Mercury Speciation of Hanford 241-AP-105 Tank Waste Samples – 21108

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ABSTRACT

Understanding the movement and fate of mercury, and its chemical consequences are important in the Hanford flowsheet. Necessary inputs to evaluate the impact of mercury include the magnitude of the mercury feed inventory, how the inventory is distributed throughout the tank farms, how the tank-by-tank mercury speciation inventory is distributed by waste phases (i.e., sludge, saltcake, and supernatant), the expected mercury species during waste processing, and finally, the degree of uncertainty in this information. Based on process knowledge, an estimated inventory of 2,100 kg of mercury is assumed to be distributed in varied amounts across 149 single-shell tanks (SSTs) and 28 double-shell tanks (DSTs) at the Hanford Site. Historically, the limited analyses of individual SSTs and DSTs for total mercury were below method detection, yielding upper limits in concentration estimates based solely on method detection limits (MDLs), resulting in an uncertain mercury inventory for Hanford tanks. Recently, Pacific Northwest National Laboratory (PNNL) researchers provided the first glimpse into the concentration of total and elemental mercury in 241-AP-107 (hereafter referred to as AP-107) tank waste feed and AP-107 treated effluents. Highly sensitive separation methods for total, elemental, and monomethyl mercury species, in tank waste, developed at PNNL's Radiochemical Processing Laboratory, and non-radiological environmental methods to quantify mercury species have been adapted for application to Hanford tank wastes and waste at other sites. This method (separation and quantification), which has been previously demonstrated at PNNL to measure AP-107 waste, was applied to four sample types from Hanford waste tank 241-AP-105 (hereafter referred to as AP-105) and designated as raw, feed, Mott grade 5 filtered samples, and cesium-decontaminated samples. These samples represent feed inventory and pretreatment process effluents prior to immobilization by vitrification. Predicting and tracking mercury speciation and associated inventories mitigates risks associated with compliance with the mercury-to-sodium ratio waste acceptance criterion for the Hanford Waste Treatment and Immobilization Plant, secondary waste disposal land disposal restriction requirements, and air permit abatement assumptions during the Direct Feed Low-Activity Waste (DFLAW) mission and beyond. This work directly aides in the understanding of mercury movements, chemical consequences, and the fate of mercury in the Hanford flowsheet, enabling engineers to reliably anticipate and control the mass movements of various mercury species through the Hanford waste processing flowsheet, thereby reducing flowsheet risk and providing reference-case data to compare to actual future DFLAW operations.

INTRODUCTION

The U.S. Department of Energy Hanford Site in southeastern Washington State stores 56 million gallons of chemical and radioactive waste in underground tanks. Mercuric nitrate, $Hg(NO_3)_2$, was introduced into the Hanford flowsheet when it was used to catalyze nitric acid dissolution of certain aluminum alloy fuels and to suppress radioiodine volatilization during nitric acid dissolution of short-cooled uranium metal fuel [1]. As a result, based primarily on Hanford process knowledge, the tank waste stored at Hanford is estimated to contain a total mercury inventory of 2,100 kg [1]. Accurate and reliable chemical speciation and quantification is needed to anticipate, predict, and abate the mass movements of various mercury species through the Hanford waste processing flowsheet. From a Hanford waste processing flowsheet perspective (both currently in DFLAW and in the future for high-level waste streams), the fate of mercury is highly dependent on liquid, solid, and gas phase mercury as dictated by the chemical and thermal conditions of the various waste processing unit operations. These assumed flowsheet model stream partitions and associated mercury species remain largely unverified by analyses of samples from representative processes using actual Hanford tank waste.

An analytical methodology to accurately determine total mercury and mercury speciation in Hanford tank waste, while maintaining low mercury detection limits, was previously developed and demonstrated at the Pacific Northwest National Laboratory (PNNL) Marine and Coastal Research Laboratory (MCRL; formerly Marine Sciences Laboratory, MSL) in Sequim, Washington [2,3]. Bottenus et al. offered the first mercury speciation of its type for Hanford tank 241-AP-107 (hereafter referred to as AP-107) [3]. This effort set out to expand on the development of this methodology and further broaden the existence of analytical data relative to the Hanford tank mercury speciation while leveraging work already being conducted with the Radioactive Test Platform[1] (RTP) in the PNNL Radiochemical Processing Laboratory (RPL). This paper focuses on representative samples of four separate supernatant sample types, originating from the raw tank waste and lab-scale waste processing unit operations of Hanford tank 241-AP-105 (hereafter referred to as AP-105) tank waste. Each sample is preserved, separated, and quantified for total, elemental, and monomethyl mercury (hereafter referred to as methyl mercury) content. Both a highly sensitive separation method developed at PNNL's RPL and the complimentary non-radiological environmental quantification method demonstrated at PNNL's MCRL are utilized to illustrate successful mercury speciation at low mercury detection limits. The following sections will discuss the sample types, the experimental and analytical methods, and the analytical results.

