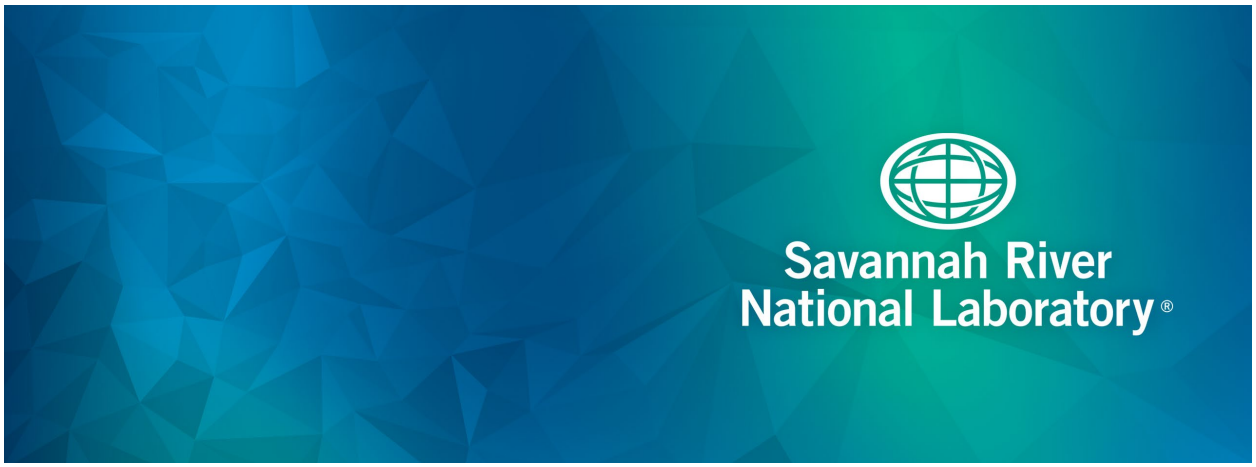


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# MERCURY TESTING WITH SLUDGE BATCH 10 TANK 40 SIMULANT

**A. M. Howe**

**D. P. Lambert**

June 2024

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At the request of Savannah River Mission Completion (SRMC), these experiments were planned and completed to understand why the Defense Waste Processing Facility (DWPF) is not collecting elemental mercury ($Hg^0$) in the Mercury Water Wash Tank (MWWT) despite long boiling times. The primary authors appreciate the support of Bill Holtzscheiter and the DWPF Chemical Processing Cell (CPC) group in supporting the planning of these experiments.

Special thanks to Katherine Miles, Matthew Siegfried, Sean Noble and Daniel Jones for providing extended coverage, including night shift. In addition, Matt Williams, Kandice Miles and Wesley Woodham were instrumental in calibrating off-gas instruments and monitoring off-gas species during each experiment.

# EXECUTIVE SUMMARY

Savannah River Mission Completion (SRMC) requested that researchers at Savannah River National Laboratory (SRNL) perform testing designed to examine why the Defense Waste Processing Facility (DWPF) is not collecting elemental mercury in the Mercury Water Wash Tank (MWWT). In order for DWPF to recover mercury, mercuric oxide must first be reduced to elemental mercury. The elemental mercury must then be steam stripped, condense, and coalesce in the Mercury Water Wash Tank (MWWT) during chemical processing in the Sludge Receipt and Adjustment Tank (SRAT). The efficiency of these steps was investigated in a series of laboratory scale SRAT experiments under the nitric-glycolic and nitric-formic flowsheets utilizing Momentive Y-17112 and Antifoam 747. Mercury speciation in the Slurry Mix Evaporator Condensate Tank (SMECT) and condensate streams was also examined. The key conclusions from these experiments are as follows:

Mercury II Oxide may not be fully reduced to elemental mercury during acid addition at 93°C. Higher temperatures, i.e., boiling may be necessary to fully reduce Mercury II Oxide.

The highest percent mercury recovery (71 %) in the MWWT was observed in the MS-NGA-17112 experiment (nitric-glycolic acid flowsheet with Momentive Y-17112), which is how DWPF is currently operating the SRAT.

More residual mercury remained in the nitric-glycolic SRAT products (12.3% and 15.0%) in contrast to the nitric-formic SRAT products (1.7% and 3.2 %) despite a higher acid stoichiometry. While more mercury was stripped from the sludge under the nitric-formic flowsheet, less mercury was recovered in the MWWT, and less mercury was accounted for in the overall mercury mass balance.

While methylmercury was observed in all four experiments, significantly higher concentrations of methylmercury (and trimethylsilanol) were measured in the experiments with Antifoam 747. Trimethylsilanol is an antifoam degradation product of Antifoam 747. The implementation of Momentive Y-17112 significantly reduces the formation and accumulation of methylmercury in the SMECT and condensate streams, as trimethylsilanol is not formed as this defoamer degrades. Other organomercury species (ethylmercury and dimethylmercury) were below method detection limits in all condensates.

Initial data suggest that total mercury and organomercury are both extremely low in the off-gas stream. Changes to the sampling location and improvements to the sampling technique, i.e., continuous mercury monitoring, however, are warranted for future testing.

SRNL recommends additional SRAT experiments with a focus on mercury chemistry. It is recommended that future testing be completed only under the nitric-glycolic acid flowsheet utilizing Momentive Y-17112 as the defoaming agent. To validate the high mercury recovery observed in the inert (nitrogen) purge experiment, additional testing with low and inert purges is advised. Furthermore, experiments with acid addition at higher temperatures, i.e., boiling, are recommended.[14]

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| | |
|---|---|
| ACTL | Aiken County Technology Laboratory |
| CPC | Chemical Process Cell |
| DMA | Direct Mercury Analyzer |
| DWPF | Defense Waste Processing Facility |
| FAVC | Formic Acid Vent Condenser |
| IC | Ion Chromatography |
| ICP-AES | Inductively coupled plasma atomic emission spectroscopy |
| MST/SS | Monosodium Titanate/Sludge Solids |
| MWWT | Mercury Water Wash Tank |
| NFA | Nitric-Formic Acid |
| NGA | Nitric-Glycolic Acid |
| PSAL | Process Science Analytical Laboratory |
| RCT | Recycle Collection Tank |
| SB | Sludge Batch |
| SCFM | Standard Cubic Feet Per Minute |
| SE | Strip Effluent |
| SME | Slurry Mix Evaporator |
| SMECT | Slurry Mix Evaporator Condensate Tank |
| SRAT | Sludge Receipt and Adjustment Tank |
| SRE | Sodium Reactor Experiment |
| SRMC | Savannah River Mission Completion |
| SRNL | Savannah River National Laboratory |
| SVOA | Semi Volatile Organics Analysis |
| TIC | Total Inorganic Carbon |
| TOC | Total Organic Carbon |
| TS | Total Solids |
| TTQAP | Task Technical and Quality Assurance Plan |
| TTR | Technical Task Request |
| VOA | Volatile Organics Analysis |

## 1.0 Introduction

Researchers at Savannah River National Laboratory (SRNL) have been requested by Savannah River Mission Completion (SRMC) to perform testing designed to understand why the Defense Waste Processing Facility (DWPF) is not collecting elemental mercury ($Hg^0$) in the Mercury Water Wash Tank (MWWT) despite long boiling times designed to recover elemental mercury ($Hg^0$).[1] In response to a Technical Task Request [2] (TTR) from SRMC, a Task Technical and Quality Assurance Plan (TTQAP) was written and approved to authorize this work.[3] The TTQAP was written to generally cover the proposed testing and Run Plans were written to include the details needed for performing this testing.

There are at least five steps that must be completed for DWPF to recover mercury:

1. *Reduce mercuric oxide [4] (HgO) to elemental mercury ($Hg^0$).* Glycolic acid, a reducing acid, is added in the Sludge Receipt and Adjustment Tank (SRAT) to reduce the HgO. Other forms of mercury such as $Hg_2Cl_2$ or organomercury compounds will not be reduced by the reducing acid.
2. *Evaporate (steam strip) $Hg^0$.* Based on the vapor pressure of water and $Hg^0$ at boiling, it should take about 250 g of steam to remove 1 g of Hg. In practice, three times as much steam [5] is needed in most sludge batches leading to longer than predicted boiling times to steam strip most of the $Hg^0$ from the SRAT. It is not practical to steam strip below about 0.45 wt % mercury.
3. *Condense $Hg^0$ in the SRAT and Slurry Mix Evaporator (SME) condensers.* The SRAT and SME condensers are undersized for the current steam flow, air purge, and air in leakage resulting in condenser liquid temperatures as high as 60°C. If DWPF returns to processing at design basis steam flow, the condenser outlet temperature will be even hotter. This means the condensers do not condense as much water and mercury as they would if they were colder. The ammonia scrubbers (chilled Slurry Mix Evaporator Condensate Tank or SMECT condensate is fed to the scrubbers) and Formic Acid Vent Condenser or FAVC (~10 °C chilled water) serve as supplementary condensers.
4. *$Hg^0$ must coalesce to be collected in the Mercury Water Wash Tank (MWWT), a mercury decanter in the SRAT cycle and in the SMECT during SME processing.* If the mercury does not coalesce, it likely won't settle in the MWWT or SMECT and will be pumped through the Recycle Collection Tank (RCT) to Tank 22. Extremely fine particles of $Hg^0$ can deposit anywhere in the off-gas system and can float on the condensate and bypass the MWWT. Fine particles of $Hg^0$ are also likely to deposit in the ammonia scrubbers, causing the differential pressure across the scrubbers to increase and eventually prevent the SRAT or SME from maintaining the slight negative pressure needed for processing. The scrubbers will then be soaked in nitric acid to dissolve the mercury, negating all the time and energy needed for recovering mercury.
5. *The Hg0 must be pumped out of the MWWT or SMECT to minimize the reaction time with nitric and nitrous acid solutions, that may oxidize the Hg0 and dissolve it in the condensate.* Nitric acid in both the MWWT (from scrubbed $NO_2$ during processing, which forms nitric acid or $HNO_3$) and SMECT (scrubbed nitric acid and intentionally added nitric acid) can oxidize and dissolve the $Hg^0$ to form other mercury species.

This testing focused on mercury chemistry. Understanding the efficiency of each of the above five steps is necessary for understanding the current mercury recovery and to understand what changes are needed to maximize the mercury recovery at DWPF.

Sludge Batch (SB) 10 Tank 40 simulant was used in these SRAT simulations.[6] These experiments were completed using the Nitric-Glycolic Acid (NGA) and Nitric-Formic Acid (NFA) flowsheets (110% Koopman Acid Stoichiometry [7]) with the addition of monosodium titanate/sludge solids (MST/SS) and Strip Effluent (SE) simulants.

Much of the testing was performed at prototypic DWPF processing conditions. The testing in this study, however, did not duplicate the entire SRAT and SME cycle. For example, short segments of DWPF processing such as acid additions and reflux boiling were used to study the reduction of HgO to $Hg^0$.

