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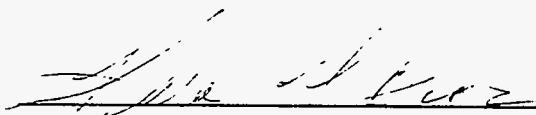
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**CALCINATION/DISSOLUTION CHEMISTRY DEVELOPMENT
FISCAL YEAR 1995**

EXECUTIVE SUMMARY

An investigation into the chemistry of alkaline Hanford Site tank waste (TTP RL4-3-20-04) was conducted in Fiscal Year (FY) 1995 at the Westinghouse Hanford Company under the support of the Efficient Separations and Processing Crosscutting Program (EM-53). The investigation had two main subtasks: liaison with the Institute of Physical Chemistry of the Russian Academy of Science and further laboratory testing of the chemistry of calcination/dissolution (C/D or thermal reconstitution) of Hanford Site tank waste.

Progress, which was achieved in the calcination/dissolution subtask in FY 1995, is summarized in this report as follows.

1. Tests were run comparing the performances of C/D and enhanced sludge washing using genuine wastes from tanks 104-S (a REDOX Process sludge containing large quantities of solid phase aluminum hydroxide), 101-SY (a complexant concentrate with high chromium concentration), and 102-SY (a high-transuranics [TRU], high-chromium, Plutonium Finishing Plant waste). The tests showed that markedly improved dissolutions of aluminum from tank 104-S and tank 102-SY wastes were achieved by C/D. Comparative tests for chromium-rich wastes from tanks 101-SY and 102-SY showed greater than 99 percent chromium dissolutions were obtained by C/D but only 5 percent and 21 percent dissolutions, respectively, by enhanced sludge

washing. Tests planned for waste from tank 110-U (a mixed aluminum cladding, REDOX process and bismuth phosphate process waste) were not performed because of budget reallocations to the Institute of Physical Chemistry. However, chemical and radionuclide analyses of tank 110-U waste were performed.

2. A mild peroxide oxidative treatment to dissolve chromium hydroxide under enhanced sludge washing conditions was tested with material simulating the composition of tank 101-SY waste and with genuine wastes from tanks 101-SY and 102-SY. The alkaline peroxide treatment dissolved approximately 65 percent of the chromium from the simulant waste. Negligible enhancement in chromium dissolution over the reference-enhanced sludge washing flowsheet was found for the peroxide treatment in tests with the tank wastes.
3. Tests to determine the speciation of plutonium and neptunium solubilized by C/D showed dissolved Pu(V) and Np(V) hydroxide complexes are produced. A Np(V) peroxide complex also was identified.
4. Reduction/coprecipitation methods to remove plutonium and neptunium solubilized by C/D were tested and shown to be rapid and efficient.
5. Recently published data on plutonium (IV) carbonate complexation in moderately alkaline (pH 12 to 13) solution led to a reexamination of previous investigations of Pu complexation by carbonate in highly alkaline (3 to 5 molar NaOH) solutions. The

comparisons showed Pu(IV) carbonate complexes may also exist in the highly alkaline solution. Visible/near infrared spectra of the suspected Pu(IV) carbonate complex were obtained.

6. Technical chemistry support was provided to calciner kinetics tests at the University of Idaho.

7. A technical report on progress in studies of the chemistry of C/D was prepared and issued. A midyear report covering both the liaison and C/D studies also was written and issued. Contributions were made to a technical presentation given at the 19th Annual Actinide Separations Conference on the solubility of plutonium compounds in alkaline solution. A paper on C/D chemistry has been submitted and accepted for presentation at the "Tank Waste Chemistry" symposium to be held during the 211th national meeting of the American Chemical Society in March 1996.

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LIST OF TERMS

C/D	calcination/dissolution
DOE	U.S. Department of Energy
ESW	Enhanced Sludge Washing
FY	fiscal year
HLW	high-level waste
IPC	"Institute of Physical Chemistry"
IPC/RAS	Institute of Physical Chemistry of the Russian Academy of Science
LLW	low-level waste
Tc	technetium
TRU	Transuranic
TTP	technical task plan
WHC	Westinghouse Hanford Company

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1.0 INTRODUCTION

The task "IPC Liaison and Chemistry of Thermal Reconstitution" is a \$300,000 program that was conducted in Fiscal Year (FY) 1995 with U.S. Department of Energy (DOE) Office of Research and Development (EM-53) Efficient Separations and Processing Crosscutting Program supported under technical task plan (TTP) RL4-3-20-04. The principal investigator was Cal Delegard of the Westinghouse Hanford Company (WHC). The task encompassed the following two subtasks related to the chemistry of alkaline Hanford Site tank waste:

1. Technical Liaison with the Institute of Physical Chemistry of the Russian Academy of Science (IPC/RAS) and its research into the chemistry of transuranic elements (TRU) and technetium (Tc) in alkaline media.
2. Laboratory investigation of the chemistry of calcination/dissolution (C/D) (or thermal reconstitution) as an alternative to the present reference Hanford Site tank waste pretreatment flowsheet, Enhanced Sludge Washing (ESW).

This report fulfills the milestone for the C/D subtask to "Provide End-of-Year Report on C/D Laboratory Test Results" due 30 September 1995. A companion report, fulfilling the milestone to provide an end-of-year report on the IPC/RAS liaison, also has been prepared (Delegard 1995c).

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2.0 BUDGET

Reallocation of funds to provide more support to the IPC/RAS (specifically, Professor A. K. Pikaev, principal investigator, and his investigations into the role of radiolysis on TRU and Tc chemistry in alkaline media) took place in April 1995. The reallocation decreased the total fiscal year funding from \$300,000 to \$260,000. The original budget allocated \$100,000 to the technical liaison and \$200,000 to the C/D subtask. To free funds for the work of Professor Pikaev, tests comparing C/D with ESW were decreased to study three rather than four genuine waste types.

In July 1995, funds spent on chemical and radionuclide concentration analyses (about \$128,000) were restored to the IPC Liaison and Chemistry of Thermal Reconstitution Task by the Tank Waste Characterization Project. The project restored funds as a means of supporting process chemistry development tasks, such as C/D, being undertaken within WHC and to make efficient use of analytical laboratory resources which were under-used in the latter half of the fiscal year. The funding restoration partially balanced the increased analytical chargeout rates (equivalent to \$63,000) which had been imposed since November 1994 and also permitted an additional work scope (on the Cesium Removal Pilot Plant) to be assumed.

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3.0 WORK ELEMENTS

Three work elements for the C/D chemistry task were identified and described in the TTP. Additional work elements, not anticipated at the time of drafting the TTP, emerged during the fiscal year and were incorporated into the task.

The original three work elements in the C/D chemistry subtask included the following:

1. Conduct C/D and ESW experiments using genuine Hanford Site tank wastes and compare the bulk chemical and radionuclide material balances.
2. Investigate candidate process steps for removing plutonium solubilized by C/D treatment of genuine wastes.
3. Support engineering laboratory tests of calcination conducted at the University of Idaho under Tank Waste Remediation System (EM-30) funding.

