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HANFORD HIGH LEVEL WASTE: SAMPLE EXCHANGE/EVALUATION (SEE) PROGRAM

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The Pacific Northwest Laboratory (PNL)/Analytical Chemistry Laboratory (ACL) and the Westinghouse Hanford Company (WHC)/Process Analytical Laboratory (PAL) provide analytical support services to various environmental restoration and waste management projects/programs at Hanford. In response to a U. S. Department of Energy - Richland Field Office (DOE-RL) audit, which questioned the comparability of analytical methods employed at each laboratory, the Sample Exchange/Exchange (SEE) program was initiated.

The SEE Program is a self-assessment program designed to compare analytical methods of the PAL and ACL laboratories using site-specific waste material. The SEE program is managed by a collaborative, the Quality Assurance Triad (Triad). Triad membership is made up of representatives from the WHC/PAL, PNL/ACL, and WHC Hanford Analytical Services Management (HASM) organizations. The Triad works together to design/evaluate/implement each phase of the SEE Program.

In Phase I of the program, various radiochemical methods were compared. Hanford tank-waste core-material fusion preparations were chosen for the initial sample exchange. Fusion preparations from both laboratories were exchanged and analyzed for a pre-determined suite of radiochemical analyses. The data were then compiled and evaluated by the Triad. Results from the first exchange revealed several noteworthy discrepancies in the methods evaluated and produced some lessons learned that were used to improve th- second phase of the program.

The following radiochemical

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procedures were evaluated during Phase I:

Total Alpha
Total Beta
^{239}Pu and ^{240}Pu
^{241}Am
^{90}Sr
^{99}Tc
^{137}Cs
Uranium (Total)

Eight sample and two blank fusion extracts along with one "blind" quality control (QC) sample were submitted from the ACL to the PAL. Four sample, two blank fusion extracts, and six QC samples were submitted from PAL to the ACL. Each sample, blank, and QC sample was analyzed once for the applicable analyses listed above. Results from each analysis were compiled by each laboratory and submitted to the Triad for final compilation and data evaluation.

The test plan used to initiate Phase I set a control limit of 20% relative percent difference (RPD). Each result was compared to its counter and evaluated based on the 20% criteria. Results that fell outside the evaluation criteria were investigated further for possible method discrepancy. After an extensive review of the data, three analyses were found to have comparative values that consistently fell outside the set evaluation criteria. Total-alpha and total-beta values reported by PAL were > 20% higher than those reported by the ACL. Almost all of the total-uranium results fell outside the 20% criteria; however, there was no pattern for the differences (i.e., neither laboratory had a consistent bias).

Further investigation into each of the outlying analyses provided demonstrable differences in the methods employed. For total-beta, it was found that the ACL calibrates its instruments using ^{90}Sr. The PAL uses ^{60}Co to calibrate its total-beta instruments. Because of the differences in efficiency of these two isotopes, a predictable difference could be calculated between the two laboratories. Once this correction factor was applied to the results, all the results were found to be well within the evaluation criteria of 20% RPD.

For total alpha, an instrumentation problem was discovered at PAL. The total-alpha counters used at PAL were found to have a slow electronic gate, which was used to filter out any beta cross-talk. The gate remained open long enough to allow erroneous beta activity to be counted as Alpha activity, thus producing total-alpha results higher than expected.

The evaluation of the total-uranium results was not as conclusive as the evaluations for total-alpha and total-beta. No significant root cause could be discovered for the differences in the analytical results. The most probable explanation is that methods employed at both laboratories (which are very similar) have a precision greater than 20% RPD.

Several lessons learned during Phase I were applied to improve the second phase of the program. In Phase I, only duplicate fusion aliquots were

analyzed by each participating laboratory. This provided relatively few data points with which to evaluate each laboratory's analytical precision. Phase II was modified to include submission of four aliquots of the same preparation to each laboratory. This will allow for both a comparability analysis and a better determination of each laboratory's analytical precision, as well as a cursory determination of the analytical error associated with each method evaluated.

Phase I included the analysis of blind QC samples; however, only one analysis was performed on each blind sample, so only an estimate of method accuracy could be performed. Phase II includes the analysis of four replicates of each blind QC sample submitted by each participating laboratory. Each blind QC sample represented a limited suite of target analyses. Results from the analysis of the blind QC samples will provide a better basis for determining the accuracy and precision of each method evaluated.

A final modification made to Phase II was the addition of both acid-digest and water-leach sample preparations. The water leach preparations allow for the expansion of the target list to include ^{3}H, ^{14}C, total organic carbon/total inorganic carbon/total carbon (TOC/TIC/TC), cyanide (CN), and ion-chromatography (IC) anions. The acid-digest samples will be used to evaluate inductively coupled plasma (ICP) metals analyses. As an additional cross-check of the ICP-metals, ^{99}Tc, and uranium (total) analyses, we have submitted four aliquots of each appropriate sample preparation to the PNL, Chemical Sciences Department, Advanced Inorganic Section for ICP-mass spectrometry (MS) analysis. The ICP-MS method is capable of determining most of the same metals as ICP-optical, as well as providing radiochemical information (for long-lived isotopes).

The SEE Program has provided important insight into the comparability of the radiochemical methods employed at the ACL and PAL. As a continuing self-assessment program, SEE will provide improved data quality and comparability for all analyses conducted at each participating laboratory. As well as improving the quality operations at each laboratory, the SEE program can serve as a model for integrated/multi-organization management of key inter-elemental programs. The SEE Program has therefor proven to be a win-win solution to evaluate and improve laboratory operations at Hanford.