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Evaluation of Hanford Tank Supernatant Availability for Technetium Management Project Studies in FY16

BM Rapko

September 2015

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Prepared for
the U.S. Department of Energy
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Pacific Northwest National Laboratory
Richland, Washington 99352

Executive Summary

This report examines the need for actual Hanford tank waste solutions to support tasks in the Technetium Management Program in fiscal year (FY) 2016. One key need is to identify both samples where a majority of the soluble technetium is present as pertechnetate and samples where it is not. The total amount of tank supernatant needed from any given tank waste supernatant was determined by polling the tasks leaders for their technology testing needs in FY16 and then arbitrarily ascribing a 10% process loss associated with consolidation and the Cs-137 removal needed to reduce the dose to a level suitable for testing in radiological fumehoods. These polling results identified a need for approximately 2.1 to 3.6 kg of any particular targeted Hanford tank waste supernatant.

It is assumed that the requested tank waste would come from archived samples currently stored at the Hanford Site's 222-S facility. Further, only tank samples removed from Hanford tanks in the last 5 years are deemed useful in order to minimize potential changes in Tc speciation as a result of extended storage. Such archived samples would be delivered to the Radiochemical Processing Laboratory (RPL) facility at Pacific Northwest National Laboratory (PNNL), where Cs-137 removal and subsequent subdividing of the tank samples would be performed before sending the aliquots to the Technetium Management Program task leads who requested samples.

Hanford tanks were identified that contain large fractions of both soluble Tc that are present either as pertechnetate or that are present as a non-pertechnetate species. Next, an inventory of samples currently stored at 222-S was obtained and the identified candidate tanks mapped against sample availability.

The results of this exercise indicate that only one tank waste supernatant dominant in soluble, non-pertechnetate technetium (AN-102, 2013 sampling event) and one tank waste supernatant dominant in soluble technetium as pertechnetate (AZ-102, either 2012 or 2014 sampling event) meet all above-mentioned criteria. Therefore, the recommendation is that at least 3.6 kg of AN-102 tank supernatant from the 2013 sampling event and all available AZ-102 tank supernatant from either the 2012 or 2014 sampling event be transferred from 222-S facility to the RPL at PNNL for FY16 testing.

Finally, efforts were made to ascertain whether suitable containers for such a transfer were currently available and whether the infrastructure (equipment, current procedures, etc.) was in place to package, ship, receive, and unpack the samples. Time limitations prevented some of these questions from being answered, but it should be noted that many similar transfers of Hanford tank supernatants from 222-S to RPL have been done in the past.

Acknowledgements

This work was completed as part of the Technetium Management Hanford Site project. Support for this project came from the U.S. Department of Energy's Office of Environmental Management.

The author would like to thank MS Wilburn for editorial assistance and RJ Serne for his technical review.

Acronyms and Abbreviations

FY	fiscal year (October 1 through September 30, e.g., FY 2015 ends on September 30, 2015)
LAW	low-activity waste
PNNL	Pacific Northwest National Laboratory
QA	quality assurance
R&D	research and development
RPL	Radiochemical Processing Laboratory
SG	specific gravity
TCP	Tank Characterization Program
TIC	total inorganic carbon
TOC	total organic carbon
WRPS	Washington River Protection Solutions
WWFTP	WRPS Waste Form Testing Program

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1.0 Introduction

As described in the Technetium Management Project Test Plan (TP-EMSP-0018),¹ Tc-99 (Tc) generated from the fission of ²³⁵U and ²³⁹Pu in high yields is a Hanford Nuclear Reservation tank waste constituent. Among radioactive constituents in the tank waste, Tc presents a unique challenge in that it has a long half-life ($\beta = 292$ keV; $T_{1/2} = 2.11 \times 10^5$ y) and exists predominantly in the liquid phase, generally in the anionic form of pertechnetate TcO_4^- , which is highly volatile at low-activity waste (LAW) vitrification melter temperatures and mobile in the Hanford Site's subsurface environment. This makes immobilization of Tc into high durability waste forms a critical technical challenge for nuclear waste management. The complex behavior of Tc under storage, treatment, and immobilization conditions significantly affects its management options, which to date remain uncertain.

To address the management challenges associated with the treatment, immobilization, and disposal of technetium in tank wastes, this project examines a number of technical challenges, including separation, disposition, and detection. A Test Plan describing a scope of study in fiscal year (FY) 2015 designed to address these technical challenges has been prepared (TP-EMSP-0018). The Test Plan breaks down the scope of study into seven tasks; Task 1 is to identify candidate Hanford tank waste supernatants for use in the other tasks as needed.