A few of these prototypic DWPF processing conditions that are key to this testing include:

1. Target heat input: DWPF controls steam addition, not boil-up rate
2. Run the SRAT/SME condenser to achieve typical DWPF cooling water exit temperatures
3. Start MWWT with a known mass of $Hg^0$ to determine extent of dissolution in each test
4. Utilize ammonia scrubber during SRAT processing
5. Utilize all condensate generated during run as feed for ammonia scrubber. A simulated heel of SMECT liquid (1500 gal of pH 2 nitric acid solution) will be needed to start each run.
6. Utilize Formic Acid Vent Condenser (FAVC) during testing
7. Utilize prototypic mixing speed in this testing as mixing likely impacts the $Hg^0$ stripping efficiency

One of the keys to this testing is to understand the mercury speciation in the liquid and vapor streams. The use of various mercury methods with the direct mercury analyzer (DMA) and the use of micro-columns to absorb mercury in the off-gas are two techniques that will help us to understand the concentration and speciation in these liquid and vapor streams.

## 2.0 Experimental Procedure

### 2.1 Testing Apparatus and Methodology

SRAT/SME simulations were performed in the Aiken County Technology Laboratory (ACTL). The experiments were performed using the Mettler Toledo RC1mx apparatus shown in Figure 2-1. In addition to a SRAT/SME condenser and Mercury Water Wash Tank (MWWT), an ammonia scrubber and a Formic Acid Vent Condenser (FAVC) were installed for mercury speciation, stripping, condensing, and coalescing testing. The ammonia scrubber utilized a 0.01 M nitric acid solution as its liquid feed during the SRAT cycle. The SRAT/SME condenser and FAVC were operated at 60°C and 10°C, respectively.


**Figure 2-1. Mettler Toledo RC1mx apparatus**

2.2 Mercury Reduction Testing

It is important to understand whether mercury is completely reduced during prototypic testing. In addition, the kinetics of this reaction are equally important, as steam stripping will not occur unless the mercuric oxide is reduced to elemental mercury. Testing was completed with both the nitric-glycolic acid (NGA) flowsheet and the nitric-formic acid (NFA) flowsheet during prototypic sludge-only acid addition at 93°C. The key testing parameters for the mercury reduction experiments are summarized in Table 2-1.

**Table 2-1. Details for Mercury Reduction Testing in the RC1mx**

| Experiment | SB10 Sludge Mass, g | MST/SS Mass, g | SE Mass, g | Sludge Hg wt. % | Acid Stoichiometry | Flowsheet |
|---|---|---|---|---|---|---|
| DR-NGA | 1,650 | 0 | 0 | 3.2 | 110% | Nitric-Glycolic |
| DR-NFA | 1,650 | 0 | 0 | 3.2 | 110% | Nitric-Formic |

Slurry samples were drawn during and after acid addition and immediately diluted (100x, 1000x, and 10,000x). Once diluted, the samples were placed into an In-Vial Sparge System where the mercury was sparged onto Adsoquick columns. The in-vial sparging system is shown in Figure 2-2.

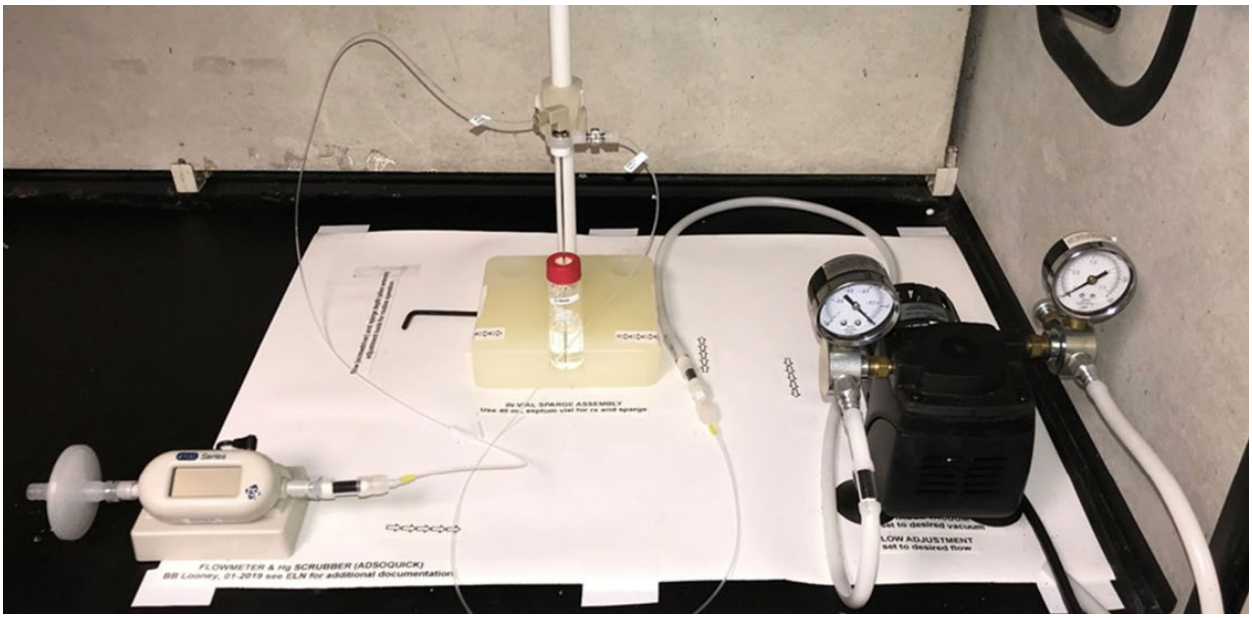

**Figure 2-2. In-Vial Sparge System**

The columns were analyzed for purgeable and total mercury via Direct Mercury Analyzer (DMA). Slurry samples were also analyzed for total mercury via Inductively coupled plasma atomic emission spectroscopy (ICP-AES) by Process Science Analytical Laboratory (PSAL). Prior to analysis, the sludge samples underwent aqua regia digestions. Approximately 1g of slurry is added to a solution consisting of 9 mL of concentrated hydrochloric acid and 3 mL of concentrated nitric acid. The solution is heated, in an open vessel, to 90°C for approximately an hour while the sample dissolves. The vessel contents are transferred to a volumetric flask and diluted with DI water to a known volume. The digestion is then run on ICP-AES for Hg analysis.

The sample plan for mercury reduction testing is provided in Table 2.2.

**Table 2-2. Sample Plan for Mercury Reduction Testing**

| Sample | Comment | Analysis |
|---|---|---|
| SRAT Receipt | SB10 Sludge, Noble Metals, HgO | Purgeable Hg via DMA, Total Hg via DMA and ICP-AES |
| Post-Nitric-Acid | Nitric Acid added in its entirety | Purgeable Hg via DMA, Total Hg via DMA and ICP-AES |
| Reducing-Acid-1 | 1/3 Reducing Acid Added | Purgeable Hg via DMA, Total Hg via DMA and ICP-AES |
| Reducing-Acid-2 | 2/3 Reducing Acid Added | Purgeable Hg via DMA, Total Hg via DMA and ICP-AES |
| Reducing-Acid-3 | Reducing Acid added in its entirety | Purgeable Hg via DMA, Total Hg via DMA and ICP-AES |
| Post-Reducing Acid 1 | 90 minutes after Reducing Acid addition | Purgeable Hg via DMA, Total Hg via DMA and ICP-AES |
| Post-Reducing Acid 2 | 3 hours after Reducing Acid addition | Purgeable Hg via DMA, Total Hg via DMA and ICP-AES |

2.3 Mercury Speciation, Stripping, Condensing, and Coalescing Testing

A series of experiments were performed to understand the effectiveness of the stripping, condensing, and coalescing of mercury during full SRAT processing. A secondary objective of these experiments was the speciation of the mercury, i.e., looking for organic and inorganic forms of mercury that were generated during processing. Furthermore, mercury mass balances were completed. The key testing parameters for these experiments are summarized in Table 2-3.

**Table 2-3. Parameters for Stripping, Condensing, and Coalescing Testing**

| Experiment ID | Antifoam | SRAT Acid Stoichiometry (%) | Coupled Operations* (Y/N) | Boil-up Rate (lbs $h^{-1}$) | Coupled Addition Rates (gal $min^{-1}$) | REDOX Target ($Fe^{2+}/\Sigma Fe$) |
|---|---|---|---|---|---|---|
| MS-NGA-17112 | Momentive Y-17112 | 110 | Yes | 5,000 | 10 | 0.1 |
| MS-NFA-17112 | Momentive Y-17112 | 100 | Yes | 5,000 | 10 | 0.1 |
| MS-NGA-747 | Antifoam 747 | 110 | Yes | 5,000 | 10 | 0.1 |
| MS-NFA-747 | Antifoam 747 | 100 | Yes | 5,000 | 10 | 0.1 |

*Coupled operations includes MST-SS and SE additions

The following additional testing parameters were followed during each experiment:

- Target SRAT product total dried solids loading: 20 wt. %
- Target SRAT condenser temperature: 60°C
- Target Rh concentration (rhodium nitrate as the precursor): $1.19\times10^{-2}$ wt. % of Total Dried Solids[a]
- Target Ru concentration (ruthenium (III) nitrosyl nitrate as the precursor): $5.84\times10^{-2}$ wt. % of Total Dried Solids[a]
- Target Pd concentration (palladium nitrate as the precursor): $2.53\times10^{-3}$ wt. % of Total Dried Solids[a]
- Target Ag concentration (silver nitrate as the precursor): $9.86\times10^{-3}$ wt. % of Total Dried Solids[a]
- Target Hg concentration (mercuric oxide as the precursor): 3.58 wt. % of Total Dried Solids
- SRAT Receipt Volume: 6000 gallons – no starting heel
- Target sludge addition: ~1.5 liters
- Target SRAT Scaling Factor: ~15,000
- Target scaled SRAT purge rate: 94 SCFM (1 atm, 21.11°C)
- Heating jacket temperature limit: 160°C
- Target temperature during acid addition: 93°C
- Antifoams: Momentive Y-17112 or Antifoam 747

[a]Addition targets for noble metals (Ag, Pd, Rh, and Ru) are calculated as 125% of the expected value in SB10 Tank 40 sludge.[10]

The following steps were performed during the experiments:

1. 1.6 kg of SB10 Tank 40 simulant (See section 2.4 for composition) was added to the test vessel. The MWWT was filled with DI water and 2 g elemental mercury was added to simulate the mercury expected to accumulate in MWWT. The starting point for the SMECT was 500 mL nitric acid solution with a pH of 2 and 30 g elemental mercury to simulate the SMECT heel and accumulated mercury.
2. Trim chemicals (SRE material, 15 wt. % MST slurry, and noble metals) were added.
3. Mercuric oxide, as depicted in Figure 2-3, was added.
4. The vessel contents were mixed for 30 minutes and SRAT receipt samples were drawn.
5. Heating was initiated (to boiling).
6. 3000 gallons (0.75 L scaled) of MST/SS simulant (soluble salt components) was added while boiling, simulated caustic boiling).
7. Vessel was cooled to 93°C.
8. Nitric acid was added.
9. Glycolic or formic acid was added.
10. SRAT was dewatered (targeting 20 wt. % solids).
11. 15,000 gallons (3.7 L scaled) Strip Effluent (SE) simulant (See Table 2.8 for composition) was added continuously while boiling.
12. SRAT was refluxed (reflux time is calculated by assuming a 750:1 $H_2O$: Hg steam stripping factor and a reduction of Hg to 0.8 wt. % of total dried solids in SRAT product).
13. SRAT product samples were drawn to assess chemical reaction extents.


