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Savannah River National Laboratory®

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Characterization of Tank 22 Demister Solids Responsible for Preventing Adequate Purge Ventilation Flow (U)

L. N. Oji

May 2021

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SAVANNAH RIVER NATIONAL LABORATORY

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LIST OF REVISIONS		
Revision Number	**Summary of Changes**	**Date**
0	Initial Issue	May 2021

EXECUTIVE SUMMARY

In December 2020, about 2.5 grams of the solid material from the Tank 22 purge ventilation demister, responsible for preventing adequate purge ventilation flow, were delivered to Savannah River National Laboratory (SRNL) by Savannah River Remediation (SRR) for analysis to identify the chemical nature of the solids and possibly address why fresh water and caustic flushes with 50 wt. % sodium hydroxide solution are essentially ineffective in dissolving or dislodging the accumulated solids from the Tank 22 demister at low ambient temperature conditions.

Analyses of the "as-received" Tank 22 demister solid material were performed using inductively coupled plasma atomic emission (ICP-AES) spectroscopy, scanning electron microscopy (SEM/EDX) and X-ray diffraction (XRD) techniques. The analysis results led to the following conclusions on the mineralogy of the Tank 22 demister solids.

- X-ray diffraction analysis of the "as-received" Tank 22 demister solid sample indicate the presence of amorphous material and a crystalline mercury compound called Mosesite. Mosesite has different chemical formulae depending on the nature of the anion species present in the medium [(Hg_2NCl*H_2O or $(Hg_2N)(Cl,SO_4,MoO_4)*H_2O$ or $Hg_2N(Cl,SO_4,MoO_4,CO_3)\bullet H_2O$)].

- The analytical variations and large uncertainties in the analytical measurement results for Fe [50.300 mg/g solid (52.20 %RSD)], Cr [7.950 mg/g solid (69.65 %RSD)], Ni [3.75 mg/g solid (67.08 %RSD)], Mn [0.847 mg/g solids (58.42 %RSD)], Cu [0.209 mg/g solid (72.03 %RSD)], Zn [0.174 mg/g solid (30.49 %RSD)], Mo [0.123 mg/g solid (66.84 %RSD)], Ba [0.11 mg/g solid (40.84 %RSD)], and)], Co [0.063 mg/g solids (72.23%RSD] reflect the inhomogeneous nature of the "as-received" Tank 22 demister solid sample.

- Other measurable elemental constituents of the Tank 22 demister solids include Hg [563.920 mg/g solid (12.54%RSD)], S [1.320 mg/g solid (2.00 %RSD)], Al [1.24 mg/g solid (18.53 %RSD)], Si [0.905 mg/g solid (17.78 %RSD)], Ca [0.114 mg/g solid (24.80 %RSD)], and Mg [0.086 mg/g solid (14.09 %RSD)]. Thus, mercury alone constitutes about 56.4 percent, by weight, of the Tank 22 demister solids.

- Most of the other compounds identified in the "as-received" Tank 22 Demister solid sample are mainly transition metal amalgams with mercury, which forms the bulk of the amorphous phases of the demister solids.

A literature review on Mosesite chemistry indicates that all forms of the mineral are only soluble in hydrochloric acid. However, since this heterogeneous "as-received" solid sample from the Tank 22 demister is partially amorphous, it is recommended to initially leach or dislodge this material from the Tank 22 demister with pressurized water and appropriate sodium hydroxide solution at greater than room temperature (45-65 degree Centigrade preferred because a temperature of about 20 ^oC, currently in use, is not effective) as the solids accumulates over time in the demister.

The Technical Assistance Request[1] (TAR) for this task also called for the evaluation of other Tank Farm compatible dissolution solutions, mainly inhibited water and 30 -50 wt.% sodium

hydroxide solutions, which could be used to solubilize and dissolve the Tank 22 demister solids. However, because of the availability of a limited quantity of the Tank 22 demister solids, these evaluations were not performed at this time. Therefore, recommended future work to prevent the fouling of the demister by these solid materials would include dissolution studies in 30-50% wt. % sodium hydroxide solutions and inhibited water at above ambient temperature conditions. It is also recommended that a complete water leaching of the demister solids be performed at above ambient temperature conditions and the resulting solutions analyzed for soluble / leachable anion components. This information will be needed to elucidate the formation mechanism for Mosesite in the demister as well as identify the version of this crystalline mercury compound present in the Tank 22 demister solids.

