A]PYRENE	450000	ug/kg
1,4-DICHLOROBENZENE	4500000	ug/kg		ISOPHORONE	4500000	ug/kg
ATRAZINE	4500000	ug/kg		ALUMINUM	49700000	ug/L
IGNITABILITY	200	degF		O-CRESOL (2-METHYLPHENOL)	4500000	ug/kg
P-PHENYLENEDIAMINE	225000000	ug/kg		FAMPHUR	4500000	ug/kg
1,4-NAPHTHOQUINONE	4500000	ug/kg		FLUORANTHENE	450000	ug/kg
ISODRIN	4500000	ug/kg		PENTACHLOROETHANE	4500000	ug/kg
1,2-DICHLOROBENZENE	4500000	ug/kg		ETHYL METHANESULFONATE	4500000	ug/kg
ANTIMONY	2000	ug/L		LEAD	4000	ug/L
SULFOTEPP	4500000	ug/kg		CARBAZOLE	450000	ug/kg
2-CHLORONAPHTHALENE	450000	ug/kg		N-NITROSODIPROPYLAMINE	4500000	ug/kg
KEPONE	4500000	ug/kg		DIALLATE	4500000	ug/kg
SILICA	23700	ug/L		PRONAMIDE	4500000	ug/kg
HEXACHLOROBENZENE	4500000	ug/kg		URANIUM	5000	ug/L
DIMETHYL PHTHALATE	4500000	ug/kg		M-NITROANILINE	4500000	ug/kg
N-NITROSODI-N-BUTYLAMINE	4500000	ug/kg		TIN	5000	ug/L
3,3'-DIMETHYLBENZIDINE	4500000	ug/kg		BARIUM	314	ug/L
2,4,6-TRICHLOROPHENOL	4500000	ug/kg		BENZYL ALCOHOL	4500000	ug/kg
DIMETHOATE	4500000	ug/kg		P-CHLORO-M-CRESOL	4500000	ug/kg
HEXACHLOROPHENE	225000000	ug/kg		ACENAPHTHYLENE	450000	ug/kg
CORROSIVITY	355	mm/yr		CADMIUM	500	ug/L
BIS(2-CHLOROETHYL)ETHER	4500000	ug/kg		4-AMINOBIPHENYL	4500000	ug/kg
4-NITROPHENOL	4500000	ug/kg		2,4-DINITROPHENOL	9010000	ug/kg
ACENAPHTHENE	450000	ug/kg		2-NAPHTHYLAMINE	4500000	ug/kg
DIBENZ[AH]ANTHRACENE	450000	ug/kg		PENTACHLOROBENZENE	4500000	ug/kg
HEXACHLOROBUTADIENE	4500000	ug/kg		BENZO[B]FLUORANTHENE	450000	ug/kg
2-NITROANILINE	4500000	ug/kg		CADMIUM	500	ug/L
PHENANTHRENE	450000	ug/kg		THIONAZIN	4500000	ug/kg
BENZO(G,H,I)PERYLENE	450000	ug/kg		THALLIUM	2000	ug/L
2,4-DICHLOROPHENOL	4500000	ug/kg		INDENO[1,2,3-CD]PYRENE	450000	ug/kg
1-NAPHTHYLAMINE	4500000	ug/kg		COPPER	2000	ug/L
METHYL PARATHION	4500000	ug/kg		ARSENIC	3000	ug/L
2-METHYLANILINE (O-TOLUIDINE)	4500000	ug/kg		BUTYL BENZYL PHTHALATE	4500000	ug/kg
1,3,5-TRINITROBENZENE	4500000	ug/kg		4-BROMOPHENYL PHENYL ETHER	4500000	ug/kg
DI-N-BUTYL PHTHALATE	4500000	ug/kg		CHRYSENE	450000	ug/kg
M/P-CRESOL	4500000	ug/kg		SODIUM	289000	ug/L
2-PICOLINE	4500000	ug/kg		1,2-DIPHENYLHYDRAZINE	4500000	ug/kg
5-NITRO-O-TOLUIDINE	4500000	ug/kg		PENTACHLORONITROBENZENE	4500000	ug/kg
DISULFOTON	4500000	ug/kg		PHENOL	4500000	ug/kg
4-CHLOROANILINE	4500000	ug/kg		ANILINE	4500000	ug/kg
N-NITROSOPYRROLIDINE	4500000	ug/kg		FLUORENE	450000	ug/kg
PYRIDINE	4500000	ug/kg		STRONTIUM	2500	ug/L
HEXACHLOROCYCLOPENTADIENE	4500000	ug/kg		2,6-DICHLOROPHENOL	4500000	ug/kg
DINITRO-O-CRESOL	4500000	ug/kg		N-NITROSODIMETHYLAMINE	4500000	ug/kg
1,3-DINITROBENZENE	4500000	ug/kg		1,2,4-TRICHLOROBENZENE	4500000	ug/kg
ARAMITE	4500000	ug/kg		3,3-DICHLOROBENZIDINE	4500000	ug/kg
COBALT	500	ug/L		4-CHLOROPHENYL PHENYL ETHER	4500000	ug/kg
SILVER	500	ug/L		N-NITROSODIPHENYLAMINE+DIPHENYLAMINE	4500000	ug/kg