The additional work elements initiated under the C/D chemistry subtask included the following:

1. Investigate the use of peroxide as a waste pretreatment agent to dissolve chromium hydroxide by oxidation to chromate.
2. Determine plutonium and neptunium solution species produced in C/D processing of simulant wastes.
3. Investigate carbonate complexation of plutonium in highly alkaline solution.
4. Prepare technical reports on C/D processing of Hanford Site tank wastes.

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4.0 CALCINATION/DISSOLUTION AND WASTE CHEMISTRY STUDIES

Studies of the technology (chemistry and engineering) of C/D have been conducted at WHC at various levels of both EM-30 and EM-50 support since FY 1992. As a result of the FY 1994 Tri-Party Agreement decision to focus on ESW as the reference pretreatment flowsheet, investigation of other pretreatment alternatives was markedly reduced. Limited support of C/D was provided by EM-30 through Tank Waste Remediation System in FY 1995.

The Tank Waste Remediation System support of C/D ultimately was limited in FY 1995 to a continuation of engineering laboratory-scale calcination tests begun in FY 1994 using a 50 kilowatt nontransferred plasma torch at the University of Idaho. A small level of support for chemistry studies of C/D also was granted by the Tank Waste Remediation System primarily to aid University of Idaho tests.

In contrast, EM-50 saw merit in the continuing pursuit of alternative pretreatment technologies in case the reference ESW technology proved inadequate to provide efficient separations of low-level and high-level wastes (LLW and HLW, respectively). On this basis, EM-50, under the present TTP, granted \$200,000 to the continued studies of C/D chemistry.

The focus of the chemistry work was to compare C/D with ESW in laboratory-scale studies using genuine waste. Determining material balances (solution and residual solid phases) in these tests would enable evaluation of the relative performances of the reference ESW and competing C/D technologies. Solubilization of key bulk constituents (primarily aluminum, chromium, and phosphate) and radioactive but chemically trace constituents of Hanford Site tank wastes were the focus of these comparisons.

Tests conducted in FY 1993 and 1994 with genuine tank wastes showed plutonium was dissolved by C/D processing. The dissolution caused the undesired partitioning of a portion of the plutonium, an HLW component, to the LLW fraction. It was postulated that plutonium dissolved by oxidation to more soluble (V) or (VI) oxidation states from the sparingly soluble $\text{Pu}^{(IV)}\text{O}_2 \cdot x\text{H}_2\text{O}$ solid phase likely to be present in the waste. In the earlier work, the plutonium behavior was compared with the demonstrated behavior of manganese, a bulk constituent in the waste, because manganese dioxide MnO_2 , undergoes a similar oxidative dissolution in C/D processing to form manganate, MnO_4^{2-} ,



These observations suggested chemical reduction and coprecipitation (which reverses the oxidative dissolution reactions of plutonium and manganese) to remove solubilized plutonium from solution. The coprecipitation was expected to be particularly effective because of the high sorptive capacity of the bulk product manganese dioxide. To explore this possibility, a work element to determine the feasibility of the reduction/coprecipitation approach was included in the TTP.

A final TTP work element was included to support the laboratory-scale plasma calciner tests conducted by Professor Patrick Taylor of the Department of Metallurgical and Mining Engineering, College of Mines, at the University of Idaho. The work performed by Professor Taylor and colleagues was supported by the Tank Waste Remediation System, EM-30.

During the year, additional related work elements were identified and adopted for study. Work elements included the following: alkaline peroxide oxidative dissolution of chromium(III) hydroxide under ESW conditions, speciation of neptunium and plutonium solubilized by C/D processing, a study of carbonate complexation of plutonium in alkaline solution, and preparation of technical reports and presentations.

4.1 CALCINATION/DISSOLUTION AND ENHANCED SLUDGE WASHING COMPARISON TESTING

The steps originally envisioned for pretreatment of Hanford Site tank waste were simple water washing followed by solid-liquid separation to segregate the high-level insoluble residue from the low-level solution fraction (produced after removal of dissolved ^{137}Cs). The possible corollary removal of dissolved ^{90}Sr and ^{99}Tc was also considered. The high-level fraction (and separated ^{137}Cs , ^{90}Sr , and ^{99}Tc) would be vitrified for storage in the HLW repository while the stripped LLW fraction would be immobilized for onsite storage as grout. Glass was chosen as the LLW form later.

Chemical and engineering studies showed that certain bulk components in the insoluble waste residue (primarily aluminum, chromium, and phosphate) contribute significantly to the volume of vitrified HLW identified for expensive packaging and deep geological repository storage. With this in mind, ESW was proposed as an improved pretreatment that would be more successful than simple water washing in dissolving the aluminum, chromium, and phosphate sludge components. ESW involves leaching washed sludges with a sodium hydroxide (NaOH) solution. Alkaline hydrolysis and metathesis reactions dissolve the aluminum-, chromium-, and phosphate-bearing solid phases in the NaOH solution leach step.

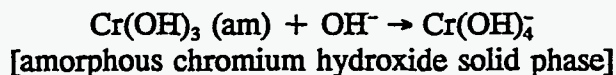
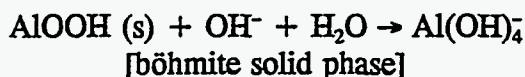
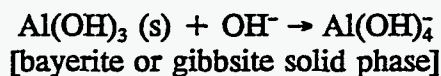
To be considered a viable alternative to the reference ESW flowsheet, the C/D pretreatment process must demonstrate advantages in performance and/or cost over ESW. In comparative water wash and C/D tests conducted with genuine wastes in FY 1993 and 1994, dissolutions of aluminum, chromium, and phosphate were higher by C/D than by water washing and often approached 100 percent dissolution (Delegard et al. 1994a, and Delegard 1995b). With the introduction of ESW as the reference pretreatment approach, similar tests, using genuine tank wastes, were required to compare the performances of C/D and ESW. These tests were performed in FY 1995.

An alternative oxidative dissolution technique for chromium-bearing sludge, based on aqueous alkaline peroxide leaching, was discovered when the C/D and ESW pretreatment processes were compared. Tests also were performed to determine the efficacy of peroxide in the (oxidative) dissolution of chromium from sludge. The results C/D and ESW testing (with and without peroxide leaching) are described.

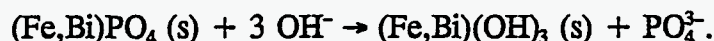
4.1.1 Enhanced Sludge Washing Reference Flowsheet Chemistry

The ESW pretreatment is a sequence of washes, alkaline leaches, and rinses intended to do the following:

1. dissolve water-soluble sodium salts (for example, NaNO_3 , NaNO_2 , Na_2CO_3 , Na_3PO_4)
2. dissolve, in sodium hydroxide solution, amphoteric aluminum and chromium hydroxides as their soluble hydroxide-complexed anions:



3. dissolve phosphates by hydroxide metathesis:



ESW leach conditions are similar to, but milder than, the conditions the aluminum industry uses to dissolve aluminum values from bauxitic ores containing the same gibbsite, bayerite, and böhmite aluminum-bearing solid phases as found in tank sludges (Hudson 1987).