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LIST OF ABBREVIATIONS

ARD	Analytical Research and Development
EMES	Environmental, Materials and Energy Sciences
ICP-AES	inductively coupled plasma atomic emission spectroscopy
%RSD	percent relative standard deviation
SEM/EDX	scanning electron microscope/X-ray diffraction
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
St.Dev.	standard deviation
TTQAP	Technical Task and Quality Assurance Plan
TAR	Technical Assistance Request
XRD	X-ray diffraction

Acknowledgements

The authors extend thanks to several members of the EMES Research Support, Shielded Cells Operations Research and Analytical Research and Development programs who assembled the test equipment, performed the experiments and provided analytical results; specifically, Scott F. McDonald, Julie Fawbush, Taylor Rush, Nathan Wyeth, Catherine Housley, Henry Ajo and Charles Coleman.

1.0 Introduction

In December 2020, about 2.5 grams of the solid material from the Tank 22 purge ventilation demister, responsible for preventing adequate purge ventilation flow, was delivered to SRNL by SRR for analysis to identify the chemical nature (elemental make-up and crystalline structure of the solid) of the solids. Information was also requested to determine how the accumulation of the solid material can be chemically or mechanically removed from the Tank 22 demister to eliminate future demister fouling since fresh water and caustic flushes with 50 wt. % sodium hydroxide solution are essentially ineffective in dissolving and dislodging the solids from the Tank 22 demister.

The Technical Assistance Request[1] defined the tasks and requirements for the performance of this sample characterization by SRNL. This report contains the results of the chemical characterization (ICP-AES) and spectroscopic analyses (XRD and SEM/EDX) of the nature of the solid material from the Tank 22 demister solids responsible for preventing adequate purge ventilation flow through the demister.

1.1 Sample Description

Pictures of the "as-received" Tank 22 demister solids material and the demister component wire gauze into which the solids were embedded are shown in Figure 1, inserts A and B, respectively. The solid material, seen in Figure 1, insert A, is gray in color and not white as the picture suggests. This color distortion is due to the yellow color influence of the SRNL leaded Shielded Cell window. Some of the embedded solid fines are still visible in the wire gauze of the demister, as shown in Figure 1, insert B.

Insert A: Tank 22 purge exhaust demister solids (2.5 g powder sample)

Insert B: Tank 22 purge exhaust demister wire gauze; 19.7 grams

Figure 1, Inserts A and B, show the Tank 22 purge exhaust demister solids and the demister exhaust wire gauze into which the solids are embedded during operation, respectively.

2.0 Experimental Details

Inserts in Figure 1 shows images of the "as-received" gray granular sample and the demister wire gauze component in which the solids are normally imbedded.

One portion (0.25 grams each) of the "as received" solid sample was submitted for solid state analysis by X-ray powder diffraction (XRD) to determine the crystallographic solid phase and the second portion for SEM analysis as shown in Figures 2 and 3, respectively. Three aliquots of the solid sample (0.5 ± 0.05 g each) were also digested in triplicate using aqua-regia with nitric/hydrochloric acid and the resulting digested liquid sample was analyzed in triplicate by Inductively Coupled Plasma–Atomic Emission Spectroscopy (ICP-AES) for elemental constituents and total mercury. The approximately 0.5 grams of solid material remaining was considered insufficient for other follow up leaching and dissolution tasks. To minimize changes in the structural morphology of the solids, no attempt was made during sample preparations to grind the sample to a uniform matrix before digestion and spectroscopic analyses.

3.0 Analytical Results and Discussion

3.1 Aqua Regia Digestion and Analysis for Elemental Constituents

As presented in Figures 2 and 3, the major SEM/EDX elemental peaks (Al, Hg, Fe, Cr, Si, Ni) match the ICP-AES peaks identified in aqua regia digestion analysis for elemental constituents; with mercury being the predominant metal constituent of the demister solids at over 56% Hg by weight per gram of the demister solid.