ANALYTE_NAME	RESULT	RESULT_UNITS
A,A-DIMETHYLPHENETHYLAMINE	4500000	ug/kg
NAPHTHALENE	450000	ug/kg
PHORATE	4500000	ug/kg
3-METHYLCHOLANTHRENE	4500000	ug/kg
2-NITROPHENOL	4500000	ug/kg
2,6-DINITROTOLUENE	4500000	ug/kg
N-NITROSOPIPERIDINE	4500000	ug/kg
1,1'-BIPHENYL	4500000	ug/kg
PENTACHLOROPHENOL	4500000	ug/kg
CHROMIUM	170	ug/L
N-NITROSOMORPHOLINE	4500000	ug/kg
BENZO[A]ANTHRACENE	450000	ug/kg
MERCURY	2	ug/L
P-DIMETHYLAMINOAZOBENZENE	4500000	ug/kg
4-NITROQUINOLINE-1-OXIDE	4500000	ug/kg
VISCOSITY, KINEMATIC	10	cSt
BENZIDINE	4500000	ug/kg
N-DIOCTYL PHTHALATE	4500000	ug/kg
ETHYL METHACRYLATE	4500000	ug/kg
SILVER	50	ug/L
CALCIUM	414000	ug/L
PYRENE	450000	ug/kg
7,12-DIMETHYLBENZ(A)ANTHRACENE	4500000	ug/kg
BENZOIC ACID	9010000	ug/kg
ZINC	13200	ug/L
SILICON	11200	ug/L
ANTHRACENE	450000	ug/kg
BENZALDEHYDE	4500000	ug/kg
SELENIUM	15000	ug/L
MANGANESE	1340	ug/L
2,4-DIMETHYLPHENOL	4500000	ug/kg
SAFROLE	4500000	ug/kg
SPECIFIC GRAVITY	1.37	none
1,3-DICHLOROBENZENE	4500000	ug/kg
2-METHYLNAPHTHALENE	450000	ug/kg
1,4-DIOXANE	4500000	ug/kg
PARATHION	4500000	ug/kg
BENZO[K]FLUORANTHENE	450000	ug/kg
METHAPYRILENE	4500000	ug/kg

Appendix C. Phase 1 Testing, R&D Directions, 05/27/2020

R&D Directions: Chemical Mixing and Loading the Dip Leg Pluggage Rig, 786-A
PI: Michael Restivo
Date: 05/27/2020

Reference Documents: 786-A Shop Activities, SRNL-HA-01255
Material Handling in 786-A, SRNL-HA-00773
Assembly and Disassembly of R&D Test Apparatus, SRNL-HA-00779
Routine Chemical Handling, 786-A, SRNL-HA-00750

JOB HAS- SRNL-HA-01385

General Hazards

- If a spill occurs that is not contained immediately invoke SWIMS[1], inform the control room (5-3401) and then management.
- Before the test begins verify that the ventilation hoods to be used have up to date inspections, are in operation (perceived by the movement of air streamers), and that the hood sash is located the proper heights, as indicated by the arrows on both sides of the sash.

Step	Hazard	Control
Mixing Chemicals in hood	Corrosive Chemicals	Wear Lab Coat, safety glasses, thin mil nitrile gloves. Hood Sash below chin level. All spills to be handled per IH instructions. Non-combustible and compatible wipes are to be used.
Working with HS-30 Silica	Carcinogen	Supplied as colloidal liquid, which is the control.
Working with 70 wt% Nitric Acid	Corrosive Chemical	Handling requires safety goggles and face shield or hood sash below chin level if in hood, chemical apron, and nitrile gloves. Safety shoes are required when transporting concentrated acid.
Operating Rig (Bubbling in tank)	Physical Splash Hazard	Ensure tank has lid or has sufficient freeboard to accommodate splashing.