Specifically, as noted in Section 2.1 of the Technetium Management Project Test Plan,¹ Task 1 seeks to identify candidate Hanford tank supernatants for technology testing under the Technetium Management Project beginning in FY16, determine their availability, and verify that the logistical aspects of shipping are in place for transfer from the Hanford Site's building 222-S to the Pacific Northwest National Laboratory (PNNL) Radiochemical Processing Laboratory (RPL). This report responds to this Task 1 scope.

2.0 Quality Assurance

This work was conducted as part of PNNL Project 54042 under the Technetium Management Program, with funding from the U.S. Department of Energy Office of Environmental Management.

All research and development (R&D) work at PNNL is performed in accordance with PNNL's laboratory-level Quality Management Program, which is based on a graded application of NQA-1-2000, *Quality Assurance Requirements for Nuclear Facility Applications*, to R&D activities. In addition to the PNNL-wide quality assurance (QA) controls, the QA controls of the Washington River Protection Solutions (WRPS) Waste Form Testing Program (WWFTP) QA program were also implemented for the work. The WWFTP QA program consists of the WWFTP Quality Assurance Plan (QA-WWFTP-001) and associated QA-NSLW-numbered procedures that provide detailed instructions for implementing NQA-1 requirements for R&D work. The WWFTP QA program is based on the requirements of NQA-1-2008, *Quality Assurance Requirements for Nuclear Facility Applications*, and NQA-1a-2009, *Addenda to ASME NQA-1-2008 Quality Assurance Requirements for Nuclear Facility Applications*, graded on the approach presented in NQA-1-2008, Part IV, Subpart 4.2, "Guidance on Graded

¹ Levitskaia TG and DJ McCabe. 2015. *Technetium Management – Hanford Site (FY 2015)*. TP-EMSP-0018, Rev 0.0, Pacific Northwest National Laboratory, Richland, WA.

Application of Quality Assurance (QA) for Nuclear-Related Research and Development.” Preparation of this report was assigned the technology level “Applied Research” and was conducted in accordance with procedure QA-NSLW-1102, *Scientific Investigation for Applied Research*. All staff members contributing to the work have technical expertise in the subject matter and received QA training prior to performing quality-affecting work. The “Applied Research” technology level provides adequate controls to ensure that the activities were performed correctly. Use of both the PNNL-wide and WWFTP QA controls ensured that all client QA expectations were addressed in performing the work.

3.0 Discussion

Section 3.1 of TP-EMSP-0018 gives three criteria for supernatant selection: quantity, availability, and technetium speciation. These criteria can be summarized by the following questions:

1. Quantity: Is there sufficient material to support the three tasks charged with performing testing on/with actual Hanford tank waste supernatant in FY16? The three tasks requiring this material are Task 2, sensor development for n-Tc (alkaline soluble non-per technetate technetium or n-Tc); Task 3, Tc treatment and removal from LAW; and Task 6, non-glass waste forms for Tc immobilization.
2. Availability: Is the material available? Addressing this criterion involves confirming that the desired tank supernatant has not been reserved for other purposes.
3. Technetium speciation: Is there good reason to believe that at least one candidate tank supernatant has a substantial fraction of the soluble technetium not present as per technetate?

To this can be added a fourth criterion:

4. How will the identified samples be packaged at 222-S building, transported from 222-S to the RPL, and received at the RPL and stored before use? Addressing this criterion involves working with Hanford Site and PNNL shipping and the RPL’s Shielded Facility Operations staff to verify that a mechanism exists to get the shipped containers into the RPL hot cells.

These criteria were addressed as follows. First, through WRPS, the current inventory of tank waste samples stored at the 222-S building as of summer 2015 was obtained. Next, it was assumed that only materials collected from those tanks that have been previously examined for their speciation of soluble technetium should be considered. Of these candidate materials, it was assumed that at least one tank supernatant should be from a tank in which the majority of its alkaline-soluble technetium is not in the form of per technetate and one supernatant should be from a tank where the majority of its soluble technetium is in the form of per technetate. Finally, the amount of tank supernatant needed was determined and mapped to the available tank supernatant inventory.

Next, the amount of material needed for FY16 testing was estimated by polling the leaders of Tasks 2, 3, and 6 and totaling their individual requirements. Once the specific inventory items corresponding to the needed tank supernatants had been identified, it was noted whether there were flags indicating that the samples were reserved for other activities. Finally, attempts were made to ascertain the availability of shipping containers and the presence of qualified sample shippers and receivers.