Elemental mercury (5.64E+02 ± 7.07E+01 mg/g demister solid) formed the bulk of the elemental constituent of the "as-received" sample solid. Other post-digestion measurable elements included iron (5.03E+01 ± 2.63E+01 mg/g demister solid), chromium (7.95E+00 ± 5.54E+00 mg/g demister solid), nickel (3.75E+00 ± 2.51E+00 mg/g demister solid), sulfur (1.32E+00 ± 2.65E-02 mg/g demister solid), aluminum (1.24E+00 ± 2.30E-01 mg/g demister solid), silicon (9.05E-01 ± 1.61E-01 mg/g demister solid), and manganese (8.49E-01 ± 4.96E-01 mg/g demister solid) as shown in Table 1.

The analytical variations and large uncertainties in the analytical measurement results for Ba (36.36 percent relative standard (%RSD)), Cu (71.77 %RSD), Co (73.02 %RSD), Fe (52 %RSD), Cr (69.65 %RSD), Ni (67.08 %RSD), Mn (58.42 %RSD), Mo (66.67 %RSD) and Zn (30.46 %RSD) reflect the inhomogeneous nature of the "as-received" Tank 22 demister sample. The analytical %RSD for most of the elementals are very large because these elements are not uniformly distributed in the solids sample. The one sigma measurement uncertainty for ICP-ES analytical results are 10%.

3.2 XRD Spectra and SEM/EDX

The XRD pattern of the Tank 22 purge exhaust demister solids, as shown in Figure 2, matches that of a hydrated mercury mineral called Mosesite (Hg_2NCl*H_2O or $(Hg_2N)(Cl,SO_4,MoO_4)\cdot H_2O$ or $Hg_2N(Cl,SO_4,MoO_4,CO_3)\bullet H_2O$). Depending on the identity of the anion moieties present in the original solution or liquid matrix from which the demister solids was precipitated, the formula for Mosesite can be chemically represented in the three different ways shown.

This Tank 22 purge exhaust demister solids are made up of partially crystalline phase (Mosesite mineral) and an amorphous phase. The XRD spectra for the powdered demister solids sample shows a shift in the X-ray spectra base line (elevated baseline), which indicates the presence of amorphous materials in addition to the main hydrated crystalline mercury compound (Mosesite).

The principal quantitative elemental constituents, other than mercury, present in the Tank 22 demister solids include iron, aluminum, nickel, sulfur, manganese, and chromium (Table 1). The transition metal mercury amalgams based on some of these elements (Al, Ni, S, Mn and Cr) may constitute the bulk of the amorphous phase material seen in the X-ray spectra. It is worth noting that mercury does not form amalgams with iron. So, the measurable quantity of iron in the solids phase implies that the iron may be associated with the other non- crystalline phases of the Tank 22 demister solids.