HANFORD TANK WASTE SAMPLE COLLECTION AND PRESERVATION

This work used raw supernatant, diluted feed, and treated samples of AP-105 tank waste obtained for and treated by PNNL's RTP. The RTP includes representative lab-scale waste processing unit operations to demonstrate filtration, cesium-decontamination by ion exchange (IX), and immobilization by vitrification. In the current RTP configuration, the composited AP-105 diluted feed was filtered using a Mott grade 5 sintered metal dead-end filter and then cesium decontaminated via IX using crystalline silicotitanate (CST) media [4,5]. Four sample types were obtained and designated as raw, feed, filtered (or filtrate), and cesium-decontaminated (or post-IX) for subsequent analytical separation and quantification of total, elemental, and methyl mercury content. Each AP-105 sample type was subsampled and preserved into individual aliquots for analyses of mercury content as described in TABLE I.

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[1] PNNL's Radioactive Test Platform uses 4- to 12-L tank waste sample volumes to provide lab-scale demonstrations of filtration, cesium IX, and low-activity waste (LAW) melter performance representative of baseline (as well as alternatives) treatment operations during the DFLAW mission phase at Hanford.

TABLE I. Hanford AP-107 tank samples analyzed for mercury.

AP-105 Source Sample Type	**Mercury Analysis**	**Parent Sample Volume (mL)**	**Preserved/Diluted Sample Volume (mL)**
Raw	Total Hg	0.2	5
	Elemental Hg	0.8	5
	Methyl Hg	0.8	5
Feed	Total Hg	2.3	8
	Elemental Hg	1.6	12
	Methyl Hg	3.1	8
Filtrate	Total Hg	2.2	8
	Elemental Hg	1.6	12
	Methyl Hg	3.0	8
Post-IX	Total Hg	1.1	8
	Elemental Hg	1.2	12
	Methyl Hg	1.9	8

Highly sensitive separation methods for total, elemental, and methyl mercury species in tank waste were employed at the RPL. Non-radiological environmental mercury quantification methods were adapted to quantify mercury species in Hanford tank samples by leveraging an existing non-radiological, trace mercury detection technique performed at the PNNL MCRL. Each mercury species requires specific preservation, preparation, separation, transportation, and analysis processes. These are briefly described in the following sections since the current analysis implemented a few improvements based on recommendations from the previous years' work. Additional details can be found in [3] and [6].

Samples for total mercury analysis need to be preserved with a strong oxidant to liberate mercury from organic complexes and particulates prior to preconcentration, separation, and analysis ([6]; MSL-I-013).[2] To accomplish this, the samples are preserved and simultaneously digested (or oxidized) using bromine monochloride (BrCl) reagent. The total mercury sample must be treated with BrCl for a minimum of 12 hours prior to separation and preconcentration, after which the sample can be stored at room temperature for up to 6 months per EPA Method 1631 guidance [6]. Additionally, all preserved samples were stored in light-limiting conditions. Two adaptations were made from the total mercury preservation methods discussed in [3]. In this work, (1) BrCl was used to convert all mercury species to ionic (Hg^{2+}) mercury immediately and (2) the headspace was no longer minimized after all the mercury was converted to Hg^{2+}. Previous work in [2,3] used concentrated hydrochloric acid (HCl) to preserve the total mercury sample and minimized headspace to limit loss of gaseous elemental mercury. Then prior to separation, BrCl was used to convert all the mercury species in the preserved solution to Hg^{2+}. The previous preservation technique was altered due to the potential for greater loss of elemental mercury to the headspace if mercury is not converted to Hg^{2+} until after storage. TABLE I provides the final preserved sample volumes for total mercury analysis sample types.

Samples for elemental mercury analysis were preserved by minimizing headspace while maintaining in situ speciation. Without proper storage, elemental mercury can volatilize into the headspace during extended sample storage, reaching an equilibrium between the dissolved liquid content and gaseous content, and then be released from the sample upon opening.

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[2] MSL-I-013. *Total Mercury in Aqueous Samples by Cold Vapor Atomic Fluorescence (CVAF)*. Marine Sciences Laboratory Standard Operating Procedure MSL-I-013, Rev. 14. 2019. Document is not publicly available.

Therefore, preservation of elemental mercury samples only included the minimization of headspace using high-purity deionized water (>18 MΩ of known low mercury content). Additionally, all preserved samples were stored in light limiting conditions. The final volume of the sample preserved for elemental mercury analysis is given in TABLE I.