Directions

1. Tare out one clean 10 L carboy.
2. Add 10 liters (13.2 kg) of $Al(NO_3)_3$ from PI identified feed carboy. (ρ=1.32 kg/L)
3. Add 380 mL of 70 wt% Nitric Acid (slowly, as heat will be generated) to carboy now containing 10 liters of $Al(NO_3)_3$. Wear PPE as indicated in table.
4. Add 7.2 mL of HS-30 to the carboy. Wear PPE as indicated in table.
5. Mix contents.
6. Pour liquid into Dip Leg Tank. Wear PPE as indicated in table.
7. Label container.
8. Mark level.
9. Slowly add air to both rotameters as directed by the PI.
10. Daily
    a. Add DI Water to return feed tank to fill line.
    b. Write down Pressures and Flow Rates in supplied log book
    c. Confirm Control Room has overnight sheet.

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[1] SWIMS = Stop, Warn, Isolate, Minimize, and Secure

Appendix D. Phase 2 Testing, R&D Directions, 2020-6-29

R&D Directions: Removal of Dip Leg and Sampling of Plugged Solids on the Dip Leg Pluggage Rig, 786-A

PI: Michael Restivo

Date: 06/29/2020

Reference Documents: 786-A Shop Activities, SRNL-HA-01255

Material Handling in 786-A, SRNL-HA-00773

Assembly and Disassembly of R&D Test Apparatus, SRNL-HA-00779

Routine Chemical Handling, 786-A, SRNL-HA-00750

JOB HAS- SRNL-HA-01385

General Hazards

- If a spill occurs that is not contained immediately invoke SWIMS[2], inform the control room (5-3401) and then management.

Step	Hazard	Control
Pump Simulant from Bubbler Tank	Corrosive Chemicals	Wear Lab Coat, safety glasses, thin mil nitrile gloves. All spills to be handled per IH instructions. Non-combustible and compatible wipes are to be used.
Lift out Bubbler Tube from Tank	Pinch points. Heavy object.	Wear leather gloves as required with hand tools. Safety shoes while lifting bubbler. Non-combustible and compatible wipes are to be used.
Sample Liquid	Corrosive Chemical	Wear Lab Coat, safety glasses, thin mil nitrile gloves. All spills to be handled per IH instructions. Non-combustible and compatible wipes are to be used.
Sample Solids	Physical Splash Hazard	Wear Lab Coat, safety glasses, thin mil nitrile gloves.
Install Bubbler Tube in Tank	Pinch points. Heavy Object.	Wear leather gloves as required with hand tools. Safety shoes while lifting bubbler.
Add simulant to Bubbler Tank.	Corrosive Chemicals	Wear Lab Coat, safety glasses, thin mil nitrile gloves. All spills to be handled per IH instructions. Non-combustible and compatible wipes are to be used.

Directions

11. Perform Pre-job briefing.
12. Stop Air flow.
13. Using pump, empty contents of Bubbler Tank into empty carboy.
14. Sample contents of carboy and be sure to label carboy and sample bottle appropriately.
15. Disconnect supply air lines from both bubbler tubes. Loosely bag the bottom tubes (1 bag) and each pressure inlet to prevent inappropriate liquid discharges.
16. Loosen clamps holding Bubbler Tubes in tank, and lift Bubbler Tubes from Tank, wiping tubes with compatible wipes while lifting. (Two-person job)
17. Place bubbler Tubes on flat surface and take photos per P.I. direction.
18. Install Bubbler Tube back in tank, making all connections.
19. If the same simulant will be used for additional testing, add simulant to tank. If not, add simulant as requested by P.I.[3]

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[2] SWIMS = Stop, Warn, Isolate, Minimize, and Secure

[3] Support personnel were trained on storing, retrieving, and downloading data by the P.I.

Appendix E. Phase 3 Testing, R&D Directions, 2020-7-20

R&D Directions: Chemical Mixing and Loading the Dip Leg Pluggage Rig (Addition), 786-A

PI: Michael Restivo

Date: 07-20-2020

Reference Documents: 786-A Shop Activities, SRNL-HA-01255

Material Handling in 786-A, SRNL-HA-00773

Assembly and Disassembly of R&D Test Apparatus, SRNL-HA-00779

Routine Chemical Handling, 786-A, SRNL-HA-00750

JOB HAS- SRNL-HA-01385

General Hazards

- If a spill occurs that is not contained immediately invoke SWIMS[3], inform the control room (5-3401) and then management.
- Before the test begins verify that the ventilation hoods to be used have up to date inspections, are in operation (perceived by the movement of air streamers), and that the hood sash is located the proper heights, as indicated by the arrows on both sides of the sash.