**Figure 2-3. Mercuric Oxide (HgO) Added to SB10 TK40 Simulant**

Table 2-4 lists the samples that were drawn during each experiment as well as the analyses that were performed. Off-gas samples were drawn using a syringe in the off-gas line between the SRAT condenser and Ammonia Scrubber. The samples were sparged onto commercial trap columns, Adsoquick and CarbotrapB columns, and the columns analyzed for total mercury and organomercury, respectively, via DMA.

**Table 2-4. Sample Plan for Stripping, Condensing, and Coalescing Testing**

| Sample Name | Sample Source | Analysis |
|---|---|---|
| SRAT Receipt (including MST solids) | Kettle | Wt. % total dried solids, density, ICP-AES, IC, Total base, TIC/TOC, pH, Hg |
| Ammonia Scrubber Baseline | SMECT | pH, purgeable, ionic, organic, and total Hg |
| Ammonia Scrubber MST/SS Dewater | SMECT | IC, pH, purgeable, ionic, organic, and total Hg |
| Ammonia Scrubber Post Glycolic Acid | SMECT | pH, purgeable, ionic, organic, and total Hg |
| SRAT Dewater | MWWT | ICP-AES, MeHg, $(Me)_2Hg$, TIC/TOC, IC, pH |
| Ammonia Scrubber Post SRAT Dewater | SMECT | pH, purgeable, ionic, organic, and total Hg |
| Condensate Post SE Dewater | MWWT | IC, Hg, pH |
| SRAT Product | Kettle | Wt. % total dried solids, density, ICP-AES, IC, TIC/TOC, pH, MeHg, $(Me)_2Hg^{\dagger}$ |
| Ammonia Scrubber End of SRAT | SMECT | IC, ICP, purgeable, ionic, organic, and total Hg, TIC/TOC, VOA, SVOA, IC |

## 2.4 Simulants

Sludge Batch 10 Tank 40 Simulant was used for this testing. The density and solids properties of the SRAT receipt conditions are given in Table 2-5. “Tank 40 Simulant Receipt” was measured from the Tk40-10 SRAT receipt sample.[10]

**Table 2-5. Density and Solids Properties of Tank 40 Simulant Receipt**

| Parameter | Tank 40 Receipt |
|---|---|
| Slurry Density (g/mL) | 1.11 |
| Supernatant Density (g/mL) | 1.05 |
| Total Dried Solids (wt.% of slurry) | 13.9 |
| Dissolved Solids (wt.% of filtrate) | 5.95 |
| Insoluble Solids (wt.% of slurry) | 8.49 |
| Soluble Solids (wt.% of slurry) | 5.44 |
| Calcined Solids (wt.% slurry) | 10.0 |

The composition of the SRAT Receipt Slurry with added Sodium Reactor Experiment (SRE) material is given in Table 2-6. The concentrations of the sludge components are reported on both a slurry and a supernatant basis. Metals in the slurry are reported on a total dried solids (TS) basis. These analyses provide enough information to determine an effective acid requirement.

**Table 2-6. Composition of Tank 40 Simulant Receipt Sludge and Supernate**

| Elements/Anions | Tank 40 Slurry | Elements/Anions | Tank 40 Supernate |
|---|---|---|---|
| Ag (wt. % of TS) | <7.00E-02 | Ag (M) | <9.27E-06 |
| Al (wt. % of TS) | 1.208E01 | Al M) | 9.08E-02 |
| B (wt. % of TS) | <7.00E-02 | B (M) | 1.36E-03 |
| Ba (wt. % of TS) | <7.00E-02 | Ba (M) | <7.28E-06 |
| Ca (wt. % of TS) | 6.70E-01 | Ca (M) | <2.50E-05 |
| Cr (wt. % of TS) | 2.30E-01 | Cr (M) | 1.20E-03 |
| Cu (wt. % of TS) | <7.00E-02 | Cu (M) | <1.57E-05 |
| Fe (wt. % of TS) | 1.131E01 | Fe (M) | <1.79E-05 |
| Hg (wt. % of TS) | 3.80E00 | Hg (M) | 2.37E-04 |
| K (wt. % of TS) | <7.00E-02 | K (M) | 1.44E-03 |
| Li (wt. % of TS) | <7.00E-02 | Li (M) | <1.44E-04 |
| Mg (wt. % of TS) | 2.20E-01 | Mg (M) | <4.11E-05 |
| Mn (wt. % of TS) | 4.16E00 | Mn (M) | <1.82E-05 |
| Na (wt. % of TS) | 1.416E01 | Na (M) | 8.55E-01 |
| Ni (wt. % of TS) | 4.30E-02 | Ni (M) | <1.70E-05 |
| P (wt. % of TS) | <7.00E-02 | P (M) | 5.97E-05 |
| Pd (wt. % of TS) | <7.00E-02 | Pd (M) | 2.29E-05 |
| Rh (wt. % of TS) | <7.00E-02 | Rh (M) | <9.72E-06 |
| Ru (wt. % of TS) | <7.00E-02 | Ru (M) | <9.89E-06 |
| S (wt. % of TS) | 2.30E-02 | S (M) | 1.51E-02 |
| Si (wt. % of TS) | 6.90E-02 | Si (M) | <3.56E-05 |
| Sn (wt. % of TS) | 9.00E-02 | Sn (M) | 2.12E-04 |
| Ti (wt. % of TS) | <7.00E-02 | Ti (M) | <2.09E-05 |
| Zn (wt. % of TS) | <7.00E-02 | Zn (M) | <1.53E-05 |
| Zr (wt. % of TS) | 1.40E-01 | Zr (M) | <1.10E-05 |
| $HCO_2^-$ (M) | <2.47E-03 | $HCO_2^-$ (M) | <2.22E-03 |
| $Cl^-$ (M) | <3.14E-03 | $Cl^-$ (M) | <2.82E-03 |
| $NO_2^-$ (M) | 2.20E-01 | $NO_2^-$ (M) | 2.41E-01 |
| $NO_3^-$ (M) | 1.32E-01 | $NO_3^-$ (M) | 1.37E-01 |
| $PO_4^{3-}$ (M) | <1.17E-03 | $PO_4^{3-}$ (M) | <1.05E-03 |
| $SO_4^{2-}$ (M) | 1.47E-02 | $SO_4^{2-}$ (M) | 1.47E-02 |
| $C_2O_4^{2-}$ (M) | 8.11E-03 | $C_2O_4^{2-}$ (M) | 8.02E-03 |
| $HOCH_2CO_2^-$ (M) | <1.48E-03 | $HOCH_2CO_2^-$ (M) | <1.33E-03 |
| $CO_3^{2-}$ (M) | 7.73E-02 | $CO_3^{2-}$ (M) | 9.41E-02 |
| $OH^-$ (M) | 1.60E-01 | $OH^-$ (M) | 1.64E-01 |

The MST/SS stream was added at a volume equivalent to 3000 gallons DWPF basis prior to acid addition. Approximately 140 g of a 15 wt. % MST slurry was added to the 1.6 kg SB10 TK40 sludge simulant with the remaining trim chemicals (SRE material, noble metals, and mercury). The remaining salt components of the MST/SS stream were continuously fed to the kettle using one of the reagent pumps during caustic boiling. This strategy, while not fully representative of the process employed at the DWPF, will accurately capture the chemical changes occurring in the SRAT by continuously adding caustic components while providing a bounding basis for foam control needs and ensuring solids are high throughout the MST/SS addition process. The pumping of MST at the laboratory scale is problematic, as the solid particles are too large to remain suspended during constant flow. The chemical composition of the salt stream simulating the soluble phase of the MST/SS addition is given in Table 2-7.

**Table 2-7. Chemical Composition of MST/SS Salt Stream**

| Component | (wt%) |
|---|---|
| Sodium Aluminate - $NaAl(OH)_4$ | 0.365 |
| Sodium Formate - $NaHCO_2$ | 0.012 |
| Sodium Nitrite - $NaNO_2$ | 0.610 |
| Sodium Nitrate - $NaNO_3$ | 0.610 |
| Sodium Carbonate - $Na_2CO_3$ | 0.488 |
| Sodium Hydroxide - NaOH | 1.098 |
| Potassium Carbonate - $K_2CO_3$ | 0.036 |

The SE stream was added at a volume equivalent to 15,000 gallons (DWPF basis). This material was continuously fed to the kettle using one of the reagent pumps during strip effluent addition while boiling. To be consistent with previous Sludge Batch 10 flowsheet testing, the SE stream consisted of a dilute boric acid solution.[10] The chemical composition of the SE simulant is given in Table 2-8.

**Table 2-8. Chemical Composition of SE Simulant**

| Component | Concentration (wt%) |
|---|---|
| Water | 99.94% |
| Boric Acid - $H_3BO_3$ | 0.06% |

## 3.0 Results and Discussion

### 3.1 Mercury Reduction Testing

Two shortened (acid addition and a three hour hold at 93°C) SRAT experiments were completed to aid DWPF in understanding mercury reduction during sludge processing. The processing conditions were identical except for the reducing acid utilized. DR-NGA was conducted under the nitric glycolic acid flow sheet with a 110% acid stoichiometry and DR-NFA was conducted under the nitric formic acid flowsheet with a 110% acid stoichiometry (Koopman basis). 8.71 g mercuric oxide was added as a trim chemical achieving a target mercury concentration of approximately 5,000 mg/L. The mercuric oxide likely contains trace quantities elemental mercury. Elemental mercury analytical results via DMA and total mercury results via aqua regia ICP-AES are provided in Table 3-1. An analytical data report detailing the elemental mercury results is provided in Appendix A. Total mercury ICP-AES results are often low for the initial sludge receipt samples due to inadequate mixing at the start of experiments.