4.0 Results

A recent report by Serne et al. (2014, Table 4.9), and references therein, summarizes the published determinations of the fraction of alkaline soluble n-Tc in various tank supernatants. This table is the basis for selecting tanks most likely to have large fractions of n-Tc as well as tanks most likely to have minimal to no n-Tc present. It should be noted that this analysis assumes that the tank supernatant to be tested by each task should come from the same source (i.e., double-shell tank and sampling event). It also assumes that a relatively recently sampled supernatant should be used. For this tank supernatant candidate selection, “relatively recent” is defined as having been sampled no earlier than FY10 (roughly no longer than 5 years ago).

Inspection of Table 4.9 in Serne et al. 2014 reveals five tank supernatants, each with multiple measurements/estimates of the n-Tc/pertechnetate ratios, where half or more of the soluble Tc is present as n-Tc. These tanks are AN-102, AN-107, AP-104, SY-101, and SY-103. Two tanks were examined where little to no measurable n-Tc is present (AZ-101 and AZ-102), although there is one outlier measurement for AZ-102 that reports 33% n-Tc against three measurements that indicate <1% n-Tc. Thus, this first screening reduced the list of candidate tank supernatant to only seven tanks.

Next, the required amount of supernatant anticipated for the FY16 studies noted above was estimated and the availability of supernatant from these seven tanks noted. A polling of the relevant FY15 EMSP Tc Management task leaders led to the following estimates of needs for any Hanford tank supernatant: from Task 2 (SA Bryan), 50 to 100 mL; from Task 3 (TG Levitskaia), 1.0 to 1.5 L; and from Task 6 (W Um), 0.5 to 1 L; for a total requested amount of 1.6 to 2.6 L. As noted below, the typical density of the candidate Hanford tank supernatants is 1.25 g/mL, so the total mass needed (the inventory amounts are provided in kilograms) is 1.9 to 3.3 kg. In addition, Cs-137 removal before testing is highly desirable; if a 10% loss during that processing step is assumed,² a total target test sample of 2.1 to 3.6 kg per tank type is needed.

WRPS supplied a spreadsheet containing the current tank waste inventory from the Tank Characterization Program (TCP) stored at 222-S in early August of 2015. The inventory list provides information about source tank(s), sampling year, type of sampling, form (sludge, saltcake, liquid), and core ID (if applicable). Thus, the spreadsheet (inventory list) provides the information needed to determine which tank samples are available. It also notes whether the sample is currently reserved for other programmatic use.

The inventory list was first reduced to the seven candidate tanks noted above: AN-102, AN-107, AP-104, AZ-101, AZ-102, SY-101, and SY-103. Inspection of the inventory list reveals that the amount of supernatant needed highly restricts the sources of tank supernatant available and all but eliminates any archived sources of salt cake from selection. Availability of saltcake was included so that the use of dissolved saltcake as a backup to actual tank supernatant could be evaluated.

Table 1 summarizes the results from the TCP inventory archived at 222-S. We were particularly focused on the timing of the sampling event and restricted our focus to only events that occurred after 2010. For AN-102, there were two sampling events: one in 2012 and one in 2013. AN-107 had one

² Rapko et al. (2003) describe Cs-137 decontamination from dissolved Hanford Tank saltcake. Using data from the report and the original run notes, a 5% loss of feed from the operation is calculated.

sampling event (in 2010), AP-104 had no sampling events over the period in question, AZ-101 had one sampling event (in 2010), and AZ-102 had two sampling events (one in 2012 and one in 2014). In addition, there was an AZ-101/AZ-102 composite sampling event in 2012, the nature of which was not investigated further.

As mentioned above, sample mass inventories significantly restrict the possibilities. With an identified need of 2.1 to 3.6 kg of sample, adequate amounts of tank supernatant are available only for the 2013 sampling (approximately 6 kg) of AN-102 supernatant and either the 2012 sampling (2.1 kg) or the 2014 sampling (2.5 kg) of AZ-102 supernatant.