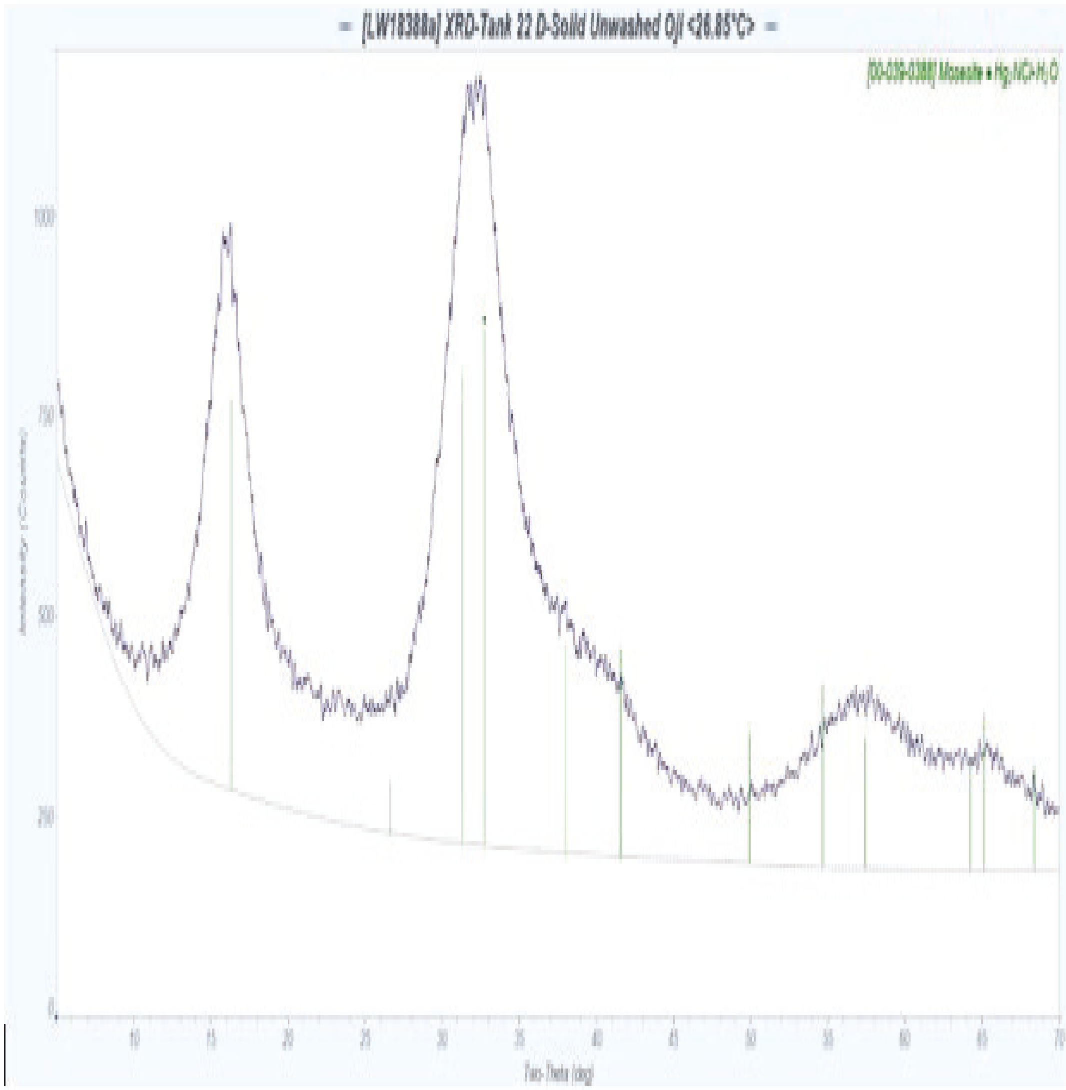

Figure 2. Tank 22 demister solids XRD Spectra

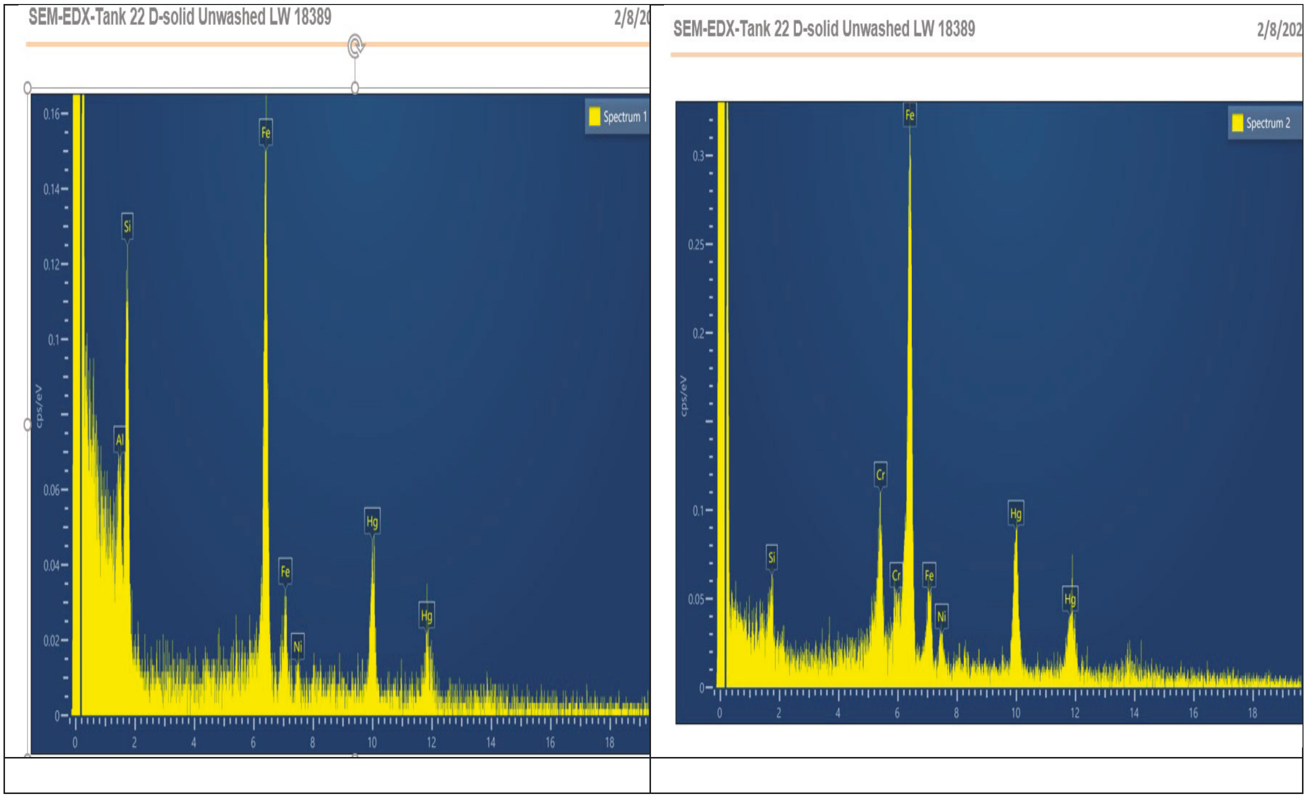

Figure 3. Tank 22 demister solids SEM/EDX [Major SEM/EDX elemental peaks (Al, Hg, Fe, Cr, Si, Ni) observed match the ICP-AES elemental constituents]

Table 1. Tank 22 demister solids elemental composition [LW18390, LW18391, LW18392]

Element	Analysis-1 mg/g solid	Analysis-2 mg/g solid	Analysis-3 mg/g solid	Average mg/g solid	1 Sigma std. deviation	% RSD	Mass % /g solid
Ag	<2.76E-03	<4.30E-03	<4.61E-03	<3.89E-03			
Al	1.29E+00	1.44E+00	9.89E-01	1.24E+00	2.30E-01	18.5	0.12
B	<1.55E-01	<2.42E-01	<2.60E-01	<2.19E-01			
Ba	8.11E-03	1.61E-02	8.63E-03	1.09E-02	4.47E-03	40.8	0.00
Be	<8.37E-04	<1.30E-03	<1.40E-03	<1.18E-03			
Ca	9.72E-02	9.73E-02	1.46E-01	1.14E-01	2.81E-02	24.8	0.01
Cd	<5.35E-03	<8.33E-03	<8.94E-03	<7.54E-03			
Ce	<6.50E-03	<1.00E-02	<1.09E-02	<9.13E-03			
Co	7.54E-02	1.02E-01	1.28E-02	6.34E-02	4.58E-02	72.2	0.01
Cr	8.98E+00	1.29E+01	1.97E+00	7.95E+00	5.54E+00	69.7	0.80
Cu	2.54E-01	3.31E-01	4.09E-02	2.09E-01	1.50E-01	72.0	0.02
Fe	5.90E+01	7.11E+01	2.08E+01	5.03E+01	2.63E+01	52.2	5.03