**Table 3-1. Mercury Reduction Testing Results**

| Sample | Experiment | Acid Added gmol | Elemental Mercury mg/L | Total Mercury mg/L | Elemental Mercury % |
|---|---|---|---|---|---|
| Sludge Receipt | DR-NGA | 0 | 72 | 3550 | 2.03 |
| | DR-NFA | 0 | 107 | 3510 | 3.05 |
| Post Nitric Acid | DR-NGA | 0.85 | 294 | 4770 | 6.16 |
| | DR-NFA | 0.18 | 161 | 4410 | 3.65 |
| Reducing Acid 1 | DR-NGA | 1.20 | 54 | 4850 | 1.11 |
| | DR-NFA | 0.75 | 537 | 4460 | 12.04 |
| Reducing Acid 2 | DR-NGA | 1.55 | 382 | 4830 | 7.91 |
| | DR-NFA | 1.32 | 1787 | 4820 | 37.07 |
| Reducing Acid 3 | DR-NGA | 1.90 | 898 | 4760 | 18.87 |
| | DR-NFA | | 1359 | 4920 | 27.62 |
| Post Reducing Acid | DR-NGA | 1.90 | 1444 | 4840 | 29.83 |
| | DR-NFA | | 1440 | 4900 | 29.39 |
| Post Reducing Acid 2 | DR-NGA | 1.90 | 1624 | 4740 | 34.26 |
| | DR-NFA | | 1341 | 4880 | 29.32 |

Figure 3-1 shows the percentage of elemental mercury present in the sludge over the span of the experiments. As expected, elemental mercury is low (< 5%) in the original sludge receipt samples. The decrease in elemental mercury after the initial glycolic acid addition is unexpected and likely caused by poor subsampling prior to sparging onto the Adsoquick column. The results indicate that the initial rate of mercury reduction is greater under the nitric-formic flowsheet. At the completion of the experiment, however, the sludge processed under the nitric-glycolic flowsheet contained a higher proportion of elemental mercury. Approximately 30% of the mercury oxide was reduced to elemental mercury three hours post reducing acid addition under the NFA flowsheet. Under the NGA flowsheet, 34% of the mercury oxide was reduced to elemental mercury three hours after the reducing acid addition was completed. The percent elemental mercury reported was low (< 50%) in both experiments. This may be due to experimental conditions such as the sludge temperature remaining at 93°C and never reaching boiling temperature or poor detection of elemental mercury resulting from the sample sparging technique that was used. Results from subsequent experiments, discussed in section 3.2, indicate that mercury reduction is significantly greater. This is based on the amount of total mercury remaining in the SRAT product and the amount of elemental mercury recovered in the MWWT.

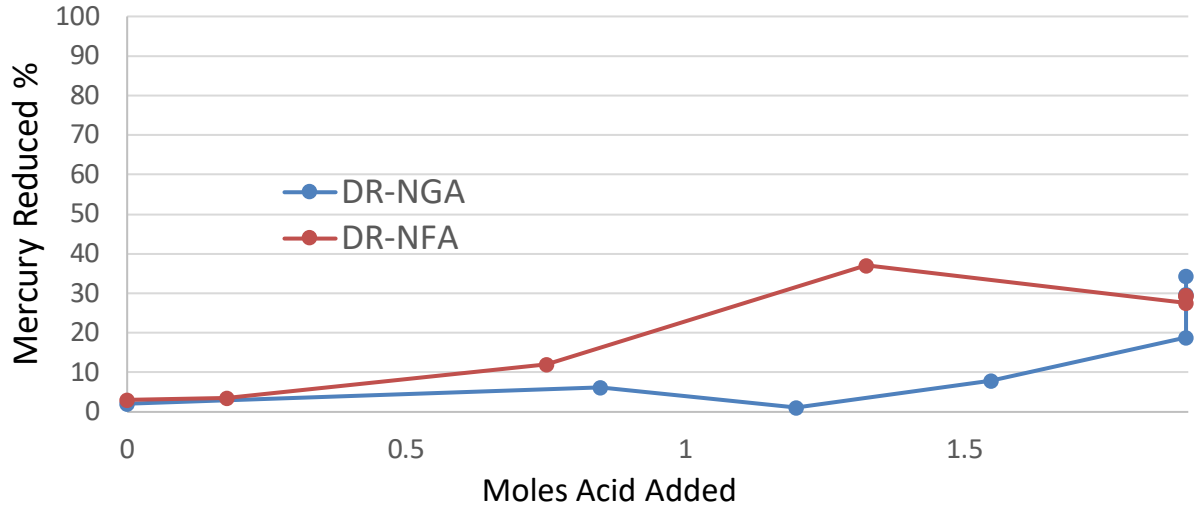


**Figure 3-1. Percent Elemental Mercury**

3.2 Mercury Speciation, Stripping, Condensing, and Coalescing Experiments

Four complete coupled SRAT simulations, including MST/SS addition, acid addition, dewater, and SE addition, were completed to aid DWPF in understanding mercury speciation, stripping, condensing, and coalescing.

1. MS-NGA-17112: nitric glycolic acid flowsheet, 110% acid stoichiometry, Momentive Y-17112
2. MS-NGA-747: nitric glycolic acid flowsheet, 110% acid stoichiometry, Antifoam 747
3. MS-NFA-17112: nitric formic acid flowsheet, 100% acid stoichiometry, Momentive Y-17112
4. MS-NFA-747: nitric formic acid flowsheet, 100% acid stoichiometry, Antifoam 747

The processing conditions were identical except for reducing acid, acid stoichiometry, and antifoam agent utilized. A timetable for the experiments is shown in Table 3-2.

**Table 3-2. Significant Experiment Events**

| Event | MS-NGA-17112 | MS-NGA-747 | MS-NFA-17112 | MS-NFA-747 |
|---|---|---|---|---|
| MST/SS Start | 6/20/22 6:43 PM | 7/18/2022 2:25 PM | 7/20/2022 4:17 PM | 9/6/2022 3:17 PM |
| Nitric Acid Start | 6/21/22 12:36 AM | 7/18/2022 8:16 PM | 7/20/2022 11:07 PM | 9/6/2022 8:32 PM |
| Reducing Acid Start | 6/21/22 2:19 AM | 7/18/2022 10:03 PM | 7/20/2022 11:43 PM | 9/6/2022 9:01 PM |
| Dewater Start | 6/21/22 4:38 AM | 7/19/2022 12:15 AM | - | - |
| SE Start | 6/21/22 4:51 AM | 7/19/2022 12:45 AM | 7/21/2022 1:52 AM | 9/6/2022 11:13 PM |
| SE End | 6/22/22 8:07 AM | 7/20/2022 2:03 AM | 7/22/2022 3:07 AM | 9/8/2022 12:21 AM |

***Temperature and pH Profiles***

Temperature and pH profiles are provided in Figure 3-2 and Figure 3-3, respectively. The temperature trends for these four SRAT simulations were very similar. There were no temperature excursions during acid addition. Also, there were no jacket temperature excursions during boiling that might be indicative of different reaction mechanisms. The non-compensated pH profiles (actual processing temperature of 93°C and boiling), as shown in Figure 3-3, are also very similar. The pH of the SRAT receipt ranged between 12 and 13. The minor differences in pH at the start of each experiment are due to offsets during pH probe calibration. The pH dropped at a similar rate for all four experiments during acid addition. For the nitric-glycolic acid flowsheet sheet experiments, the pH reduced to and maintained at 3. For the nitric-formic acid flowsheet experiments, the pH dropped to 4 during acid addition but rebounded to 6 during the remainder of the SRAT cycle, i.e., SE addition. The difference in pH is due to varying acid stoichiometries.