Gibbsite and böhmite phases are especially prominent in REDOX process sludge.

The dissolution of Cr(OH)_3 by alkaline treatment is analogous to that exhibited by Al(OH)_3 ; that is, both are based the amphoterism of the respective trivalent metal ions. Quantification of the Cr(III) hydrolysis reaction is underway at the Pacific Northwest Laboratory (Felmy et al. 1994). According to this reference, approximately 0.0006 M chromium should dissolve under ESW alkaline leaching conditions (that is, 3 M NaOH).

4.1.2 Enhanced Sludge Washing with Peroxide Addition

An alternative method for dissolving $\text{Cr}(\text{OH})_3$ (am) is through aqueous oxidation reactions to form soluble chromate (CrO_4^{2-}). The relevant oxidation potential is comparatively low (-0.09 V; [Bratsch 1989]). The chemical literature shows that many oxidants, for example, hypochlorite, hypobromite, peroxide, and pressurized oxygen are effective in oxidizing $\text{Cr}(\text{OH})_3$ (am) alkaline solution (Kirk-Othmer 1981 and Wulfsberg 1991). In fact, peroxide in NaOH solution is used in sample preparations for spectrophotometric analyses of chromium. The alkaline peroxide treatment dissolves chromium(III) compounds and forms the highly-absorbing chromate chromophore (Oelschläger 1955, Sandell 1959, and Kirk-Othmer 1993).

Recent work at the Pacific Northwest Laboratory has shown that permanganate (MnO_4^-) can be used to oxidize $\text{Cr}(\text{OH})_3$ (am) to chromate. Oxidation of $\text{Cr}(\text{OH})_3$ (am) by pressurized air (oxygen) in simulated wastes has been observed by the Pacific Northwest Laboratory in developing wet-air oxidation of organic compounds contained in Hanford Site tank wastes. Oxidative dissolution of $\text{Cr}(\text{OH})_3$ (am) present in tank 101-SY waste by ozone has been demonstrated at WHC (Delegard et al. 1993).

Of these oxidants, peroxide, introduced as 30 wt% H_2O_2 aqueous solution, is perhaps the most interesting for possible pretreatment operations. It does not contribute to the halide loading of the waste (as would hypochlorite and hypobromite), it does not contribute to the sludge volume (as would permanganate), and it does not require the pressurized equipment of wet air oxidation or the specialized equipment for ozonation.

The balanced reaction for the oxidation of $\text{Cr}(\text{OH})_3$ with peroxide



indicates the 3:2 mole ratio of peroxide:Cr(III) required to achieve dissolution. In practice, excess peroxide probably would be required because of loss caused by disproportionation of peroxide (catalyzed by other waste components) to form oxygen gas and water. The balanced reaction shows that hydroxide would be consumed in the dissolution of $\text{Cr}(\text{OH})_3$. No hydroxide would be lost by the disproportionation of excess hydrogen peroxide. The consumption of hydroxide could affect aluminum solution concentration for wastes at or near the solubility limit for gibbsite [$\text{Al}(\text{OH})_3$] by the reaction



Side reactions of peroxide with other waste components also are possible. For example, peroxide complexes of the actinides in their (V) and (VI) oxidation states in alkaline media exist and have been described in the technical literature [Chernyaev 1966 for U(VI) and Musikas 1976 for Np(V) and Pu(V)]. Salts of the peroxide complexes are generally more soluble in alkaline solution than their starting actinide compounds and potentially could increase the solution concentrations of these actinide elements.

The Pu(V) peroxide complex has been shown to be relatively unstable and to decompose quickly at room temperature to precipitate PuO₂ (Musikas 1976). The Np(V) peroxide complex, at approximately 0.26 g Np/L solution concentration, was observed by spectrophotometry in some tests involving C/D conditions. The Np(V) peroxide complex also was found to decompose with time but required a few weeks at room temperature to occur. Knowledge of the thermal stability of the U(VI) peroxide complex in alkaline solution is not available.

If a stable U(VI) complex forms, its existence may offer an attractive way to dissolve uranium from the sludge. This would allow separation of uranium from the waste or inclusion of uranium in the soluble LLW fraction for immobilization. In either case, removing uranium from the sludge would reduce the HLW glass volume.

4.1.3 Experimental Materials and Methods

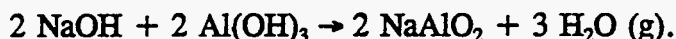
Materials and experimental methods used in the comparative tests of C/D and ESW are described.

4.1.3.1 Calcination/Dissolution Tests. The C/D experimental technique has been developed in tests performed over the past two years (Delegard et al. 1994a and Delegard 1995b). Weighed aliquots of genuine waste sludge materials are dried in an alumina crucible at approximately 120 °C for several hours to remove solution water, heated at 300 °C for 20 minutes to remove bound (hydrate) water, and finally transferred directly to an 850 °C furnace for 20 minutes calcination. This stepwise drying eliminates rapid boil-up and dispersal of the radioactive materials thereby improving operational safety and achieving a material balance.

The calcine is cooled and dissolved, in three contacts, with "Q" (purified) water. The initial water contact is conducted directly in the crucible. The solution and insoluble materials from the first water contact are transferred to a centrifuge cone. The product slurry is centrifuged, the supernatant solution decanted, and the solution weight and volume determined. Two subsequent Q water wash contacts (with centrifugation and decantation) are made on the residual solids, the two separate solution fractions are composited, and their volumes are determined and weighed. The residue is dried at 100 °C and weighed. The solutions and residues are analyzed for chemical and radionuclide concentrations in the Analytical Laboratories of 222-S at WHC, and a material balance is calculated.

For many sludges, which are deficient in salts (nitrate, nitrite, hydroxide) to supply the fluxing sodium hydroxide required for C/D reactions, an additional source of flux is required. If C/D were applied to the Hanford Site tank waste sludges, the source of the fluxing agent would be the abundant tank salt cake wastes. Hanford Site salt cake wastes are predominantly NaNO₃. Prior C/D laboratory tests used waste from tank 101-SY or reagent sodium nitrate (NaNO₃) to supply the flux.

Samples of genuine salt cake are not currently available in the laboratory; therefore, reagent NaNO_3 now is used in C/D tests where additional fluxing agent is required. The amounts of NaNO_3 added are calculated to be sufficient to exceed the NaOH requirements of the genuine waste aliquot at least two-fold on the basis of fusion reactions to form soluble sodium salts. The reaction of aluminum hydroxide with sodium hydroxide to form the water-soluble sodium aluminate salt is one such fusion reaction:



4.1.3.2 Enhanced Sludge Washing Tests. The ESW test procedure was adopted from a laboratory protocol provided by Dr. Gregg Lumetta of PNL. The steps used in the ESW tests are described by the following general laboratory instructions.