Unlike total mercury samples, samples for methyl mercury analysis cannot be exposed to a strong oxidant that could destroy the organomercuric bond. Therefore,, the addition of concentrated HCl was used to decrease sample pH to ≤ 2, stabilizing the methyl mercury in solution [8]. Acidification of the sample is a necessary preparation step for distillation, which creates a less complex matrix for preconcentration. Lastly, the sample is brought to a known volume using high-purity deionized water (>18 MΩ of known low mercury content) and stored in light-limiting conditions. The identical preservation steps were used in [3]. The final volume of the sample preserved for methyl mercury analysis is given in TABLE I.

RPL SEPARATION PROTOCOL

A goal of this work was to maximize mercury detection ability through minimal sample dilution while also reducing the radioactive sample dose prior to mercury quantification at MCRL. It was necessary to establish that the mercury separation processes at RPL were efficient and effective at managing and mitigating radiological risk associated with the raw tank waste supernatant feed sample. Demonstration of the mercury separation processes and radiological dose measurements for various components was documented previously [2,3]. These results were instrumental for anticipating the safe execution of the full process composed of separations at RPL, sample transport, and quantification at MCRL. Several performance checks of the mercury separation system constructed in RPL were implemented to provide continued quantification of the system components and process.

It was previously demonstrated that methyl mercury is most stable for transport (e.g., between RPL and MCRL) in the distillate form based on stability experiments [3]. Additionally, the efficiency of the separation processes (distillation and purging) at RPL was initially established in the earlier work and re-evaluated in this study using mercury standards. For example, elemental and total mercury measurements rely on purging onto gold-coated sand columns using an inert gas. Determining and confirming the efficiency of this separation and ensuring the tank waste sample size did not saturate the gold-coated bead traps were essential to validate measurements of actual tank waste mercury. While the separation of methyl mercury requires a distillation step, the mercury recovered in the distillate can vary depending on the separation system used. The distillation efficiency factor was determined for the RPL methyl mercury separation system and factored into final quantification results. With RPL separation performance determined, the final mercury separation and quantification protocol for Hanford tank waste samples was established and implemented with the raw, feed, filtered, and cesium-decontaminated samples as described below.

Total mercury was isolated by purging the mercury species from the aqueous samples (specified for total mercury analysis) onto a gold-coated sand substrate using a bubbler system as depicted in Fig. 1. Prior to separation, the sample is brought to a known volume using high-purity deionized water (>18 MΩ of known low mercury content) and treated with hydroxylamine hydrochloride ($NH_2OH{\cdot}HCl$) to destroy excess BrCl. Replicate aliquots are added to a bubbler system containing a low concentration of stannous chloride ($SnCl_2$) in water, which reduces the inorganic Hg^{2+} species present to elemental Hg^0. The elemental species is then purged from solution with nitrogen gas at a rate of 350 mL/min onto a gold-coated sand trap inside of a contamination area (CA) fume hood. At this point, the column is known to have a minimum radiological content based on prior total and gamma activity measurements [2]. The gold-coated sand trap is then removed from the CA fume hood and shipped in a chilled cooler to MCRL for mercury quantification using cold vapor atomic fluorescence spectrometry (CVAFS).

The aqueous samples for preconcentration of elemental mercury from the aqueous sample (specified for elemental mercury analysis) were purged, shipped, and quantified in the same manner, though in separate bubblers and with the exception of the $SnCl_2$ addition in the bubbler.