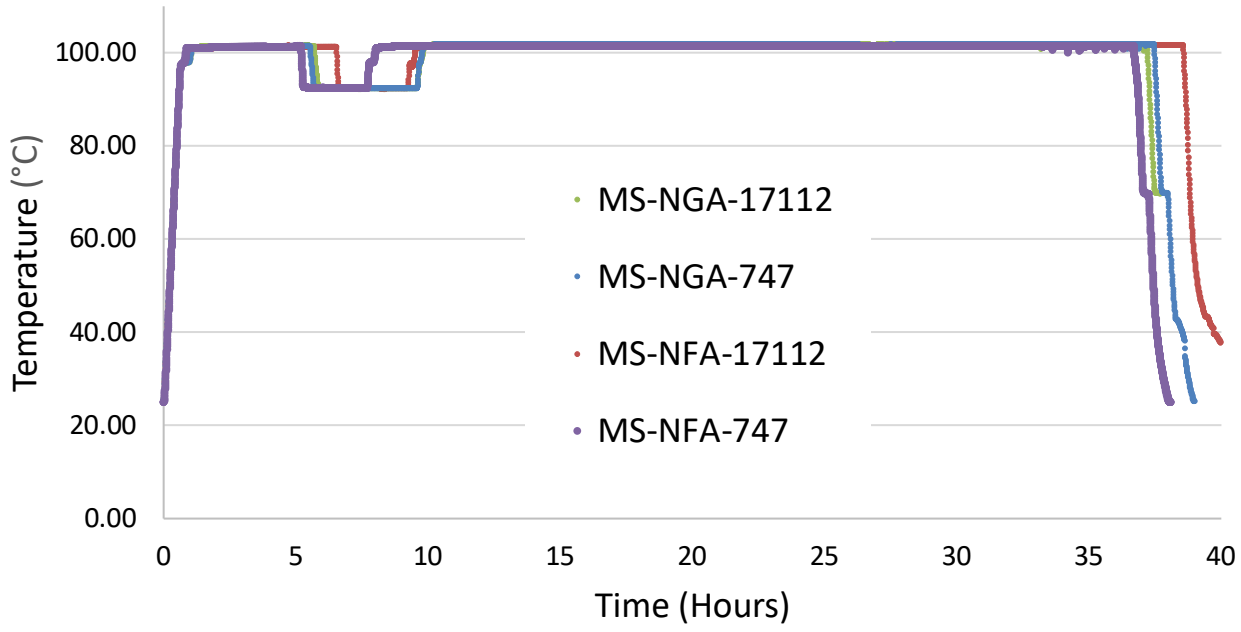


**Figure 3-2. Temperature Profiles of Mercury Stripping Experiments**

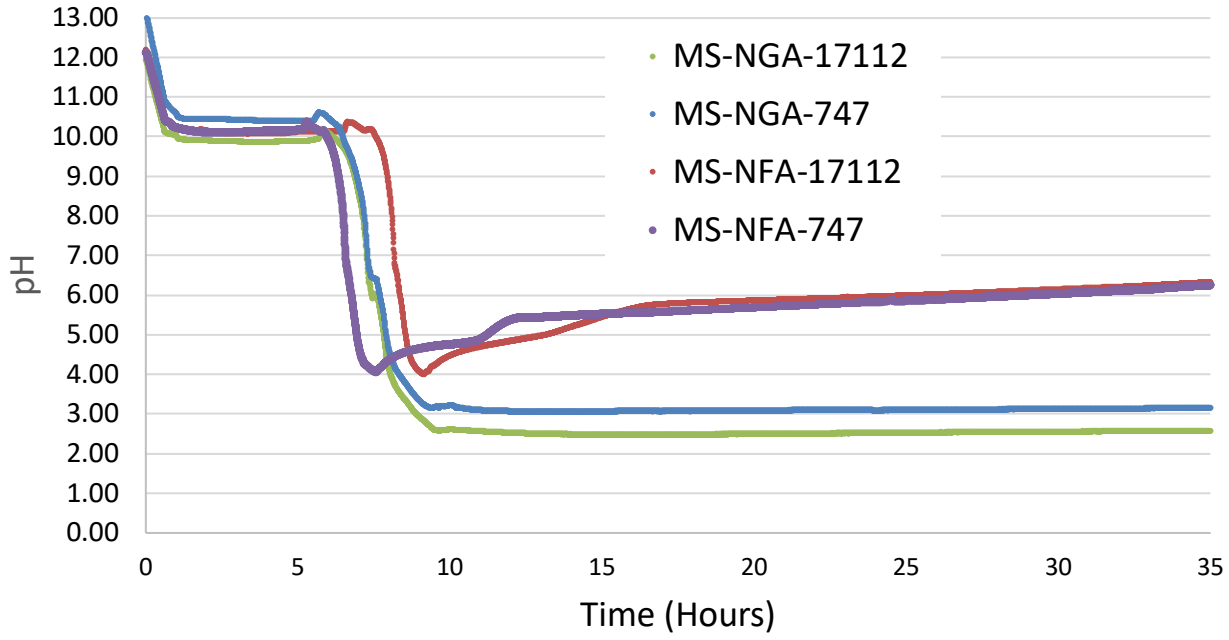


**Figure 3-3. Non-Compensated pH Profiles of Mercury Stripping Experiments**

***Mercury Results***

Mercury results for the SRAT receipt samples are provided in Table 3-3. Approximately 9 grams (3.58% of total solids) of mercury was added to the sludge in the form of mercuric oxide prior to starting each experiment. Total mercury ICP-AES results can be low for the initial sludge receipt samples if there is inadequate mixing at the start of the experiments.

**Table 3-3. Mercury Results - SRAT Receipt**

| Experiment | SRAT Receipt Mass | Mercury added (as HgO[1]) | Total mercury | |
|---|---|---|---|---|
| | g | g | mg/kg | mg |
| **NGA-17112** | 1920 | 9.014 | 3695 | 7094 |
| **NGA-747** | 1920 | 9.016 | 4015 | 7709 |
| **NFA-17112[2]** | 1920 | 9.017 | 4650 | 8928 |
| **NFA-747[2]** | 1920 | 9.009 | 4545 | 8726 |

[1] Equivalent to 9.73 g HgO

[2] Extended mixing time prior to sampling to ensure SRAT receipt samples were adequately blended

Table 3-4 shows that more residual mercury remained in the nitric-glycolic SRAT products (12.3% and 15.0%) in contrast to the nitric-formic SRAT products (1.7% and 3.2 %) despite a higher acid stoichiometry. Processing under the NFA flowsheet and the NGA flowsheet sufficiently removes mercury to the levels needed for processing in the DWPF melter. The mercury was below the target of 0.8 wt. % for every SRAT product ranged from 0.083 to 0.28 wt. %. The stripping rates were also below the target of 750 g steam/g Hg ranging from 507.9 to 593.8 g steam/g Hg. The implementation of the NGA flowsheet and Momentive Y-17112 mitigated the generation of hydrogen and other flammable antifoam degradation products at DWPF. This may allow for acid addition at higher temperatures, i.e., boiling. This in return, may lead to improved mercury reduction and recovery.

**Table 3-4. Mercury Results – SRAT Product**

| Experiment | SRAT Product Mass | Total Hg | | Residual Mercury | Hg Wt % Total Solids | Stripping Rate |
|---|---|---|---|---|---|---|
| | g | mg/kg | mg | wt % | wt % | g steam/g Hg |
| **MS-NGA-17112** | 1968 | 562 | 1106 | 12.3 | 0.28 | 573.4 |
| **MS-NGA-747** | 1943 | 695 | 1350 | 15.0 | 0.32 | 593.8 |
| **MS-NFA-17112** | 1850 | 83.4 | 154 | 1.71 | 0.045 | 507.9 |
| **MS-NFA-747** | 1857 | 154 | 286 | 3.17 | 0.083 | 515.3 |

At the completion of the SRAT cycle, the MWWT contents were drained to a tared sample bottle, liquid was removed from the mercury suspension and the remaining mercury was placed in a desiccator to remove any residual liquid, and the mass of the recovered mercury was recorded. Photos of the desiccated mercury collected from the MWWT are provided in Figure 3-4.

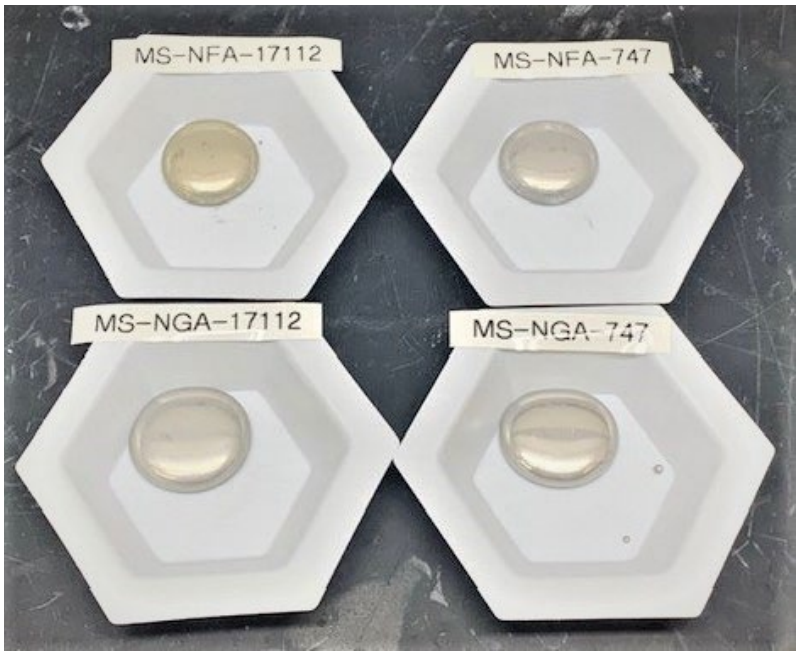


**Figure 3-4. Mercury Recovered from MWWT after Desiccation**

The mass of mercury recovered and percent mercury recovery from the MWWT are shown in Table 3-5. Mercury recovery in the MWWT ranged from 50.7% to 71.0%. The highest mercury recovery in the MWWT was observed in MS-NGA-17112 (nitric-glycolic acid flowsheet with Momentive Y-17112).

**Table 3-5. Mercury Recovered - MWWT**

| Experiment | Hg Recovered MWWT | Hg Recovered MWWT |
|---|---|---|
| | g | % |
| **MS-NGA-17112** | 6.4002 | 71.0 |
| **MS-NGA-747** | 5.5046 | 61.1 |
| **MS-NFA-17112** | 4.9638 | 55.1 |
| **MS-NFA-747** | 4.5665 | 50.7 |

The MST/SS Dewater, SRAT Dewater, SE Dewater, and FAVC condensates were analyzed for total mercury. These results are shown in Table 3-6. As expected, total mercury was low (1.98 mg/L – 3.85 mg/L) in the initial MST/SS condensates. During MST/SS addition, the mercury remained in its oxide form and could not be steam stripped. After acid addition, much of the mercury was reduced to elemental mercury and could be steam stripped. Therefore, a higher concentration of total mercury was observed in the SRAT Dewater and SE Dewater condensates, ranging from 25.7 mg/L to 5455 mg/L. Total mercury in the FAVC ranged from 483 mg/L to 742 mg/L. Higher FAVC mercury concentrations were observed in MS-NGA-17112 and MS-NFA-17112, i.e., the experiments utilizing Momentive Y-17112 as the defoaming agent.

**Table 3-6. Mercury Results – Condensates**

| Experiment | Condensate | Mass | Density | Total Mercury | | Total |
|---|---|---|---|---|---|---|
| | | g | g/mL | mg/L | mg | mg |
| NGA-171112 | MST/SS Dewater | 762.5 | 0.9971 | 3.85 | 2.94 | 226.2 |
| | SRAT Dewater | 24.46 | 1.0206 | 3,940 | 94.4 | |
| | SE Dewater | 3737.08 | 0.9976 | 32.9 | 123 | |
| | FAVC | 8.48 | 1.0046 | 675 | 5.70 | |
| NGA-747 | MST/SS Dewater | 751.56 | 0.9971 | 1.98 | 1.49 | 363.3 |
| | SRAT Dewater | 49.48 | 1.0296 | 5,455 | 262 | |
| | SE Dewater | 3744.6 | 0.9974 | 25.7 | 96.5 | |
| | FAVC | 6.21 | 1.0054 | 483 | 2.98 | |
| NFA-171112 | MST/SS Dewater | 745.98 | 0.9972 | 1.94 | 1.45 | 317.4 |
| | SRAT Dewater | - | - | - | - | |
| | SE Dewater | 3748.9 | 0.9984 | 82.8 | 311 | |
| | FAVC | 6.89 | 1.0142 | 742 | 5.04 | |
| NFA-747 | MST/SS Dewater | 746.1 | 0.9983 | 2.94 | 2.20 | 132.6 |
| | SRAT Dewater | - | - | - | - | |
| | SE Dewater | 3742.6 | 0.9984 | 34.0 | 128 | |
| | FAVC | 5.96 | 1.0186 | 490 | 2.87 | |

To examine mercury speciation, the SMECT was periodically sampled after several experiment events (MST/SS addition, reducing acid addition, SRAT dewater, and SE addition). Total mercury, inorganic mercury, and purgeable mercury results are provided in Table 3-7. Both inorganic and purgeable mercury were detected in nearly all SMECT samples ranging from 2.95 mg/L to 264 mg/L. Organomercury results are provided in Table 3-8. Ethylmercury and dimethylmercury were below instrument detection limit for every sample analyzed and therefore are not reported. While methylmercury was observed in SMECT samples in all four experiments, higher concentrations of methylmercury are linked to the presence of trimethylsilanol, a degradation product of Antifoam 747.[8] For MS-NGA-17112 and MS-NFA-17112 methylmercury ranged from <1 mg/L to 4.24 mg/L and for MS-NGA-747 and MS-NFA-747 methylmercury ranged from <1 to 53.4 mg/L. Trimethylsilanol was only detected in MS-NGA-747 and MS-NFA-747, i.e., the experiments that utilized Antifoam 747 as the defoaming agent.