1. Weigh a 5 gram genuine waste aliquot accurately into a tare-weighed, capped 50-mL polypropylene centrifuge cone.
2. Add 15 mL of 0.01 M NaOH /0.01 M NaNO_2 wash solution to the waste aliquot.
3. Heat the cone and contents to 100 °C for two hours.
4. Remove the cone/contents from the oven, cool, centrifuge, and decant the liquid into a tare-weighed container. Reweigh to determine solution weight.
5. Repeat steps 2 to 4 for two more wash contacts (that is, three total). Composite the three washes for analysis.
6. Contact the residue with 15 mL 3 M NaOH leach solution. Heat at 100 °C for five hours.
7. Remove the cone/contents from the oven, cool, centrifuge, and decant the liquid into a tare-weighed container. Reweigh to determine solution weight.
8. Repeat steps 6 and 7 for another contact. Composite the two collected leach solutions for analysis.
9. Rinse the residual solids with 15 mL water (no heating or extended contact time required). Centrifuge, decant, and repeat the rinse. Composite the two rinses for analysis in a tare-weighed container and reweigh.
10. Dry the residual solids at 100 °C and record the final weight.

The solutions and residues produced by the ESW tests are analyzed for chemical and radionuclide concentrations in the Analytical Laboratories of 222-S at WHC and material balances are calculated.

4.1.3.3 Enhanced Sludge Washing Tests with Peroxide. Tests of ESW with peroxide were performed using a procedure similar to the standard ESW procedure described in Section 4.1.3.2. To perform the ESW tests with peroxide, the steps 6 through 8 below were used in place of steps 6 through 8 above.

6. Contact the residue with 15 ml 3 M NaOH leach solution and 200 μ L 30 percent H_2O_2 solution. Agitate to mix the solids and leach solution; continue agitation for a minimum two days contact at room temperature.
7. Centrifuge and decant the liquid into a tare-weighted container. Reweigh to determine solution weight.
8. Repeat steps 6 and 7 for another contact. Collect the two separate leach solutions for individual analyses.

4.1.3.4 Alkaline Peroxide Leaching of Simulated Tank 101-SY Waste. Scouting tests of the alkaline peroxide leach process were run to determine the efficacy of this technique to dissolve $Cr(OH)_3$ from simulated tank 101-SY waste. The tests began with room temperature water washing of simulated tank 101-SY waste. The remaining residue contained the bulk of the chromium. Alkaline peroxide leaching of the residue then was tested using 3 M NaOH and varying quantities of added 30 percent H_2O_2 leachant at room temperature. Leachant was added at the ratio 3 mL of NaOH solution per gram of original tank 101-SY simulant. Two leach contacts were made.

The leach solutions and residues were agitated and solution samples were taken periodically. The sample aliquots were diluted in 0.5 M NaOH, and the absorbance caused by the dissolved chromate was determined spectrophotometrically at the 372 nm absorbance maximum. Calibration tests were run with sodium chromate (Na_2CrO_4) standard solutions prepared from reagent chemical in 0.5 M NaOH solution. Excellent adherence to the Beer-Lambert law was observed for the concentration range of interest.

4.1.3.5 Materials. Sludges used in the C/D and ESW tests were taken from tank 104-S, a REDOX process waste with high amounts of aluminum hydroxide phases; tank 101-SY, a complexed-concentrate waste containing a relatively high concentration of chromium in the sludge phase; and from tank 102-SY, a chromium-rich waste with high TRU concentrations from the Plutonium Finishing Plant. A sludge from tank 110-U containing mixed REDOX, aluminum cladding, and bismuth phosphate process wastes was to be tested, but the reallocation of \$40,000 to support Professor Pikaev at the IPC/RAS did not permit completion of this work. However, chemical and radionuclide analyses for tank 110-U waste were performed and are reported.

The amount of waste materials and added chemicals for tests with wastes from tanks 104-S, 101-SY, and 102-SY are summarized in Table 4-1. $NaNO_2$ and NaOH were used in the ESW leachants. For the ESW tests with peroxide, 30 percent H_2O_2 was added. For the C/D tests with tank 104-S waste, $NaNO_3$ was added as a salt cake simulant. The relative amount

of sodium added in the tests also is compared. As shown in Table 4-1, the tank 104-S ESW test used more than twice as much sodium, per unit waste, than the C/D test. For the tanks 101-SY and 102-SY C/D tests, no added NaNO_3 was used, whereas the prescribed amounts of NaNO_2 and NaOH were used for the ESW tests (with and without added peroxide).

It is important to recognize that the sodium required for ESW originates from additions of freshly purchased NaNO_2 and NaOH . The added sodium in these salts ultimately becomes contaminated waste and contributes directly to the vitrified LLW waste bulk (LLW glass bulk is sodium limited). In contrast, the C/D sodium requirements are met using salt cake already present in the waste tanks thereby adding nothing to the LLW waste bulk.

Table 4-1. Materials Used in ESW, ESW/Peroxide, and C/D Tests.

Tank Waste Source	Weights of Materials Used in Tests			
	ESW		C/D	
	waste/ NaNO_2 / NaOH (30% H_2O_2)	g Na/ g waste	waste/ NaNO_3	g Na/ g waste
104-S	4.999 g/0.031 g/3.618 g	0.42	10.011 g/7.150 g	0.19
101-SY	5.083 g/0.031 g/3.618 g	0.41	5.98 g/0 g ¹	0
102-SY	5.290 g/0.031 g/3.618 g	0.40	18.086 g/0 g ²	0
101-SY (H_2O_2)	4.805 g/0.031 g/3.618 g (0.132 g)	0.38		
102-SY (H_2O_2)	4.842 g/0.031 g/3.618 g (0.132 g)	0.39		

Notes:

¹Delegard et al. 1994a.

²Delegard 1995b.

The waste compositions used in the ESW and C/D comparison tests (from tanks 104-S, 101-SY, 102-SY) and 110-U are shown in Table 4-2. The simulant nonradioactive tank 101-SY waste composition is also shown in Table 4-2. This simulant was used in scouting tests of alkaline peroxide leach treatment to dissolve chromium hydroxide. The preparation of the simulant, which has been used in previous laboratory tests of C/D and ozone waste pretreatment, is described (Delegard et al. 1993).

4.1.4 Results of Calcination/Dissolution and Enhanced Sludge Washing Testing for Tank 104-S Waste

ESW and C/D test results for tank 104-S waste can be compared based on the quantities of bulk chemical components and trace radionuclides dissolved by the two processing alternatives. Data tables related to experiments with tank 104-S waste are provided in Appendix A.

The dissolutions of bulk chemical constituents by the two processes are compared in Figure 4-1. The amounts dissolved are displayed next to the amounts present in the initial waste mixtures (that is, mixtures which include the $\text{NaNO}_2/\text{NaOH}$ processing chemicals used in ESW and the NaNO_3 salt cake simulant used in the C/D of the tank 104-S waste).

Figure 4-1. Dissolution of Bulk Waste Constituents in ESW and C/D Processing of Tank 104-S Waste.