Gd	<3.94E-03	<6.12E-03	<6.58E-03	<5.55E-03			
K	<1.35E-01	<2.11E-01	<2.26E-01	<1.91E-01			
La	<1.94E-03	<3.02E-03	<3.25E-03	<2.74E-03			
Li	<1.47E-02	<2.29E-02	<2.46E-02	<2.07E-02			
Mg	7.97E-02	7.78E-02	9.96E-02	8.57E-02	1.21E-02	14.1	0.01
Mn	9.59E-01	1.28E+00	3.07E-01	8.49E-01	4.96E-01	58.4	0.08
Mo	1.23E-01	2.05E-01	4.07E-02	1.23E-01	8.22E-02	66.8	0.01
Na	<8.92E-02	<1.39E-01	<1.49E-01	<1.26E-01			
Ni	4.25E+00	5.97E+00	1.02E+00	3.75E+00	2.51E+00	67.1	0.37
P	<2.92E-01	<4.55E-01	<4.88E-01	<4.12E-01			
Pb	<4.88E-02	<7.60E-02	<8.16E-02	<6.88E-02			
S	1.34E+00	1.33E+00	1.29E+00	1.32E+00	2.65E-02	2.0	0.13
Sb	<6.34E-02	<9.86E-02	<1.06E-01	<8.93E-02			
Si	7.95E-01	1.09E+00	8.31E-01	9.05E-01	1.61E-01	17.8	0.09
Sn	<4.51E-02	<7.02E-02	<7.54E-02	<6.36E-02			
Sr	<9.74E-04	<1.51E-03	<1.63E-03	<1.37E-03			
Th	<4.24E-02	<6.59E-02	<7.08E-02	<5.97E-02			
Ti	<9.93E-03	<1.55E-02	<1.66E-02	<1.40E-02			
U	<6.62E-02	<1.03E-01	<1.11E-01	<9.34E-02			
V	<2.09E-02	<3.26E-02	<3.50E-02	<2.95E-02			
Zn	2.03E-01	2.07E-01	1.13E-01	1.74E-01	5.32E-02	30.5	0.02
Zr	<1.55E-03	<2.42E-03	<2.60E-03	<2.19E-03			
Hg, $_{tot}$	6.30E+02	5.73E+02	4.89E+02	5.64E+02	7.07E+01	12.5	56.39