Step	Hazard	Control
Mixing Chemicals in hood	Corrosive Chemicals	Wear Lab Coat, safety glasses, thin mil nitrile gloves. Hood Sash below chin level. All spills to be handled per IH instructions. Non-combustible and compatible wipes are to be used.
Working with HS-30 Silica	Carcinogen	Supplied as colloidal liquid, which is the control.
Working with 70 wt% Nitric Acid	Corrosive Chemical	Handling requires safety goggles and face shield or hood sash below chin level if in hood, chemical apron, and nitrile gloves. Safety shoes are required when transporting concentrated acid.
Operating Rig (Bubbling in tank)	Physical Splash Hazard	Ensure tank has lid or has sufficient freeboard to accommodate splashing.

Directions

20. Tare out one clean 1 L container.
21. Add <u>1 liters (1.3 kg)</u> of $Al(NO_3)_3$ from PI identified feed carboy. (ρ=1.32 kg/L) (Actual mass=_____)
22. Add <u>38</u> mL of 70 wt% Nitric Acid (slowly, as heat will be generated) to container now containing 1 liter of $Al(NO_3)_3$. Wear PPE as indicated in table. (Actual Volume=_____)
23. Add <u>0.72 mL</u> of HS-30 to the carboy. Wear PPE as indicated in table. (actual volume=_____)
24. Mix contents.
25. Pour liquid into Dip Leg Tank. Wear PPE as indicated in table.
26. Label container.
27. Mark level.
28. Slowly add air to both rotameters as directed by the PI.
29. Daily
    a. Add DI Water to return feed tank to fill line.
    b. Write down Pressures and Flow Rates in supplied log book[4]
    c. Confirm Control Room has overnight sheet.

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[3] SWIMS = Stop, Warn, Isolate, Minimize, and Secure

[4] Although the data was recorded by the computer, technicians took periodic readings for familiarity of rig status

Appendix F. Phase 4 Testing R&D Directions, 2020-9-8

R&D Directions: Chemical Mixing and Loading the Dip Leg Pluggage Rig (Addition), 786-A

PI: Michael Restivo

Date: 09-08-2020

Reference Documents: 786-A Shop Activities, SRNL-HA-01255

Material Handling in 786-A, SRNL-HA-00773

Assembly and Disassembly of R&D Test Apparatus, SRNL-HA-00779

Routine Chemical Handling, 786-A, SRNL-HA-00750

JOB HAS- SRNL-HA-01385

General Hazards

- If a spill occurs that is not contained immediately invoke SWIMS[4], inform the control room (5-3401) and then management.
- Before the test begins verify that the ventilation hoods to be used have up to date inspections, are in operation (perceived by the movement of air streamers), and that the hood sash is located the proper heights, as indicated by the arrows on both sides of the sash.

Step	Hazard	Control
Mixing Chemicals in hood	Corrosive Chemicals	Wear Lab Coat, safety glasses, thin mil nitrile gloves. Hood Sash below chin level. All spills to be handled per IH instructions. Non-combustible and compatible wipes are to be used.
Working with HS-30 Silica	Carcinogen	Supplied as colloidal liquid, which is the control.
Working with 70 wt% Nitric Acid	Corrosive Chemical	Handling requires safety goggles and face shield or hood sash below chin level if in hood, chemical apron, and nitrile gloves. Safety shoes are required when transporting concentrated acid.
Operating Rig (Bubbling in tank)	Physical Splash Hazard	Ensure tank has lid or has sufficient freeboard to accommodate splashing.

Directions

30. Tare out one clean 50 L container.
31. Add 30 liters (30L*1.32kg/L=**39.6 kg**) of $Al(NO_3)_3$ from PI identified feed container. (ρ=1.32 kg/L) (**Actual mass**=_____)
32. Add 2,250 mL (2.25 L) of 70 wt% Nitric Acid (slowly, as heat will be generated) to container now containing 30 liter of $Al(NO_3)_3$. Wear PPE as indicated in table. (**Actual Volume Added**=_____). Total volume should now be 32.25 L.
33. Add 3.33*0.25g/L*32.25 L=26.8 g of HS-30 (30 wt% Silica) to the carboy. Wear PPE as indicated in table. (**Actual mass added**=_____)
34. Add 4,234 g Al(NO3)3-9H20 from source identified by PI. (**Actual Mass Added**=_____)
35. Mix contents.
36. Pour liquid into Dip Leg Tank. Wear PPE as indicated in table.
37. Label container.
38. Mark level.
39. Slowly add air to both rotameters as directed by the PI.
40. Daily
    a. Add DI Water to return feed tank to fill line.
    b. Write down Pressures and Flow Rates in supplied log book
    c. Confirm Control Room has overnight sheet.

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[4] SWIMS = Stop, Warn, Isolate, Minimize, and Secure