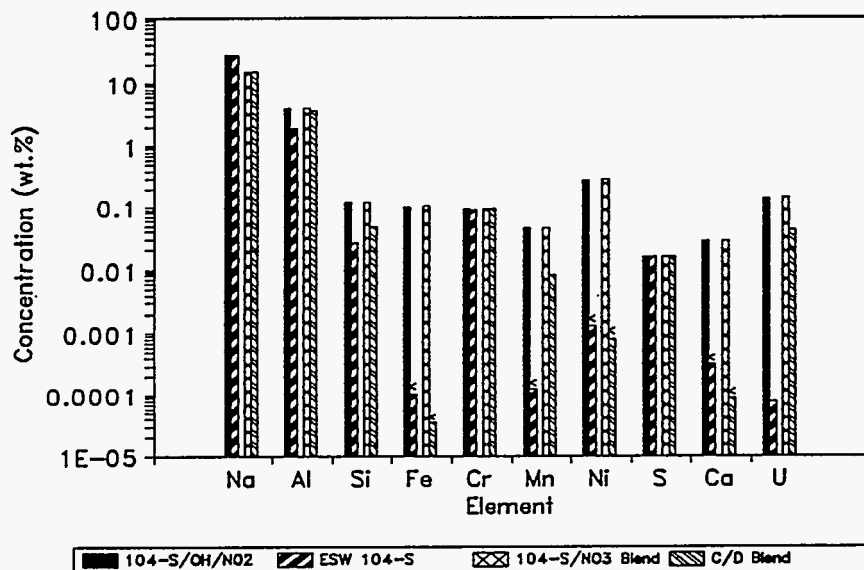


Table 4-2. Compositions of Hanford Site Tank Wastes Used in C/D and ESW Tests.

Component	Concentration (wt%)				
	104-S	101-SY ¹	102-SY ²	110-U	Simulant 101-SY ³
Al	6.78	3.8	3.04	3.77	3.2
Bi	—	—	—	0.94	—
Ca	0.049	0.027	0.15	0.068	0.023
Cr	0.157	0.48	1.15	0.060	0.41
Fe	0.177	0.028	0.82	0.97	0.028
K	—	0.38	0.18	0.035	0.326
Mn	0.080	0.0068*	0.26	0.42	—
Na	7.00	24.4	8.13	7.89	20.7
Ni	0.49	0.018	0.22	0.0009	0.015
Si	0.21	0.046*	0.13	0.50	—
U	0.25	0.0074	0.14	0.67	—
Cl ⁻	0.20	0.93	0.18	0.092	0.79
F ⁻	<0.0007	0.04	0.056	0.37	0.03
NO ₂	1.90	12.4	2.44	1.45	10.5
NO ₃	14.2	13.8	5.71	4.89	11.7
PO ₄ ³⁻	<0.007	0.76	1.26	4.20	0.64
SO ₄ ²⁻	0.091	0.47	4.17	0.12	0.40
CO ₃ ²⁻	1.39	3.72	1.96	0.78	3.15
OH ⁻	0.94	2.92	0.96	—	2.47
TOC	0.76	1.81	0.54	0.46	1.53
H ₂ O	60.5	23.8*	62.5	77.0	35.5
Nuclide	Concentration (μCi/g)				
⁹⁰ Sr	154	25	85.8	0.113	—
⁹⁹ Tc	0.012	0.24	0.10	0.0055	—
¹³⁷ Cs	38.5	396	51.9	20.7	—
²³⁷ Np	<0.013	0.000032	0.00086	0.00265	—
^{239,240} Pu	0.199	0.0076	4.38	0.292	—
²⁴¹ Am	0.061	0.12	14.9	0.1225	—

Notes:

¹ Based on Herting et al. 1992 and corrected for water content; * value from current analysis.

² From Delegard 1995b.

³ From Delegard et al. 1993.

ESW and C/D dissolved approximately 99 percent of the sodium. This behavior would be expected except for wastes which contain significant concentrations of both aluminum and silicon. In such cases, sodium aluminosilicates (NaAlSiO_4), for example, sodalite and cancrinite of low solubility, form in alkaline solution, and sodium is precipitated. Because the tank 104-S waste contains high concentrations of aluminum but relatively minor concentrations of silicon, little NaAlSiO_4 should form.

Aluminum dissolutions were markedly different for the ESW and C/D processes. C/D dissolved approximately 91 percent of the aluminum in tank 104-S waste; ESW dissolved 47 percent. X-ray diffractometry of tank 104-S starting material revealed the presence of the difficultly-soluble phase böhmite (AlOOH) but not the less refractory gibbsite [$\text{Al}(\text{OH})_3$].

When böhmite is encountered in aluminum-bearing ore, the aluminum industry uses severe digestive conditions (200 to 230 °C) to achieve greater and faster aluminum dissolution. These temperatures are 90 to 100 °C higher than those used to dissolve gibbsitic ores (Hudson 1987) and 100 to 120 °C higher than those used in the present ESW tests.

Concentrations of aluminum and hydroxide in ESW caustic leach solutions, 0.228 M and 2.57 M, respectively, are near the solubility limit for gibbsite in NaOH solution at room temperature (solubility curves derived by Barney [1976] from technical literature data). These results may indicate that increased aluminum phase dissolution requires further leach contacts. If so, an additional 1½ volumes of leach solution would be required to completely dissolve aluminum under ESW conditions. Nevertheless, for tank 104-S waste, aluminum dissolution by C/D processing was superior to that of ESW.

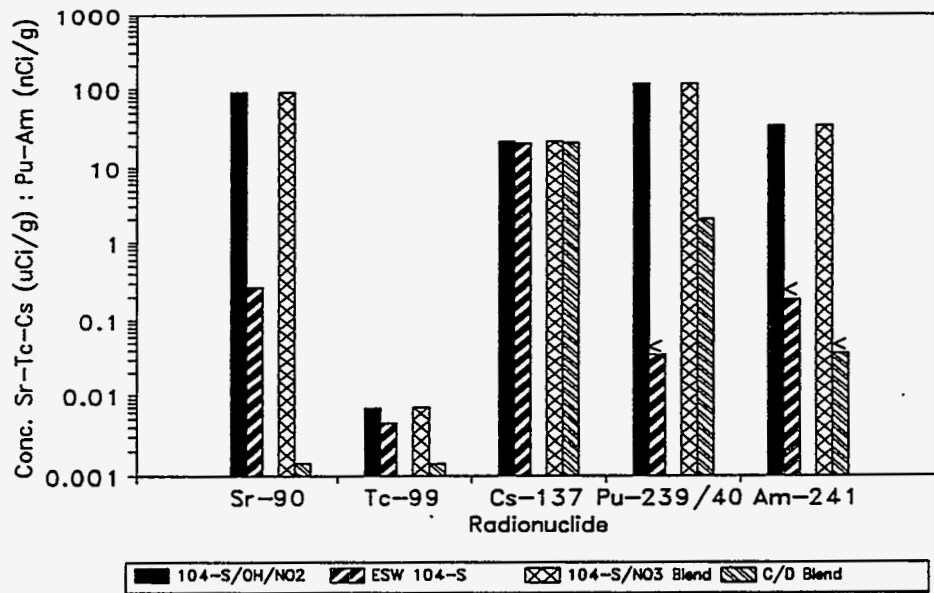
Both approaches dissolved almost all the chromium (approximately 97 percent dissolved by ESW and >99.9 percent dissolved by C/D). Inspection of the ESW data revealed that chromium dissolution was complete in the initial dilute $\text{NaNO}_2/\text{NaOH}$ wash steps. Analyses of the original tank 104-S waste also showed the contained chromium to be water soluble, and it reported to solution as chromate (CrO_4^{2-}). Because the chromium in this tank 104-S waste sample was present only as CrO_4^{2-} , the $\text{Cr}(\text{OH})_3$ alkaline hydrolysis reaction could not be demonstrated for ESW.