4.0 Conclusions

In December 2020, about 2.5 grams of the solid material from the Tank 22 purge ventilation demister, responsible for preventing adequate purge ventilation flow, was delivered to SRNL by SRR for analysis to identify the chemical nature of the solids and possibly address why fresh water and caustic flushes with 50 wt. % sodium hydroxide solution are essentially ineffective in dissolving or dislodging the solids from the Tank 22 demister.

Analysis results of the "as-received" Tank 22 demister solid material by ICP-AES spectroscopy, SEM/EDX and XRD techniques led to the following conclusions on the mineralogy of the demister solids.

- X-ray diffraction analysis of the "as-received" Tank 22 demister solid sample indicate the presence of amorphous material and a crystalline mercury compound called Mosesite. Mosesite has different chemical formulae depending on the nature of the anion species present in the medium [(Hg_2NCl*H_2O or $(Hg_2N)(Cl,SO_4,MoO_4)*H_2O$ or $Hg_2N(Cl,SO_4,MoO_4,CO_3)\bullet H_2O$)].

- The analytical variations and large uncertainties in the analytical measurement results for Fe [50.300 mg/g solid (52.20 %RSD)], Cr [7.950 mg/g solid (69.65 %RSD)], Ni [3.75 mg/g solid (67.08 %RSD)], Mn [0.847 mg/g solids (58.42 %RSD)], Cu [0.209 mg/g solid (72.03 %RSD)], Zn [0.174 mg/g solid (30.49 %RSD)], Mo [0.123 mg/g solid (66.84 %RSD)], Ba [0.11 mg/g solid (40.84 %RSD)], and)], Co [0.063 mg/g solids (72.23%RSD] reflect the inhomogeneous nature of the "as-received" Tank 22 demister solid sample.

- Other measurable elemental constituents of the Tank 22 demister solids include Hg [563.920 mg/g solid (12.54%RSD)], S [1.320 mg/g solid (2.00 %RSD)], Al [1.24 mg/g solid (18.53 %RSD)], Si [0.905 mg/g solid (17.78 %RSD)], Ca [0.114 mg/g solid (24.80 %RSD)], and Mg [0.086 mg/g solid (14.09 %RSD)]. Thus, mercury alone constitutes about 56.4 percent, by weight, of the Tank 22 demister solids.

- Most of the other compounds identified in the "as-received" Tank 22 Demister solid sample are mainly transition metal amalgams with mercury, which forms the bulk of the amorphous phases of the demister solids.