**Table 3-7. Mercury Results - SMECT**

| Experiment | Sample | SMECT Mass* | Density | Total Hg | | Inorganic Hg | | Purgeable Hg | |
|---|---|---|---|---|---|---|---|---|---|
| | | g | g/mL | mg/L | mg | mg/L | mg | mg/L | mg |
| MS-NGA-17112 | AS-Baseline | 499.6 | 0.9976 | 88.7 | 44.4 | < 1 | < 1 | 17.4 | 8.71 |
| | AS-MST/SS | 471.0 | 0.9975 | 56.3 | 26.6 | < 1 | < 1 | 7.97 | 3.76 |
| | AS-Glycolic | 409.3 | 0.9982 | 147.0 | 60.3 | 4.39 | 1.80 | 30.1 | 12.3 |
| | AS-Dewater | 380.4 | 0.9986 | 113.0 | 43.0 | 6.00 | 2.29 | 51.6 | 19.7 |
| | AS-SE | 348.9 | 0.9992 | 211.0 | 73.9 | 2.95 | 1.03 | 154.0 | 53.8 |
| MS-NGA-747 | AS-Baseline | 499.7 | 0.9989 | 80.8 | 40.4 | 16.4 | 8.20 | 80.7 | 40.4 |
| | AS-MST/SS | 467.1 | 0.9989 | 80.8 | 37.8 | 18.3 | 8.56 | 19.8 | 9.26 |
| | AS-Glycolic | 405.8 | 0.9997 | 108.0 | 43.8 | 13.3 | 5.40 | 47.1 | 19.1 |
| | AS-Dewater | 373.8 | 1.0000 | 119.0 | 44.5 | 22.5 | 8.41 | 60.3 | 22.5 |
| | AS-SE | 339.6 | 1.0008 | 170.0 | 57.7 | 86.6 | 29.4 | 153.0 | 51.9 |
| MS-NFA-171112 | AS-Baseline | 499.9 | 0.9989 | 25.3 | 12.7 | 5.72 | 2.86 | 14.9 | 7.46 |
| | AS-MST/SS | 468.3 | 0.9993 | 32.2 | 15.1 | 2.66 | 1.25 | 30.5 | 14.3 |
| | AS-Formic | 403.0 | 0.9997 | 37.6 | 15.2 | 4.26 | 1.72 | 35.5 | 14.3 |
| | AS-Dewater | - | - | - | - | - | - | - | - |
| | AS-SE | 371.6 | 1.0026 | 176.0 | 65.2 | 26.5 | 9.82 | 264.0 | 97.8 |
| MS-NFA-747 | AS-Baseline | 500.1 | 0.9980 | 115.0 | 57.6 | 13.6 | 6.81 | 116.0 | 58.1 |
| | AS-MST/SS | 469.7 | 0.9972 | 116.0 | 54.60 | 9.75 | 4.59 | 128.0 | 60.3 |
| | AS-Formic | 411.7 | 0.9986 | 101.0 | 41.6 | 5.2 | 2.14 | 49.6 | 20.4 |
| | AS-Dewater | - | - | - | - | - | - | - | - |
| | AS-SE | 381.6 | 1.0011 | 214.0 | 81.6 | 15.7 | 5.98 | 178.0 | 67.9 |

*The mass of the SMECT solution decreases over time due to sampling

**Table 3-8. Methylmercury Results - SMECT**

| Experiment | Sample | SMECT Mass* | Density | Trimethylsilanol | Methyl Mercury | |
|---|---|---|---|---|---|---|
| | | g | g/cm^3 | mg/L | mg/L | mg |
| MS-NGA-17112 | AS-Baseline | 499.6 | 0.9976 | < 0.2 | < 1 | <0.5 |
| | AS-MST/SS | 471.0 | 0.9975 | < 0.2 | < 1 | <0.5 |
| | AS-Glycolic | 409.3 | 0.9982 | < 0.2 | 1.11 | 0.46 |
| | AS-Dewater | 380.4 | 0.9986 | < 0.2 | 1.66 | 0.63 |
| | AS-SEFT | 348.9 | 0.9992 | < 0.2 | 1.02 | 0.36 |
| MS-NGA-747 | AS-Baseline | 499.7 | 0.9989 | < 0.2 | < 1 | <0.5 |
| | AS-MST/SS | 467.1 | 0.9989 | 0.43 | 3.82 | 1.79 |
| | AS-Glycolic | 405.8 | 0.9997 | 0.58 | 40.7 | 16.5 |
| | AS-Dewater | 373.8 | 1.0000 | 0.55 | 53.4 | 20.0 |
| | AS-SE | 339.6 | 1.0008 | 0.44 | 14.6 | 4.95 |
| MS-NFA-17112 | AS-Baseline | 499.9 | 0.9989 | < 0.2 | < 1 | <0.5 |
| | AS-MST/SS | 468.3 | 0.9993 | < 0.2 | 1.58 | 0.74 |
| | AS-Formic | 403.0 | 0.9997 | < 0.2 | 3.18 | 1.28 |
| | AS-Dewater | - | - | - | - | - |
| | AS-SE | 371.6 | 1.0026 | < 0.2 | 4.24 | 1.57 |
| MS-NFA-747 | AS-Baseline | 500.1 | 0.9980 | < 0.2 | 1.35 | 0.68 |
| | AS-MST/SS | 469.7 | 0.9972 | 0.83 | 3.37 | 1.59 |
| | AS-Formic | 411.7 | 0.9986 | 1.3 | 36.0 | 14.8 |
| | AS-Dewater | - | - | - | - | - |
| | AS-SE | 381.6 | 1.0011 | 0.63 | 8.04 | 3.06 |

*The mass of the SMECT solution decreases over time due to sampling

***Mercury Balance***

Mercury mass balances performed for each experiment are shown in Table 3-9. This was achieved utilizing the total mercury results from the SRAT products, SRAT condensates (MST/SS Dewater, SRAT Dewater, SE Dewater, SMECT, and FAVC), and the mercury recovered from the MWWT. It is worth noting that 9.01 g of mercury (9.73 g mercuric oxide) was added to the SRAT Receipt and the total mercury accounted for ranged from 5.01 g (MS-NFA-747) to 7.76 g (MS-NGA-17112). The mercury balance was highest for MS-NGA-17112 and MS-NGA-747 at 86.1% and 80.2% respectively. For MS-NFA-17112 and MS-NFA-747, only 60.9% and 55.6% of the total mercury was accounted for. In the future, improvements to the mercury mass balance will be achieved by preserving and analyzing all rinses and residues generated during cleanup after each experiment.

**Table 3-9. Mercury Mass Balance**

| Source | MS-NGA-17112 | MS-NGA-747 | MS-NFA-17112 | MS-NFA-747 |
|---|---|---|---|---|
| | Hg (mg) | Hg (mg) | Hg (mg) | Hg (mg) |
| SRAT Product | 1106 | 1350 | 154 | 286 |
| MWWT | 6400 | 5505 | 4964 | 4567 |
| MST/SS Dewater | 2.94 | 1.49 | 1.45 | 2.20 |
| SRAT Dewater | 94.4 | 262 | - | - |
| SE Dewater | 123 | 96.5 | 311 | 128 |
| SMECT | 29.5 | 17.3 | 52.5 | 24.0 |
| FAVC | 5.70 | 2.98 | 5.04 | 2.87 |
| **Total** | 7781 | 7238 | 5499 | 5012 |
| **% Accounted For** | 86.4 | 80.3 | 61.1 | 55.7 |

***Off-gas Mercury***

Periodic mercury off-gas samples were drawn from the off-gas line between the SRAT condenser and the ammonia scrubber using a syringe during significant experimental events. The off-gas samples were sparged onto Adsoquick and CarbotrapB columns and analyzed for total mercury and organomercury via DMA. Total mercury (µg/L) and organomercury (µg/L) results from these samples are provided in Table 3-10. Considering the low mercury concentrations (microgram per liter) and the purge rate of 0.165 L/min, mercury in the off-gas is insignificant in respect to the overall mass balance. Organomercury was more prevalent in the experiments with Antifoam 747 (up to 39.4% ). In the experiments with Momentive Y-17112, organomercury did not exceed 4.5%. While this data suggests that total mercury and organomercury are both extremely low in the off-gas stream, changes to the sampling location and improvements to the sampling technique, i.e., continuous mercury monitoring, are warranted for future testing.

**Table 3-10. Off-gas Mercury**

| Experiment | Sample | Total Mercury | Organo Mercury | Organo Mercury |
|---|---|---|---|---|
| | | µg/L | µg/L | % |
| NGA-17112 | MST/SS-1HR | 23.4 | - | - |
| | MST/SS-4HR | 20.5 | - | - |
| | Glycolic-Midpoint | <1 | <0.05 | - |
| | Glycolic-End | 2.16 | 0.085 | 3.94 |
| | SRAT-Dewater | 55.7 | 0.831 | 1.49 |
| | SE-1HR | 5.28 | 0.053 | 1.00 |
| | SE-4HR | <1 | 0.045 | 4.5 |
| | SE-12HR | 85.6 | 0.586 | 0.68 |
| | SE-End | 194 | 1.16 | 0.60 |
| NGA-747 | MST/SS-1HR | 22.4 | - | - |
| | MST/SS-4HR | 42.2 | - | - |
| | Glycolic-Midpoint | 6.5 | 0.367 | 5.65 |
| | Glycolic-End | 6.3 | 0.605 | 9.60 |
| | SRAT-Dewater | 29.4 | 0.127 | 0.43 |
| | SE-1HR | 9.5 | <0.05 | <0.53 |
| | SE-4HR | 4.0 | 0.160 | 4.00 |
| | SE-12HR | 4.7 | 0.185 | 3.94 |
| | SE-End | 8.8 | 3.47 | 39.4 |
| NFA-17112 | MST/SS-1HR | 26.4 | - | - |
| | MST/SS-4HR | 93.0 | - | - |
| | Formic-Midpoint | 28.2 | 0.141 | 0.50 |
| | Formic-End | 29.1 | 0.196 | 0.67 |
| | SE-1HR | 71.7 | 0.038 | 0.05 |
| | SE-4HR | 4.37 | 0.153 | 3.50 |
| | SE-12HR | 40.3 | 0.481 | 1.19 |
| | SE-End | 7.68 | 0.189 | 2.46 |
| NFA-747 | MST/SS-1HR | 8.5 | - | - |
| | MST/SS-4HR | 12.9 | - | - |
| | Formic-Midpoint | 2.4 | 0.830 | 34.58 |
| | Formic-End | 3.7 | <0.05 | <1.35 |
| | SE-1HR | 27.0 | 0.044 | 0.16 |
| | SE-4HR | 0.45 | <0.05 | <11.11 |
| | SE-12HR | <1 | 0.438 | 43.8 |
| | SE-End | 1.81 | 0.436 | 24.09 |

## 4.0 Conclusions

The key conclusions from these experiments are as follows:

- Mercury II Oxide may not be fully reduced to elemental mercury during acid addition at 93°C. Higher temperatures, i.e., boiling may be necessary to fully reduce Mercury II Oxide.