All the sulfur (sulfate) was dissolved by ESW and C/D. Because tank 104-S waste contained phosphorus concentrations below analytical detection limits, phosphate dissolution behavior could not be determined. Dissolutions of silicon and manganese were higher for C/D than for ESW. Approximately 40 percent of the silicon dissolved by C/D as compared to 22 percent by ESW. Manganese dissolution was approximately 18 percent by C/D and less than 0.3 percent by ESW. Dissolutions of iron, nickel, and calcium were negligible by C/D and ESW.

The dissolutions of radioactive components achieved by ESW and C/D processing of tank 104-S sludge are shown in Figure 4-2. As expected for wastes containing no complexing agents (for example, EDTA), neither process dissolved significant amounts of ^{241}Am (less than 1 percent dissolved in both cases) or ^{90}Sr (less than 0.3 percent dissolved).

In contrast and as expected, both processes dissolved most of the ¹³⁷Cs (approximately 96 percent by ESW and 98 percent by C/D). Technetium-99 dissolution was approximately 64 percent for ESW and approximately 20 percent by C/D. The behavior of ⁹⁹Tc is not expected (most ⁹⁹Tc should be easily dissolved as the pertechnetate ion, TcO₄⁻) and may be related to the low concentrations in tank 104-S waste thus making accurate analyses difficult. Tests with wastes having higher concentrations of ⁹⁹Tc (for example, tanks 101-SY and 102-SY) are described in the following sections.

Figure 4-2. Dissolution of Radioactive Components in ESW and C/D Processing of Tank 104-S Waste.



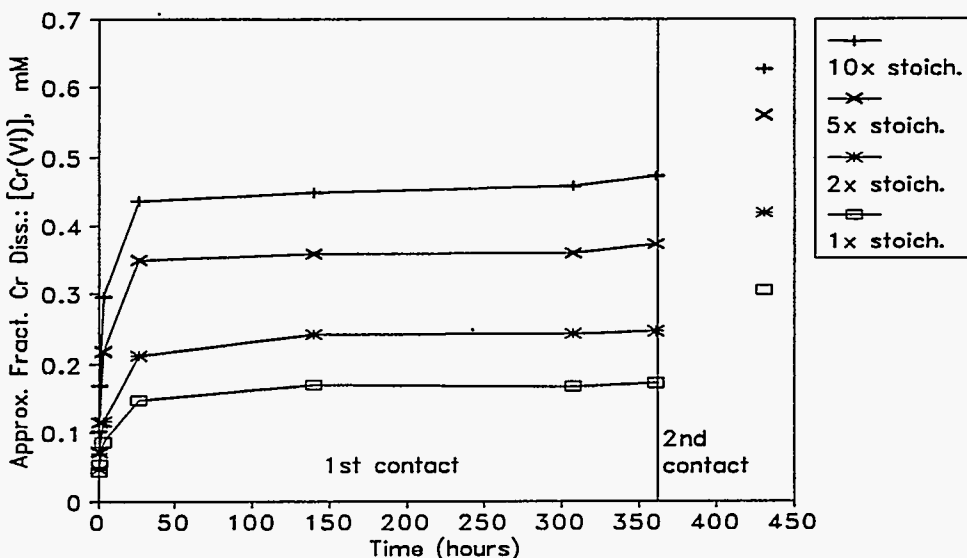
Because the concentrations of ^{237}Np in tank 104-S waste were below the analytical detection limits, the behavior of this important TRU isotope could not be determined. In contrast, the concentrations of uranium present in the waste were relatively high (approximately 0.6 weight percent on a dry weight basis). The ESW process dissolved approximately 0.05 percent of the uranium (see Figure 4-1); the C/D process, almost 30 percent.

The $^{239,240}\text{Pu}$ dissolved in ESW processing was less than 0.02 percent. In contrast, the $^{239,240}\text{Pu}$ dissolved by C/D was approximately 2 percent. Enhanced dissolution of plutonium by C/D processing has been noted in earlier studies (Delegard et al. 1994a and Delegard 1995b) and was postulated to be caused by oxidative dissolution in analogy with the behavior of manganese. Further experiments to confirm this hypothesis and to determine methods to remove the solubilized plutonium were conducted and are described in a later section.

4.1.5 Results of Alkaline Peroxide Leaching of Simulated Tank 101-SY Waste

Results of the scouting test of alkaline peroxide dissolution of chromium from simulated tank 101-SY waste are shown in Figure 4-3 (experimental data are given in Appendix B). Most of the reaction occurred in the first day of contact. As expected, the peroxide was not completely efficient in oxidizing $\text{Cr}(\text{OH})_3$ to chromate. An amount of peroxide just

Figure 4-3. Chromium Oxidative Dissolution from Simulated Waste of Tank 101-SY by Alkaline Peroxide Treatment.



sufficient, stoichiometrically, to oxidize all the chromium hydroxide to chromate resulted in approximately 15 percent dissolution. Increasing the amount of peroxide to 10 times stoichiometry increased chromate yield approximately three times.

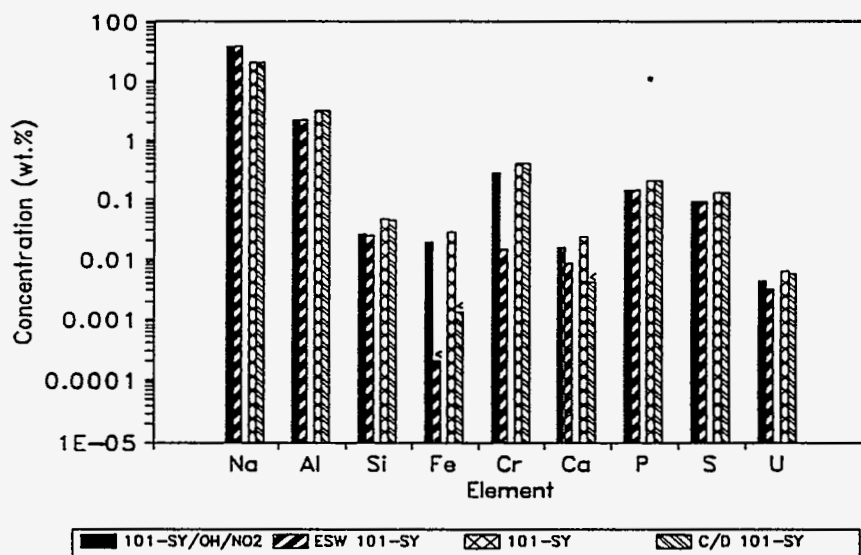
Each of the four experimental tests underwent a second contact of 3 M NaOH with peroxide. The peroxide again was added at 1-, 2-, 5-, and 10-times stoichiometry. At the end of the second (three day) contact, the total amount of chromium dissolved ranged from 30 percent (at stoichiometry) to 65 percent (at 10 times stoichiometry). Based on these results, ESW tests with peroxide were conducted at room temperature with two leach contacts at 20 times stoichiometry in peroxide and with contact times of at least two days.