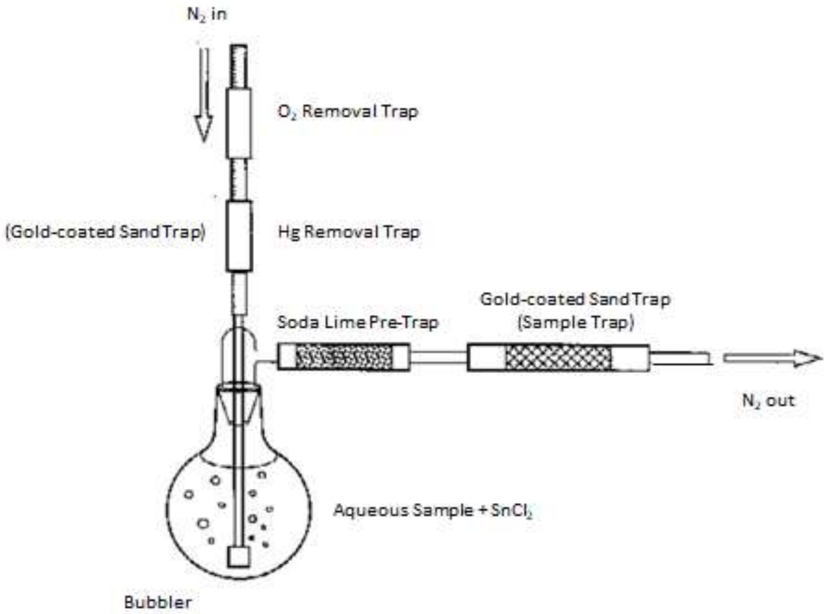

Fig. 1. System schematic for total and elemental mercury purging. No $SnCl_2$ is added to the bubbler for elemental mercury measurements (figure reproduced from EPA Method 1631[6]).

Based on earlier investigations of the stability of methyl mercury samples, (i.e., as a distillate sample derived from the parent sample, or as methyl mercury stripped from solution followed by ethylation to methylethyl mercury and captured on a Carbotrap®[3]-filled trap), the methyl mercury samples degraded less when transported in the liquid distillate form versus captured on the Carbotrap®-filled trap. To minimize degradation, the isolation of methyl mercury species is conducted as a two-step process: (1) distillation of the sample at RPL, followed by (2) preconcentration of the methyl mercury species onto a Carbotrap® substrate at MCRL. At RPL, the preserved sample is first distilled in a CA fume hood to decrease the presence of other constituents, including other mercury species, to produce a less complex matrix and reduce the radiological background (Fig. 2). The distillate is then surveyed out of the CA fume hood and shipped in a chilled cooler to MCRL for preconcentration and quantification of methyl mercury species using CVAFS. Based on prior total and gamma activity measurements, the distillate solution is considered to have minimal radiological content [2] and can be more safely shipped and handled at MCRL.

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[3] Carbotrap is a registered trademark of Sigma Aldrich Co. LLC.

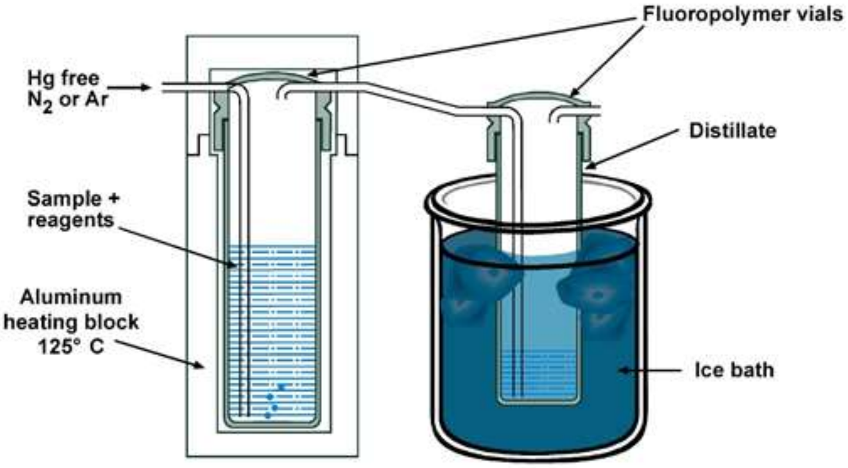

Fig. 2. System schematic for methyl mercury distillation (figure reproduced from EPA Method 1630 [8]).

MSL SAMPLE PRECONCENTRATION AND QUANTIFICATION

The CVAFS system for total and elemental mercury analysis consists of a two-stage gold amalgamation gas train to introduce elemental mercury vapor into the gas cell of an atomic fluorescence spectrometer [12]. A schematic of the layout of this system is given in Fig. 3. For quantification, mercury collected onto a gold-coated bead trap was placed in the “field column” position (far left gold trap in Fig. 3). The mercury collected is transferred to the “analytical column” (far right gold trap in Fig. 3) by releasing elemental mercury into the argon carrier gas stream using controlled heating (>450 °C). The elemental mercury is adsorbed onto the analytical column, where it re-amalgamates. The mercury on the analytical column is then released by controlled heating and swept into the gas cell of the atomic fluorescence detector, where its signal is recorded.