- The highest percent mercury recovery (71 %) in the MWWT was observed in the MS-NGA-17112 experiment (nitric-glycolic acid flowsheet with Momentive Y-17112), which most closely resembles how DWPF is currently operating the SRAT.

- More residual mercury remained in the nitric-glycolic SRAT products (12.3% and 15.0%) in contrast to the nitric-formic SRAT products (1.7% and 3.2 %) despite a higher acid stoichiometry. While more mercury was stripped from the sludge under the nitric-formic flowsheet, less mercury was recovered in the MWWT, and less mercury was accounted for in the overall mercury mass balance. Processing under either the NFA flowsheet or the NGA flowsheet sufficiently removes mercury to the levels needed for processing in the DWPF melter. The mercury proportion of the total solids was below the target of 0.8 wt. % in every SRAT product ranging from 0.083 to 0.28 wt. %.

- While methylmercury was observed in all four experiments, significantly higher concentrations of methylmercury (and trimethylsilanol) were measured in the experiments with Antifoam 747. Trimethylsilanol is an antifoam degradation product of Antifoam 747. The implementation of Momentive Y-17112 significantly reduces the formation and accumulation of methylmercury in the SMECT and condensate streams, as trimethylsilanol is not formed as this defoamer degrades. Other organomercury species (ethylmercury and dimethylmercury) were below method detection limits in all four experiments.

- Initial data suggest that total mercury and organomercury are both extremely low in the off-gas stream. Changes to the sampling location and improvements to the sampling technique, i.e., continuous mercury monitoring, however, are warranted for future testing.

## 5.0 Recommendations

SRNL recommends additional SRAT experiments with a focus on mercury chemistry.[14] It is recommended that future testing be completed only under the nitric-glycolic acid flowsheet utilizing Momentive Y-17112 as the antifoam. It is important to perform these laboratory scale SRAT simulations at prototypic DWPF processing conditions (target heat input, total acid, typical condenser operating conditions, mercury heel, targeting mixing, etc.). To validate the high mercury recovery observed in the inert (nitrogen) purge experiment, additional testing with low and inert purges is advised.[14] Furthermore, experiments with acid addition at higher temperatures, i.e., boiling, are also recommended. In the future, improvements to the mercury mass balance will be achieved by preserving and analyzing all rinses and residues generated during cleanup after each experiment. One of the keys to this future testing is to better understand mercury speciation in the liquid and vapor streams. The use of various analytical methods with a Direct Mercury Analyzer (DMA) and the use of micro-columns to absorb mercury in the off-gas are two techniques that will facilitate a better understanding of the concentration and speciation in these separate streams. Changes to the sampling location and adjustments to the sampling technique will be help improve analytical results for these findings in the future.

## 6.0 References

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# Appendix A. Analytical Data Report – Elemental and Total Mercury Analysis

## Analytical Data Report - Elemental and Total Mercury Analysis

| | | | | |
|---|---|---|---|---|
| **Method:** | **modified EPA 7473 (total mercury)** | | | |
| | using sequential pyrolysis - catalysis - amalgamation - thermal desorption - atomic absorption spectroscopy | | | |
| **Date Analyzed:** | **2/14/2022 - 2/24/2022** | | | |
| **Instrument:** | **Milestone DMA-80** | | | |
| | SN 18032439 | | | |
| | location - 999-1W (ACTL) room 109 | | | |
| **Analyst: Brian B Looney** | **Holly H. VerMeulen** | | **Customer: Anthony Howe** | **Dan Lambert** |
| brian02.looney@srnl.doe.gov | holly.vermeulen@srnl.doe.gov | | anthony.Howe@srnl.doe.gov | dan.lambert@srnl.doe.gov |
| 803 507 4425 (mobile) | 803 522 4373 (mobile) | | 803 295 7716 | 803 819 8466 |
| **QA Check 10 ng** | **9.1** | **ng** | ✓ OK | - 8.5 to 11.5 ng acceptable |

DATA:

| Sample ID | Date analyzed | Concentration (mg/L) | Sample information |
|---|---|---|---|
| **Mercury Data** | | | |
| SiThiol reagent blank | | **0.1** | (ng, df = na, stabilizer = SiThiol) |
| **Elemental Hg** | | | |
| DR-NGA-SLUDGE | 2/14/2022 | 72 | **(sparging of dilute sample onto sorbent; df = 1,000)** |
| DR-NGA-POST-NITRIC | 2/15/2022 | 294 | **(sparging of dilute sample onto sorbent; df = 1,000)** |
| DR-NGA-GLYCOLATE-1 | 2/15/2022 | 54 | **(sparging of dilute sample onto sorbent; df = 1,000)** |
| DR-NGA-GLYCOLATE-2 | 2/15/2022 | 382 | **(sparging of dilute sample onto sorbent; df = 1,000)** |
| DR-NGA-GLYCOLATE-3 | 2/15/2022 | 898 | **(sparging of dilute sample onto sorbent; df = 1,000)** |
| DR-NGA-POST-GLYCOLATE-1 | 2/15/2022 | 1444 | **(sparging of dilute sample onto sorbent; df = 1,000)** |
| DR-NGA-POST-GLYCOLATE-2 | 2/15/2022 | 1624 | **(sparging of dilute sample onto sorbent; df = 1,000)** |
| DR-NGA-PRODUCT | 2/16/2022 | 1561 | **(sparging of dilute sample onto sorbent; df = 1,000)** |
| DR-NFA-SLUDGE | 2/24/2022 | 107 | **(sparging of dilute sample onto sorbent; df = 10,000)** |
| DR-NFA-POST-NITRIC | 2/24/2022 | 161 | **(sparging of dilute sample onto sorbent; df = 10,000)** |
| DR-NFA-FORMATE-1 | 2/24/2022 | 537 | **(sparging of dilute sample onto sorbent; df = 10,000)** |
| DR-NFA-FORMATE-2 | 2/24/2022 | 1787 | **(sparging of dilute sample onto sorbent; df = 10,000)** |
| DR-NFA-FORMATE-3 | 2/24/2022 | 1359 | **(sparging of dilute sample onto sorbent; df = 10,000)** |
| DR-NFA-POST-FORMATE-1 | 2/24/2022 | 1440 | **(sparging of dilute sample onto sorbent; df = 10,000)** |
| DR-NFA-POST-FORMATE-2 | 2/24/2022 | 1341 | **(sparging of dilute sample onto sorbent; df = 10,000)** |

notes:
All concentrations for each species are reported in mg/L (as Hg).
In-vial sparging of dilute solution (10 min; air:water ratio of 50) was used for purging/collection of elemental mercury onto Adsoquick sorbent traps prior to analysis. Traps were then placed directly on DMA carousel and analyzed.
Elemental mercury data/results for original samples are all significantly greater than solubility ( ~ 0.05 mg/L), indicating elemental mercury is present as a separate phase liquid in the original sludge material and all the processed samples. Exceedance of solubility ranges ranges from 1,000x to 50,000x.

# Appendix B. Analytical Data Report – Vapor Samples

## Analytical Data Report - Vapor Sampling to Support ACTL Flowsheet and Process Chemistry Studies

| | | | | | |
|---|---|---|---|---|---|
| Method: | EPA 7473 | (total mercury) | | | |
| | using sequential pyrolysis - catalysis - amalgamation - | | | | |
| | thermal desorption - atomic absorption spectroscopy | | | | |
| Date Analyzed: | 06/20/2022 to 06/22/2022 | | | | |
| Instrument: | Milestone DMA-80 | | | | |
| | SN 18032439 | | | | |
| | location - 999-1W (ACTL) room 109 | | | | |
| Analyst: Brian B Looney | | | Customer: Dan Lambert | | |
| brian02.looney@srnl.doe.gov | | | dan.lambert@srnl.doe.gov | | |
| 803 507 4425 (mobile) | | | 803-646-5614 (mobile) | | |
| 100 ng/mL check std | 100.3 ng/mL | ✓ OK | | | |
| total mercury reagent blank | 0.863 ng | | - adsoquick blank | | |
| organomercury reagent blank | 0.152 ng | | - carbotrapB blank | | |

DATA:

| Sample ID | volume (mL) | raw data (ng of Hg) | blank corrected Hg (ng of Hg) | **Concentration (ug/L as Hg)** | Notes |
|---|---|---|---|---|---|
| **TOTAL Hg** | | | | | |
| **PRFT-1HR** | 1 | 24.25 | 23.387 | **23.4** | ID: MS-NGA-17112 |
| **PRFT-4HR** | 1 | 21.33 | 20.467 | **20.5** | |
| **GLYCOLIC-MIDPOINT** | 0.4 | 0.9316 | 0.0686 | **<1** | recommend 0.5 to 1 mL for future sampling |
| **GLYCOLIC-END** | 0.4 | 1.7264 | 0.8634 | **2.16** | recommend 0.5 to 1 mL for future sampling |
| **DEWATER** | 0.4 | 23.1404 | 22.2774 | **55.7** | recommend 0.5 to 1 mL for future sampling |
| **SEFT-1HR** | 0.4 | 2.9768 | 2.1138 | **5.28** | recommend 0.5 to 1 mL for future sampling |
| **SEFT-4HR** | 0.4 | 0.6528 | -0.2102 | **<1** | recommend 0.5 to 1 mL for future sampling |
| **SEFT-12HR** | 0.4 | 35.0977 | 34.2347 | **85.6** | recommend 0.5 to 1 mL for future sampling |
| **SEFT-END** | 0.4 | 78.5222 | 77.6592 | **194** | recommend 0.5 to 1 mL for future sampling |
| **Organo Hg** | | | | | |
| **GLYCOLIC-MIDPOINT** | 30 | 0.586 | 0.434 | **<0.05** | recommend 50 to 90 mL for future sampling |
| **GLYCOLIC-END** | 30 | 2.712 | 2.560 | **0.085** | recommend 50 to 90 mL for future sampling |
| **DEWATER** | 30 | 25.07 | 24.92 | **0.831** | recommend 50 to 90 mL for future sampling |
| **SEFT-1HR** | 30 | 1.738 | 1.586 | **0.053** | recommend 50 to 90 mL for future sampling |
| **SEFT-4HR** | 30 | 1.489 | 1.337 | **0.045** | recommend 50 to 90 mL for future sampling |
| **SEFT-12HR** | 30 | 17.7362 | 17.5842 | **0.586** | recommend 50 to 90 mL for future sampling |
| **SEFT-END** | **30** | 34.974 | 34.822 | **1.16** | recommend 50 to 90 mL for future sampling |

notes:

All concentrations represent total mercury in the vapor sample and are reported in ng/mL as Hg (= µg/L as Hg)