4.1.6 Results of C/D, ESW, and ESW/Peroxide Testing for Tank 101-SY Waste

Test results for C/D and ESW treatment were compared based on the fractions of bulk chemical components and trace radionuclides dissolved. Separate comparisons of ESW and ESW/peroxide were made to determine the effects of peroxide treatment. Experimental data are given in Appendix C.

4.1.6.1 Comparison of C/D and ESW for Tank 101-SY Waste. The dissolution of bulk chemical constituents by C/D and ESW processing are compared in Figure 4-4. The amounts dissolved are shown next to the amounts present in the initial waste mixtures (including processing chemicals used for ESW).

Figure 4-4. Dissolution of Bulk Waste Constituents in ESW and C/D Processing of Tank 101-SY Waste.



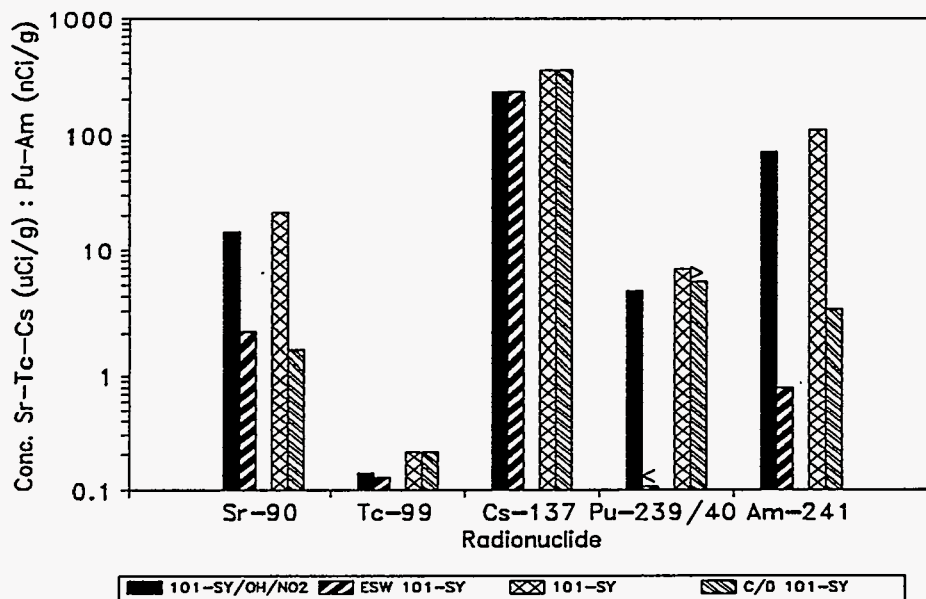
As expected for tank 101-SY waste composition, both treatments dissolved all the sodium. Aluminum also was largely dissolved by C/D (99.8 percent) and ESW (98.8 percent). This was expected because most aluminum is in the solution phase in tank 101-SY waste. Phosphate and sulfate also were completely dissolved.

Chromium dissolution behaviors were different for C/D and ESW. While chromium dissolution was complete for C/D (reporting to solution as chromate), approximately 5.2 percent of the chromium dissolved by ESW. Most of that chromium was dissolved in the 3 M NaOH leach step. The solution concentrations relevant for the ESW alkaline hydrolysis reactions are as follows: $[OH^-] = 3.12 \text{ M}$, $[Cr] = 0.00095 \text{ M}$.

The dissolved chromium concentration (that is, 0.00095 M in 3.12 M NaOH) is similar to that predicted by the work of Felmy et al. (1994) (approximately 0.0006 M chromium). However, the dissolved chromium concentration is low compared with the concentration available in tank 101-SY tank waste (approximately 0.13 M, nearly all in the solid phase) (Herting et al. 1992). To dissolve this solid phase chromium by ESW, approximately $0.13/0.00095 = 140$ waste volumes of 3.1 M NaOH leachant would be required.

The dissolution behaviors of the radioactive components under ESW and C/D are shown in Figure 4-5. The behavior of uranium, shown in Figure 4-4 as a bulk component, is similar for both techniques: approximately 90 percent dissolution by C/D and 73 percent dissolution by ESW. However, the concentration of uranium in tank 101-SY waste is relatively low. The fraction dissolved may reflect only a solubility limit imposed, for example, by a sodium diuranate solid phase. Almost complete dissolution of ^{137}Cs (exceeding 99 percent) is shown

Figure 4-5. Dissolution of Radioactive Components in ESW and C/D Processing of Tank 101-SY Waste.



by both techniques. Technetium-99 dissolution also is complete for C/D and approximately 93 percent by ESW. As expected, dissolutions of ^{90}Sr and ^{241}Am were low by C/D and ESW. Approximately 7 percent of the ^{90}Sr dissolved by C/D versus approximately 14 percent by ESW; the analogous figures for ^{241}Am were 3 percent and 1 percent.

The behavior of $^{239,240}\text{Pu}$ in C/D again was consistent with the oxidative dissolution postulated for plutonium. Over 78 percent of the plutonium dissolved by C/D versus less than 3 percent dissolved by ESW. Because of low concentrations, neptunium behavior could not be determined reliably.

4.1.6.2 Comparison of ESW and ESW/Peroxide for Tank 101-SY Waste. The effects of adding peroxide on the reference ESW flowsheet were determined by comparing the leaching results for bulk chemical (chromium and aluminum) and radionuclide (uranium and $^{239,240}\text{Pu}$) components in ESW and ESW/peroxide tests with tank 101-SY waste.

Although markedly enhanced solubilization of chromium (caused by oxidative dissolution to chromate) was shown in the alkaline peroxide leaching tests with the simulant tank 101-SY waste, little increase in chromium dissolution was shown in the comparative tests with genuine tank 101-SY waste. For ESW, approximately 5.2 percent chromium dissolution was obtained; with ESW/peroxide, the chromium dissolution increased to 8.2 percent. Aluminum dissolution decreased from 98.8 percent by ESW to 93 percent by ESW/peroxide.