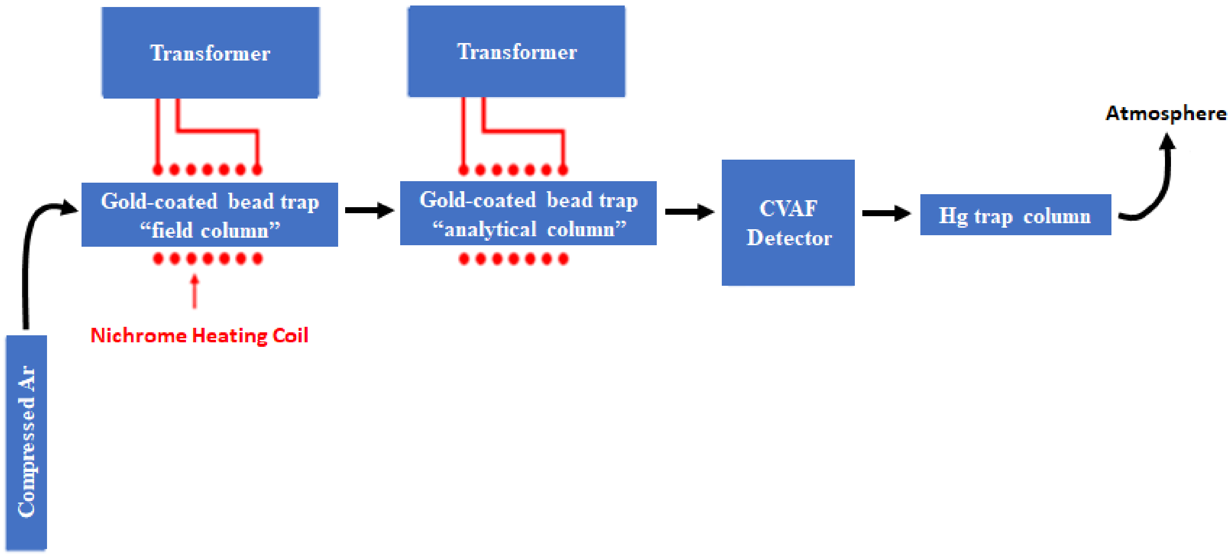

Fig. 3. Two-stage gold amalgamation gas train for introducing elemental mercury into an atomic fluorescence detector for quantification of mercury collected on a gold-coated sand trap.

For analytical preconcentration at MCRL, replicates of the methyl mercury distillate (received at MCRL from RPL) are transferred into a glass bubbler system containing a dilute acetate buffer (Fig. 4). Tetraethyl borate is then added as an ethylating reagent to the bubbler and allowed to react with the sample to convert methyl mercury to a methylethyl mercury, a volatile species. The volatile species is then purged from solution using N_2 at a rate of 200 mL/min and trapped onto a Carbotrap®-filled trap ([8]; MSL-I-014).[4]

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[4] *MSL-I-014. Methylmercury in Aqueous Samples by Cold Vapor Atomic Fluorescence (CVAF).* Marine Sciences Laboratory Standard Operating Procedure MSL-I-014, Rev 10. 2019. Document is not publicly available.

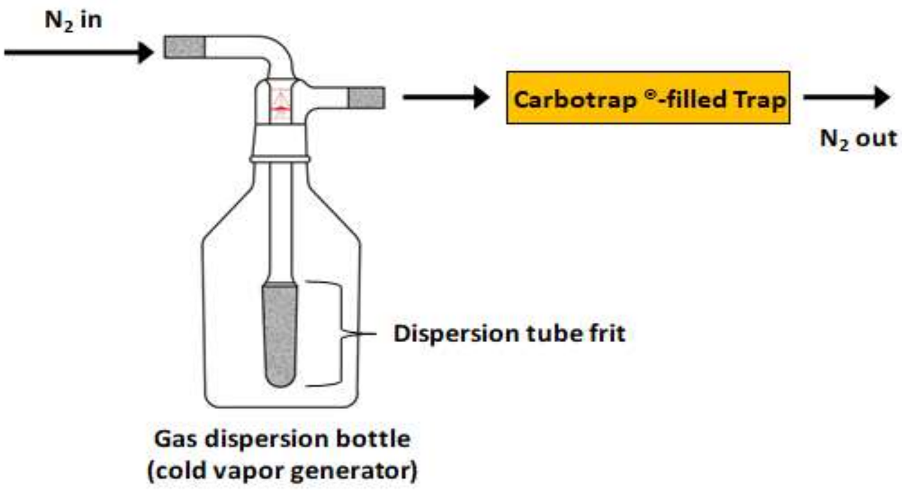

Fig. 4. System schematic for methylethyl mercury purging. (Carbotrap is a registered trademark of Sigma Aldrich Co. LLC.)