Table 1. Sample Availability Table for Liquid Tank Wastes AN-102, AN-107, AP-104, AZ-101, AZ-102, SY-101, and SY-103

Tank Waste	Name	Weight (g)	ID	Matrix
AN-102	2AN-12-01 DUP	97	S14T016411	CL
AN-102	2AN-12-01	39.6	S12T005502	GL
AN-102	2AN-12-02	301.9	S12T005519	GL
AN-102	2AN-12-03	23.7	S12T005521	GL
AN-102	2AN-12-04	72.2	S12T005530	GL
AN-102	2AN-12-06	74.8	S12T005556	GL
AN-102	2AN-12-07	86.9	S12T005565	GL
Total		696		
AN-102	2AN-13-01	546	S13T007060	LIQUID
AN-102	2AN-13-02	546.9	S13T007063	LIQUID
AN-102	COMP(2013)	118.3	S13R000408	GL
AN-102	COMP(2012)	55	S12T008945	GL
AN-102	COMP(2012)	105.9	S12T008946	GL
AN-102	2AN-13-04	535.4	S13T007071	GL
AN-102	2AN-13-05	284.8	S13T007076	GL
AN-102	2AN-13-06	265.5	S13T007080	GL
AN-102	2AN-13-03A	655.3	S13T007069	GL
AN-102	2AN-13-03B	651.7	S13T007070	GL
AN-102	2AN-13-04A	650.4	S13T007074	GL
AN-102	2AN-13-04B	660.3	S13T007075	GL
AN-102	2AN-13-05A	660.5	S13T007076	GL
AN-102	2AN-13-03	538.4	S13T007066	GL
Total		6274		
Total - COMP		5995		
AN-107	AN-107-10-1	215.8	S10T003006	GL
AN-107	AN-107-10-1DUP	336.5	S10T003027	DL
AN-107	AN-107-10-3DUP	99.1	S10T003030	GL
AN-107	AN-107-10-3	69.9	S10T003029	GL
AN-107	AN-10	136.6	S10T003031	GL
Total		858		

Tank Waste	Name	Weight (g)	ID	Matrix
AP-104	NONE REPORTED	190.1	S07T001300	GL
Total		190		
AZ-101	AZ-101-10-6	7.2	S10T000431	GL
Total		7.2		
AZ101/AZ102	NONE REPORTED	529.5	S12R000152	LIQUID
AZ101/AZ102	NONE REPORTED	547	S12R000152	LIQUID
AZ101/AZ102	NONE REPORTED	549.5	S12R000152	LIQUID
Total		1626		
AZ-102	2AZ-12-01	112.1	S12T014345	GL
AZ-102	2AZ-12-01A	245.2	S12T014346	GL
AZ-102	2AZ-12-01DUP	144.5	NONE	GL
AZ-102	2AZ-12-02	216.5	S12T014368	GL
AZ-102	2AZ-12-03	126.5	S12T014370	GL
AZ-102	2AZ-12-03A	228.9	NONE	GL
AZ-102	2AZ-12-04	143.6	S12T014418	GL
AZ-102	2AZ-12-01DUP	137	S12T014429	GL
AZ-102	2AZ-12-06	130.1	S12T014451	GL
AZ-102	2AZ-12-07	145.3	S12T014440	GL
AZ-102	2AZ-12-08	151.9	S12T014473	GL
AZ-102	2AZ-12-09	166.5	S12T014484	GL
AZ-102	2AZ-12-010	156	S12T014495	GL
Total		2104		
AZ-102 EVAP	2AZ-14-01FB	142.9	S14T002802	GL
AZ-102 EVAP	2AZ-14-01	230.1	S14T002898	GL
AZ-102 EVAP	2AZ-14-02	303.3	S14T002902	GL
AZ-102 EVAP	2AZ-14-03DUP	216.2	S14T002919	GL
AZ-102 EVAP	2AZ-14-03A	195.9	S14T002930	GL
AZ-102 EVAP	2AZ-14-03B	306.4	S14T002936	GL
AZ-102 EVAP	2AZ-14-03C	306.5	S14T002935	GL
AZ-102 EVAP	2AZ-14-04A	269.9	S14T024358	GL
AZ-102 EVAP	2AZ-14-05A	275.9	S14T024371	GL
AZ-102 EVAP	2AZ-14-06	251.4	S14T024373	GL
Total		2499		
SY-101	NONE	-	-	-
Total		NA		
SY-103	-	82	S13T005351	DL
SY-103	-	237.5	S13T005350	DL
Total		320		

GL = grab sample liquid; DL = drainable liquid; CL = centrifuged liquid.

Note that the name and ID both record the year information (after S in the ID and after the tank farm information in the name).