A literature review on Mosesite chemistry indicates that all forms of the mineral are only soluble in hydrochloric acid. However, since this heterogeneous "as-received" solid sample from the Tank 22 demister is partially amorphous, it is recommended to initially leach or dislodge this material from the Tank 22 demister with pressurized water and appropriate sodium hydroxide solution at greater than room temperature (45-65 degree Centigrade preferred because a temperature of about 20 ^{o}C, currently in use, is not effective) conditions as the solids accumulates over time in the demister.

The TAR[1] for this Tank 22 demister solid sample characterization also called for the evaluation of other Tank Farm compatible dissolution solutions, mainly inhibited water and 30 -50 wt. % sodium hydroxide solutions, which could be used to solubilize and dissolve the Tank 22 demister solids. However, because of the availability of a limited quantity of the Tank 22 demister solids, these evaluations were not performed at this time (See Appendix B for Information on the availability of more Tank 22 demister solids).

We currently do not have sufficient information to accurately quantify the amount of Mosesite mineral present in the Tank 22 demister solids. However, if one assumes that all the total mercury, which forms about 56.4%, by weight, the elemental composition of Mosesite, it may be conservative enough to conclude that Mosesite constitutes about 56.4%, by weight, the total mass of the Tank 22 demister solids; with the difference of 46% by weight, being those parts of the demister solids which are amorphous.

Taking advantage of the existence of about 46 wt.% of the solids being amorphous material, which may be more easily dislodged from the entire solids network of the demister solids in comparison to the pure Mosesite mineral, it is recommended that future work to prevent the fouling of the demister by these solid materials would include dissolution studies in 30-50 wt. %

sodium hydroxide solutions and inhibited water at above ambient temperature conditions. It is also recommended that a complete water leaching of the demister solids be performed at above ambient temperature conditions and the resulting solutions analyzed for soluble / leachable anion components. This information, including particle size distribution for the solids, will be needed to elucidate the formation mechanism for Mosesite in the demister as well as identify the version of this crystalline mercury compound present in the Tank 22 demister solids.

5.0 Quality Assurance

Appendix A contains the Laboratory Information Management System (LIMS) numbers for tracking the analytical data presented in this report. The sample analysis completion dates are tracked in LIMS. Data are recorded in SRNL Electronic Notebook[2] and various AD notebooks contain the analytical/experimental data. Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. This document, including all calculations was reviewed by Design Verification by Document Review [3, 4]. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2. The TAR requested no specific functional classification.

6.0 References

1. Paul E. Carroll, "Tank 22 purge ventilation demister pluggage" Technical Assistance Request (TAR), U-TAR-H-00018, August 20, 2020.
2. L. N. Oji: ELN: L5575-00080-14 (Electronic Notebook (Production); SRNL, Aiken, SC 29808 (2014).
3 "Savannah River National Laboratory Technical Report Design Check Guidelines", WSRC-IM-2002-00011, Revision 2, August 2004.
4 "Technical Reviews", Manual E7, Procedure 2.60, Revision 18, December 2, 2019.

Appendix A. Sample Characterization ARD tracking numbers

Analytes	Method (s)	SRNL AD Tracking Number (LIMS):
Elemental	ICP-AES	LW18390, LW18391, LW18392, LW18397
Hg	DMA	LW18390, LW18391, LW18392, LW18397
XRD	XRD	LW18388
SEM/EDX	SEM/EDX	LW18389

*Project: IDs: LW-AD-PROJ-200817-2.

Appendix B. Information on the availability of more Tank 22 demister solids

RE: More Tank 22 Demister solids; More samples?

Paul02 Carroll
Wed 02/24/2021 10:29 AM
To:

Lawrence Oji

Cc:

- Boyd Wiedenman;
- Lorrie Mobley;
- Jimmie Stuberfield

Larry

We will not pursue further dissolution studies on the Tank 22 demister. There is an initiative moving forward to reduce DWPF mercury returns to the Tank Farm, specifically Tank 22. This should improve demister performance in itself. We will continue the strategy of minimizing purge fan run time to lessen demister fouling rate. Modifications we are planning are improved flushing arrangements, a wider diameter demister (slow fouling rate) and improving demister replacement access to make future replacements ALARA.

I suspect we need a TTR revision to reduce the original scope.

Paul

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Cc: Boyd Wiedenman <Boyd.Wiedenman@srnl.doe.gov>
Subject: RE: More Tank 22 Demister solids; More samples?

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