The CVAFS system for methyl mercury analysis consists of a two-step chromatographic adsorbent column followed by a pyrolysis column to introduce elemental mercury vapor into the gas cell of an atomic fluorescence spectrometer. A schematic of the layout of this system is given in Fig. 5. For quantification, methyl mercury (in the form of methylethyl mercury) collected on a Carbotrap®[5]-filled trap is placed in the nichrome heating coil and desorbed from the Carbotrap®-filled trap into the argon carrier gas stream using controlled heating (450 - 500 °C), separated in a chromatographic adsorbent column (held at 120 °C), converted to elemental mercury via a pyrolysis column (~700 °C), and then quantified using CVAFS, where its signal is recorded (Fig. 5).

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[5] Carbotrap is a registered trademark of Sigma Aldrich Co. LLC.

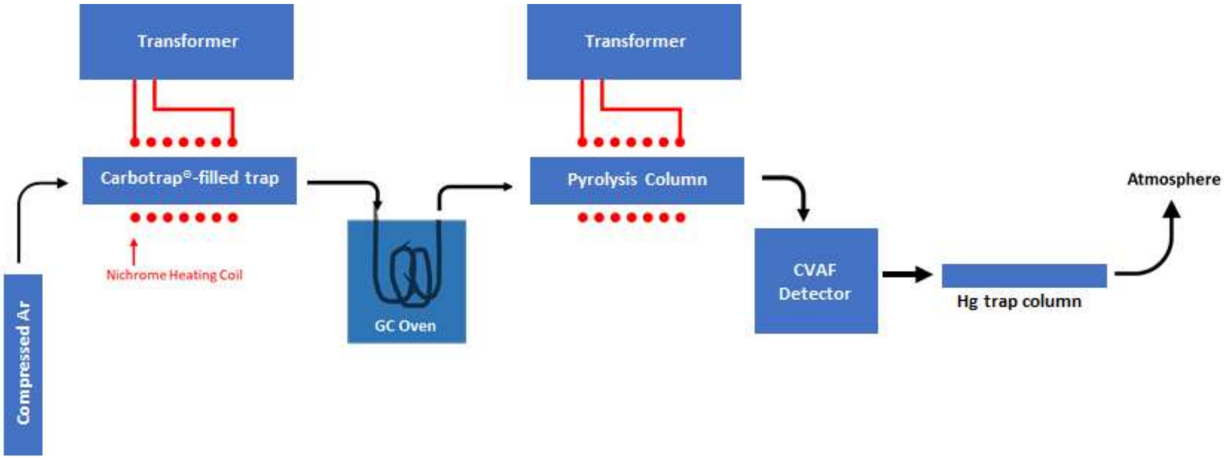

Fig. 5. Schematic of system used to quantify methyl mercury collected on a Carbotrap®-filled trap by ballistic heating, GC separation, pyrolysis of methylethyl mercury to elemental mercury, and detection of elemental mercury by CVAF.

The three mercury species were successfully quantified for AP-105 for all four source sample types (raw, feed, filtrate, and post-IX). The AP-105 raw sample material is a concentrated supernatant (~8.5M Na); it was then diluted with raw river water (1.56 dilution factor, [9]) to achieve the desired target sodium molar concentration thus becoming the AP-105 feed sample. The feed was then filtered using a Mott Model 6610 media grade 5 (i.e., filter collects 90% of particle ≥5 µm) sintered metal dead-end filter supplying the filtrate sample, and finally cesium-decontaminated via IX using CST media to generate the post-IX sample. The results from the determination of mercury content are listed in TABLE II. Included in these results are the parent source mercury concentrations for the four sample types; due to the raw river dilution that took place between the raw and feed sample, the pretreatment sample (feed, filtrate, and post-IX) results would necessitate a dilution factor (i.e., 1.56) adjustment to be comparable to the raw samples.

Total mercury parent samples were diluted to a known volume just prior to purging. This resulted in an approximate 6.3- to 10-fold dilution of elemental mercury samples, 3.5- to 25.3-fold dilution of total mercury samples, and 2.5- to 6.3-fold dilution of methyl mercury samples. Replicates were analyzed for mercury species and reported in TABLE II along with estimated parent sample concentrations, correcting for the original aliquot volume and sample dilution. Initially smaller parent sample volumes were prepared for the methyl mercury speciation of the AP-105 raw samples but resulted in peaks below detection. After these initial samples, parent sample volumes were increased for the later methyl mercury samples (feed, filtered, and post IX) to allow for successful quantification. This study was the first to quantify methyl mercury values in tank waste as previous AP-107 methyl mercury analysis attempts encountered instrument problems, preventing quantification.

TABLE II. Determination of elemental, total, and methyl mercury in Hanford tank AP-105 waste.