Based only on having a measured n-Tc/pertechnetate ratio and sufficient sample availability, the list of preferred candidate tank supernatants can be reduced to two: the 2013 supernatant sampling of the high n-Tc-containing AN-102, and either the 2012 or 2014 samplings from the low n-Tc-containing AZ-102.

5.0 Selected Characterization Data for the Seven Candidate Tank Supernatants

Following requests by task leaders in the Technetium Management Project for selected characterization information about the candidate tank supernatants, a query was made to the Best Basis Inventory for characterization information from the initial seven candidate tank supernatants. The requested information included the supernatant's Sr, Tc-99, free hydroxide, Pd, Pt, Ru, and Rh concentrations; the supernatant's total organic carbon (TOC) and total inorganic carbon (TIC); and the supernatant's measured specific gravity (SG). The average values from multiple measurements are provided in Table 2.

There is little soluble strontium in these tank supernatants; the reported measurements are of total Sr rather than Sr-90 because of the lack of Sr-90 measurements for most of these samples. Few to none of the noble metals Pd, Pt, Ru, and Rh are detected. TOC and TIC concentrations vary widely, with TOC being more than an order of magnitude greater in AN-102 than AZ-102 and TIC in AN-102 also being greater, albeit to a lesser extent. Finally, AN-102 has a much greater specific gravity than in AZ-102.

Table 2. Summary of Requested Tank Waste Information Network System Tank Data

Tank	Year	[Sr], μg/mL	SG	TOC, μg/mL	TIC, μg/mL	Tc-99, μg/mL	Cs-137, μCi/mL	[OH], μg/mL	[Pd], μg/mL	[Pt], μg/mL	[Rh], μg/mL	[Ru], μg/mL
AN-102	2012	< 6	1.446	30,900	13,650	8.089	287	6659	< 200	No Data	< 100	< 60
AN-102	2013	NA	NA	22,233	12,400	NA	NA	9969	NA	No Data	< 100	22
AN-107	2010	< 10	1.421	36,533	15,683	5.576	278	12591	< 100	No Data	< 50	31***
AP-104	No sample	-	-	-	-	-	-	-	-	-	-	-
AZ-101	2010	< 1.5	1.232	533	8,513	20.083	1285	8483	66*	No Data	< 25	< 15
AZ-102	2012	< 1	1.136	476	4,262	4.284	247	6108	6	No Data	6*	4*
AZ-102	2014	< 1	NA	986	6,220	NA	NA	10577	< 12	No Data	< 9	4**
SY-101	No sample	-	-	-	-	-	-	-	-	-	-	-
SY-103	No sample	-	-	-	-	-	-	-	-	-	-	-

* Based on the average of all actual measured values (less than measurements ignored).

** Only one data point, all others <4.

*** Only one data point, all others <30.

6.0 Logistical Aspects

An attempt was made to evaluate the readiness of the infrastructure for the packaging and shipment of targeted Hanford tank supernatants from 222-S and their receipt, unpackaging, and storage until needed at the RPL. Key questions included the availability and current readiness of suitable packaging for the shipment of these radioactive solutions, and the status of operating procedures to allow packaging of the tank supernatants at 222-S and their unpackaging and safe storage in the RPL hot cells.

No progress beyond the initial queries was made. However, such shipments have been fairly routine over the years and it is deemed likely that the documentation is current and in place for such sample transfers. The major unresolved question is the availability of suitable certified shipping containers.

7.0 Conclusions

This report was prepared to identify the best available tank waste supernatants for planned FY16 testing under the auspices of the Technetium Management Program. Both samples containing large fractions of supernatant-soluble Tc present as pertechnetate and samples containing soluble, non-pertechnetate Tc were desired. Determining the ratio of non-pertechnetate Tc to Tc present as pertechnetate requires specific, non-standard measurements. These measurements have only been performed with a few Hanford tank supernatants. Consideration of this subset of tank waste supernatants led to candidate supernatants from seven tanks: AN-102, AN-107, AP-104, AZ-101, AZ-102, SY-101, and SY-103. Considering the availability of recently collected samples from these tanks reduced the targeted candidates to two: the high n-Tc-containing supernatant from the 2013 sampling of AN-102 and the low n-Tc-containing supernatant from the 2012 sampling or the 2014 sampling of AZ-102.

Since tank waste samples, both sludges and solutions, are shipped from 222-S to the RPL, no difficulties in receiving these tank waste supernatants are expected, although an attempt to verify the presence and readiness of suitable shipping containers and the existence of a current, in-place infrastructure to perform such shipments was not verified but should be completed prior to any specific shipment scheduling.

8.0 References

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