Samples for total mercury collected on high surface area activated carbon (Adsoquick)

Samples for organo-mercury collected using a silver precolumn (to remove elemental Hg) and a Carbortrap B collection column for selective organo-mercury sorption

All samples blank corrected - Adsoquick reagent blank for total Hg samples and Carbotrap B reagent blanks for organo-mercury

Raw data and results files uploaded to electronic lab notebook: ELN T6751-00351-16

## Analytical Data Report - Vapor Sampling to Support ACTL Flowsheet and Process Chemistry Studies

| | | | | | |
|---|---|---|---|---|---|
| Method: | EPA 7473 | (total mercury) | | | |
| | using sequential pyrolysis - catalysis - amalgamation - | | | | |
| | thermal desorption - atomic absorption spectroscopy | | | | |
| Date Analyzed: | 07/18/2022 to 07/20/2022 | | | | |
| Instrument: | Milestone DMA-80 | | | | |
| | SN 18032439 | | | | |
| | location - 999-1W (ACTL) room 109 | | | | |
| Analyst: Brian B Looney | | | Customer: Dan Lambert | | |
| brian02.looney@srnl.doe.gov | | | dan.lambert@srnl.doe.gov | | |
| 803 507 4425 (mobile) | | | 803-646-5614 (mobile) | | |
| 100 ng/mL check std | 113 ng/mL | ✓ OK | | | |
| total mercury reagent blank | 0.48 ng | | - adsoquick blank | | |
| organomercury reagent blank | 1.59 ng | | - carbotrapB blank | | |

DATA:

| Sample ID | volume (mL) | raw data (ng of Hg) | blank corrected Hg (ng of Hg) | **Concentration (ug/L as Hg)** | Notes |
|---|---|---|---|---|---|
| **TOTAL Hg** | | | | | |
| **PRFT-1HR** | 0.5 | 11.67 | 11.19 | **22.4** | ID: MS-NGA-747 |
| **PRFT-4HR** | 0.5 | 21.56 | 21.08 | **42.2** | |
| **GLYCOLIC-MIDPOINT** | 0.5 | 3.74 | 3.26 | **6.5** | |
| **GLYCOLIC-END** | 0.5 | 3.63 | 3.15 | **6.3** | |
| **DEWATER** | 0.5 | 15.18 | 14.7 | **29.4** | |
| **SEFT-1HR** | 0.5 | 5.22 | 4.74 | **9.5** | |
| **SEFT-4HR** | 0.5 | 2.48 | 2 | **4.0** | |
| **SEFT-12HR** | 0.5 | 2.84 | 2.36 | **4.7** | |
| **SEFT-END** | 0.5 | 4.86 | 4.38 | **8.8** | |
| **Organo Hg** | | | | | |
| **GLYCOLIC-MIDPOINT** | 50 | 19.930 | 18.34 | **0.367** | |
| **GLYCOLIC-END** | 50 | 31.860 | 30.27 | **0.605** | |
| **DEWATER** | 50 | 7.94 | 6.35 | **0.127** | |
| **SEFT-1HR** | 50 | 1.030 | -0.56 | **<0.05** | |
| **SEFT-4HR** | 50 | 9.590 | 8.00 | **0.160** | |
| **SEFT-12HR** | 50 | 10.84 | 9.25 | **0.185** | |
| **SEFT-END** | 50 | 175.3 | 173.71 | **3.47** | |

notes:

All concentrations represent total mercury in the vapor sample and are reported in ng/mL as Hg (= µg/L as Hg)

Samples for total mercury collected on high surface area activated carbon (Adsoquick)

Samples for organo-mercury collected using a silver precolumn (to remove elemental Hg) and a Carbortrap B collection column for selective organo-mercury sorption

All samples blank corrected - Adsoquick reagent blank for total Hg samples and Carbotrap B reagent blanks for organo-mercury

Raw data and results files uploaded to electronic lab notebook: ELN T6751-00351-16

## Analytical Data Report - Vapor Sampling to Support ACTL Flowsheet and Process Chemistry Studies

| | | | | | |
|---|---|---|---|---|---|
| Method: | EPA 7473 (total mercury) | | | | |
| | using sequential pyrolysis - catalysis - amalgamation - | | | | |
| | thermal desorption - atomic absorption spectroscopy | | | | |
| Date Analyzed: | 07/21/2022 to 07/22/2022 | | | | |
| Instrument: | Milestone DMA-80 | | | | |
| | SN 18032439 | | | | |
| | location - 999-1W (ACTL) room 109 | | | | |
| Analyst: Brian B Looney | | | Customer: Dan Lambert | | |
| brian02.looney@srnl.doe.gov | | | dan.lambert@srnl.doe.gov | | |
| 803 507 4425 (mobile) | | | 803-646-5614 (mobile) | | |
| 100 ng/mL check std | 113 ng/mL | ✓ OK | | | |
| total mercury reagent blank | 0.48 ng | | - adsoquick blank | | |
| organomercury reagent blank | 1.59 ng | | - carbotrapB blank | | |

DATA:

| Sample ID | volume (mL) | raw data (ng of Hg) | blank corrected Hg (ng of Hg) | **Concentration (ug/L as Hg)** | Notes |
|---|---|---|---|---|---|
| **TOTAL Hg** | | | | | |
| **total_Hg_prft_1_hr** | 0.5 | 13.7 | 13.2 | **26.4** | ID: MS-NFA-17112 |
| **total_Hg_prft_4_hr** | 0.5 | 47.0 | 46.5 | **93.0** | |
| **total_Hg_formic_@_midpoint** | 0.5 | 14.6 | 14.1 | **28.2** | |
| **total_Hg_formic_@_end** | 0.5 | 15.0 | 14.5 | **29.1** | |
| **total_Hg_seft_@_1_hr** | 0.5 | 36.3 | 35.9 | **71.7** | |
| **total_Hg_seft_@_4_hr** | 0.5 | 2.67 | 2.19 | **4.37** | |
| **total_Hg_seft_@_12_hr** | 0.5 | 20.6 | 20.1 | **40.3** | |
| **total_Hg_seft_@_end** | 0.5 | 4.32 | 3.84 | **7.68** | |
| **Organo Hg** | | | | | |
| **organo_Hg_formic_@_midpoint** | 50 | 8.66 | 7.07 | **0.141** | |
| **organo_Hg_formic_@_end** | 50 | 11.4 | 9.79 | **0.196** | |
| **organo_Hg_seft_@_1_hr** | 50 | 3.48 | 1.89 | **0.038** | |
| **organo_Hg_seft_@_4_hr** | 50 | 9.23 | 7.64 | **0.153** | |
| **organo_Hg_seft_@_12_hr** | 50 | 25.6 | 24.0 | **0.481** | |
| **organo_Hg_seft_@_end** | 50 | 11.0 | 9.44 | **0.189** | |

notes:

All concentrations represent total mercury in the vapor sample and are reported in ng/mL as Hg (= µg/L as Hg)

Samples for total mercury collected on high surface area activated carbon (Adsoquick)

Samples for organo-mercury collected using a silver precolumn (to remove elemental Hg) and a Carbortrap B collection column for selective organo-mercury sorption

All samples blank corrected - Adsoquick reagent blank for total Hg samples and Carbotrap B reagent blanks for organo-mercury

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## Analytical Data Report - Vapor Sampling to Support ACTL Flowsheet and Process Chemistry Studies

| | | | | | |
|---|---|---|---|---|---|
| Method: | EPA 7473 | (total mercury) | | | |
| | using sequential pyrolysis - catalysis - amalgamation - | | | | |
| | thermal desorption - atomic absorption spectroscopy | | | | |
| Date Analyzed: | 9/8/2022 | | | | |
| Instrument: | Milestone DMA-80 | | | | |
| | SN 18032439 | | | | |
| | location - 999-1W (ACTL) room 109 | | | | |
| Analyst: Brian B Looney | | | Customer: Dan Lambert | | |
| brian02.looney@srnl.doe.gov | | | dan.lambert@srnl.doe.gov | | |
| 803 507 4425 (mobile) | | | 803-646-5614 (mobile) | | |
| 100 ng/mL check std | 80.1 ng/L | ✓ OK | | | |
| total mercury reagent blank | 1.32 ng | | - adsoquick blank | | |
| organomercury reagent blank | 0.27 ng | | - carbotrapB blank | | |

DATA:

| Sample ID | volume (mL) | raw data (ng of Hg) | blank corrected Hg (ng of Hg) | **Concentration (ug/L as Hg)** | Notes |
|---|---|---|---|---|---|
| **TOTAL Hg** | | | | | |
| **Total_HG_PRFT_@_1_hr** | 0.5 | 5.59 | 4.27 | **8.5** | ID: MS-NFA-747 |
| **TOTAL_HG_PRFT_@_4_hr** | 0.5 | 7.79 | 6.47 | **12.9** | |
| **TOTAL_Hg_FORMIC_@_mid** | 0.5 | 2.50 | 1.18 | **2.4** | |
| **TOTAL_Hg_FORMIC_@_end** | 0.5 | 3.18 | 1.86 | **3.7** | |
| **Total_HG_SEFT_@_1_hr** | 0.5 | 14.84 | 13.52 | **27.0** | |
| **TOTAL_HG_SEFT_@_4_hr** | 0.5 | 1.54 | 0.22 | **0.45** | |
| **TOTAL_Hg_SEFT_@_12_hr** | 0.5 | 1.28 | -0.04 | **< 1** | |
| **TOTAL_Hg_SEFT_@_end** | 0.5 | 2.22 | 0.90 | **1.81** | |
| **Organo Hg** | | | | | |
| **ORGANO_Hg_FORMIC_@_mid** | 50 | 41.8 | 41.5 | **0.830** | |
| **ORGANO_Hg_formic_@_end** | 50 | 0.93 | 0.66 | **<0.05** | |
| **ORGANO_Hg_SEFT_@_1_hr** | 50 | 2.49 | 2.22 | **0.044** | |
| **ORGANO_Hg_SEFT_@_4_hr** | 50 | 1.10 | 0.83 | **<0.05** | |
| **ORGANO_Hg_SEFT_@_12_hr** | 50 | 22.2 | 21.9 | **0.438** | |
| **ORGANO_Hg_SEFT_@_end** | 50 | 22.1 | 21.8 | **0.436** | |

notes:

All concentrations represent total mercury in the vapor sample and are reported in ng/mL as Hg (= µg/L as Hg)

Samples for total mercury collected on high surface area activated carbon (Adsoquick)

Samples for organo-mercury collected using a silver precolumn (to remove elemental Hg) and a Carbortrap B collection column for selective organo-mercury sorption

All samples blank corrected - Adsoquick reagent blank for total Hg samples and Carbotrap B reagent blanks for organo-mercury

Raw data and results files uploaded to electronic lab notebook: ELN T6751-00351-16

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