AP-105 Source Sample Type	Mercury Analysis	Volume of Parent Sample Analyzed (mL)	Parent Source MDL (ng/L)[a]	Parent Sample Mercury Concentration (ng/L)[b]
Raw	Total Hg	0.2	24.0	115[d]
	Elemental Hg	0.8	5.9	49[d]
	Methyl Hg	0.8	3.0	NPD[d]
Feed[c,f]	Total Hg	2.3	2.1	22
	Elemental Hg	1.6	3.0	6[e]
	Methyl Hg	3.1	0.8	<0.8
Filtrate[c,f]	Total Hg	2.2	2.2	30
	Elemental Hg	1.6	3.0	<3.0
	Methyl Hg	3.0	0.8	3
Post-IX[c,f]	Total Hg	1.1	4.3	35
	Elemental Hg	1.2	4.0	<4.0
	Methyl Hg	1.9	1.3	4[e]

(a) Sample-specific MDL is scaled to determine the minimum mercury concentration that can be detected in the parent source and determined based on the method described by 40 CFR Part 136, Appendix B, rev 1.11.
(b) Concentrations based on the average of duplicate sample results except where noted.
(c) Due to the raw river water dilution (with Columbia River water) that took place between the raw and feed sample, the pretreatment sample concentrations (feed, filtrate, and post-IX) presented are as-measured and do not account for any prior dilution by raw river water.
(d) Values were based on triplicate measurements.
(e) Values were based on single measurement which was greater than MDL.
(f) Herger et al. (2017) [11] reported total mercury concentration of Columbia River water to be between 0.500 and 1.900 ng/L. It is estimated that the dilution water contributed between 0.5% and 3% of the total mercury reported in tank waste sample concentrations based on the reported river water quality.
< refers to sample quantification is less than MDL.
NPD = no peak detected.

The concentrations of total mercury were determined, through duplicate or triplicate measurements, in the raw, feed, filtered, and post-IX AP-105 parent samples to be 115, 22, 30, and 35 ng/L, respectively. Elemental mercury content was determined, through duplicate measurements, in raw, feed, filtrate, and post-IX samples of AP-105 Hanford tank waste supernatant, averaging 49, 6, <3 and <4 ng/L, respectively. Elemental mercury measurements conducted on sample aliquots were all low and some were at or near the MDL compared to the total mercury content of respective sample types. Methyl mercury content was determined, through duplicate measurements, in feed, filtrate, and post-IX samples of AP-105 Hanford tank waste supernatant, averaging < 0.8, 3, and 4 ng/L, respectively. Measurements of methyl mercury in AP-105 raw samples were below detection, and thus could not be quantified due to the small sample size. Across all mercury species, the sample pretreatments (i.e., filtration and cesium decontamination) do not appear to influence mercury concentration. When both species were quantified, elemental mercury in the raw and feed waste forms is estimated to comprise an average of 36% of the total mercury in the AP-105 tank waste, while the elemental mercury in the treated waste forms (filtered or filtered and cesium-decontaminated) is estimated to comprise an average of 10% of the total mercury in the AP-105 tank waste. Future determination of elemental mercury should increase the parent sample size that is purged onto the gold-coated sand column to increase detection capability.

The quantification of elemental and total mercury in Hanford tank waste from 241-AP-107 raw, filtered and post-IX samples are summarized in Bottenus et al. [3] Concentrations of total mercury are higher in AP-107 than in AP-105 across all sample types [3]. However elemental mercury comprised an average of only 2.4% of the total mercury in raw and treated Hanford waste from AP-107 across all sample types (raw, filtered, or filtered and cesium-decontaminated). Additionally, in this set of samples the total mercury concentrations demonstrated that the trend of decreasing with each processing step, suggesting that processing of tank waste, by filtration and ion exchange, results in removal of substantial mercury from the waste stream, which is ultimately beneficial in meeting secondary waste disposal requirements. It is not clear at this time if the retained mercury is associated only with filtered solids, some other removal mechanism like plating on surfaces, or a combination of both. Similarly, the reduction in total mercury concentration due to CST ion exchange could be both from solids filtration and sorption capacity. The sample pretreatments (i.e., filtration and IX) did not appear to influence the elemental mercury concentrations in the AP-107 samples. Cumulatively, comparing the trends from the previous AP-107 mercury analysis and the current AP-105 mercury analysis demonstrates differences in tank concentrations and in speciation across supernatant waste found in the Hanford tank complex.

CONCLUSION

The direct measurement of mercury species in Hanford tank waste samples from AP-105 was completed. Previous approaches for analyzing tank waste samples at other U.S. Department of Energy sites used large dilution factors (often 100,000-fold or greater) to reduce radiological background to levels that would permit safe handling and sample analysis. Our work has demonstrated that quantitative results can be obtained from Hanford tank waste samples using ultra-trace environmental mercury techniques for the analysis of mercury from small sample sizes with much less dilution. Both expanding the existence of analytical data of total, elemental, and methyl mercury to include Hanford tank waste sample types from AP-107, AP-105, and their respective treated effluents, and demonstrating that once the samples are isolated from the parent tank waste, they can be shipped and quantified in a low-level radiological facility. Ultimately, comparison with the previous data suggests distinct variability in total mercury and mercury speciation between waste tanks.

REFERENCES

1. K. J. KUPFER, A. L. BOLDT, K. M. HODGSON, L. W. SHELTON, B. C. SIMPSON, R. A. WATROUS, M. D. LECLAIR, G. L. BORSHEIM, R. T. WINDWARD, B. A. HIGLEY, R. M. ORME, N. G. COLTON, S. L. LAMBERT, D. E. PLACE, and W. W. SCHULZ, *Standard Inventories of Chemicals and Radionuclides in Hanford Site Tank Wastes*, HNF-SD-WM-TI-740, Rev. 0C, Lockheed Martin Hanford Corporation, Richland, WA (1999). https://www.hanford.gov/files.cfm/HNF-SD-WM-TI-740_Rev_0C.pdf

2. M. S. FOUNTAIN, S. D. BRANCH, and G. A. GILL, *Separation and Speciation of Mercury from Hanford 241-AP-107 Tank Waste Supernatant*, PNNL-28067, Pacific Northwest National Laboratory, Richland, WA (2018). https://www.osti.gov/servlets/purl/1489836

3. C. L. H. BOTTENUS, M. S. FOUNTAIN, S. D. BRANCH, G. A. GILL, and J. R. WOOD, *Mercury Speciation and Quantification of Hanford 241-AP-107 Tank Waste Feed and Treated*

Samples, PNNL-29555, Rev. 0; RPT-OSIF-005, Rev. 0, Pacific Northwest National Laboratory, Richland, Washington (2019).

4. K. CRAMER, *Mercury Pathway and Treatment Assessment for the WTP,* 24590-WTP-RPT-PR-01-011, Rev. 0, Bechtel National, Inc., Richland, Washington (2001).
5. R. W. GOLES, W. C. BUCHMILLER, L. K. JAGODA, B. D. MACISAAC, and S. RASSAT, *Test Summary: WTP Flowsheet Evaluation of Mercury-Containing Hanford Waste Simulant,* PNWD-3437; WPT-RPT-122, Rev. 0, Battelle—Pacific Northwest Division, Richland, Washington (2004).
6. C. L. H. BOTTENUS, K. A. MULLER, S. D. BRANCH, M. S. FOUNTAIN, C. R. MYERS, K. M. MUNSON, and G. A. GILL, "Mercury Speciation and Quantification of Hanford Tank Waste Samples from 241-AP-105, 241-AP-107, and 241-AN-102," Attachment to *Mercury Speciation and Quantification of Hanford Tank Waste Samples from 241-AP-105, 241-AP-107, and 241-AN-102*." PNNL-SA-158076, Pacific Northwest National Laboratory, Richland, Washington (2020).
7. U.S. EPA, Method 1631, Revision E, *Mercury in water by oxidation, purge and trap, and cold vapor atomic fluorescence spectrometry*, U.S. Environmental Protection Agency, Washington, D.C. (2002).
8. U.S. EPA, Method 1630, *Methyl mercury in water by distillation, aqueous ethylation, purge and trap, and CVAFS*, U.S. Environmental Protection Agency, Washington, D.C. (1998).
9. J. R. ALLRED, J. G. H. GEETING, A. M. WESTESEN, E. C. BUCK, and R. A. PETERSON, *Fiscal Year 2020 Filtration of Hanford Tank Waste 241-AP-105*, PNNL-30485, Rev. 0; RPT-DFTP-021, Rev. 0, Pacific Northwest National Laboratory, Richland, Washington (2020).
10. 40 CFR Part 136, Appendix B, "Definition And Procedure For The Determination Of The Method Detection Limit—Revision 1.11," *Code of Federal Regulations* (2011).
11. L. HERGER, L. EDMOND L, and G. HAYSLIP, *Mid-Columbia River Fish Toxics Assessment EPA Region 10 Report*, EPA-910-R-17-002, U.S. Environmental Protection Agency, Seattle, Washington (2017). https://www.epa.gov/sites/production/files/2017-03/documents/mid-columbia-river-fish-toxics-assessment-march2017.pdf
12. W. F. FITZGERALD and G. A. GILL, *Subnanogram determination of mercury by two-stage gold amalgamation and gas phase detection applied to atmospheric analysis*. Anal. Chem. 51: 1714-1720 (1979).