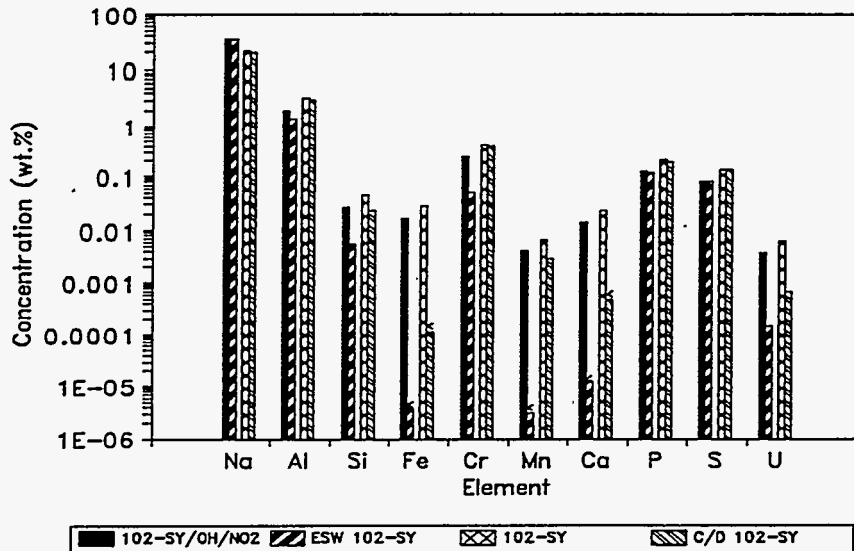
Uranium dissolution increased to greater than 94 percent by ESW/peroxide versus 73 percent dissolved by ESW. Plutonium dissolution also increased from 2.4 percent by ESW to 4.2 percent by ESW/peroxide. In summary, the ESW/peroxide treatment showed marginal differences from the reference ESW treatment for tank 101-SY waste.

4.1.7 Results of C/D, ESW, and ESW/Peroxide Testing for Tank 102-SY Waste

ESW and C/D test results for tank 102-SY waste can be compared based on bulk chemical components and trace radionuclides dissolved by the respective processing techniques. The effects of the alkaline peroxide ESW then can be compared with those obtained by ESW. Experimental data are given in Appendix D.

4.1.7.1 Comparison of C/D and ESW for Tank 102-SY Waste. Comparisons of the dissolutions of bulk chemical components by ESW and C/D processes are shown in Figure 4-6. Taken together, the sodium, aluminum, and silicon dissolution behaviors for the ESW tests give evidence for the possible formation of a limited amount of sodium aluminosilicate. Nevertheless, sodium dissolutions for ESW and C/D were almost complete. Silicon, the limiting reagent in tank 102-SY waste for the formation of sodium aluminosilicates, was approximately 20 percent dissolved in ESW and approximately 50 percent dissolved in C/D.

Figure 4-6. Dissolution of Bulk Waste Constituents in ESW and C/D Processing of Tank 102-SY Waste.



Aluminum dissolution was approximately 70 percent by ESW versus approximately 95 percent dissolved by C/D. The aluminum solution concentration achieved in the ESW alkaline leach step (0.143 M at 2.65 M NaOH) is a little lower than that expected in the solubility limit for gibbsite (approximately 0.2 M [Barney 1976]); it may reflect the formation of sodium aluminosilicates.

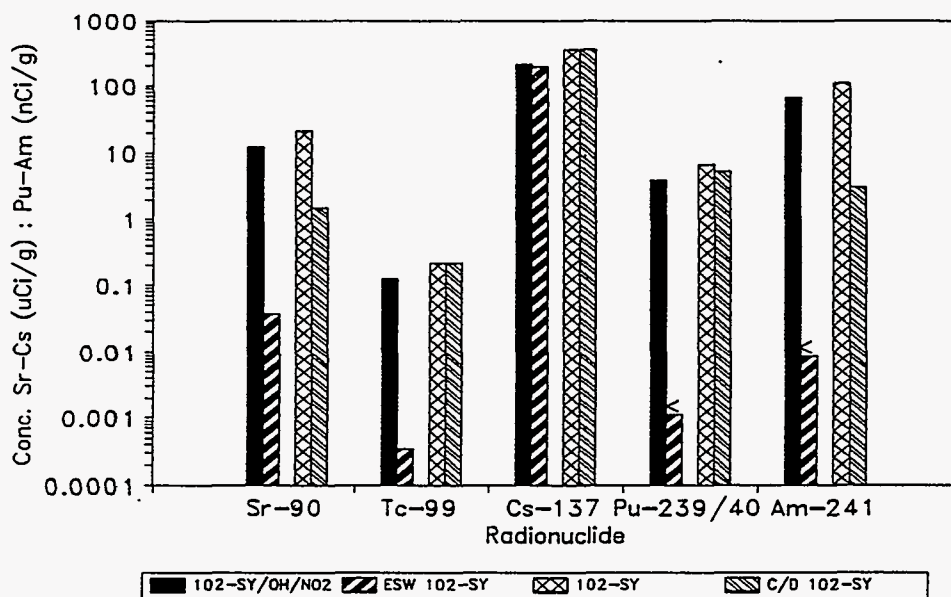
As with tests with tank 101-SY waste, chromium dissolution behaviors were different for ESW (approximately 21 percent dissolved) and C/D (over 99 percent dissolved). In C/D tests, the chromium dissolved by the oxidative dissolution imposed by the separate calcination and water dissolution steps. For the ESW test, most of the chromium reporting to the solution was chromate in the initial dilute alkali wash steps. The solution concentration of chromium found in the 2.65 M NaOH ESW leach step was approximately 0.002 M. According to the work of Felmy et al. (1994), the solubility limit of $\text{Cr}(\text{OH})_3$ at 2.65 M NaOH should be approximately 0.0005 M. The pale yellow color observed in the leach solution indicates that "excess" dissolved chromium may be caused by chromate that is not completely removed in the initial wash. The concentration of chromium remaining in tank 102-SY waste after a dilute alkali wash is approximately 0.26 M (based on the original waste volume). To dissolve this chromium by alkaline hydrolysis would require 0.26/0.0006 or approximately 430 waste volumes of 3 M NaOH.

Iron dissolution was less than 1 percent by ESW and C/D. Manganese dissolutions were different. Less than 1 percent of the manganese dissolved by ESW. Approximately 42 percent of the manganese dissolved by C/D processing. Oxidative dissolution of manganese was evident by the observation of the intensely green manganate (MnO_4^{2-}) solution species.

The dissolution behaviors of radioactive components in tank 102-SY waste by ESW and C/D processing are compared in Figure 4-7. The dissolution of uranium, a bulk waste component that is also radioactive, is shown in Figure 4-6. Uranium dissolution was approximately 4 percent in the ESW tests and about 10 percent by the C/D processing. Dissolutions of ^{137}Cs were high for ESW (95 percent) and C/D (over 99 percent) while the dissolutions of ^{90}Sr were low (0.3 percent and 7 percent, respectively). Technetium-99 dissolutions were complete for C/D but less than 1 percent by ESW. This is an unexpected result (most ^{99}Tc should be dissolved). Further experimental testing for technetium behavior in sludge processing is required. ^{241}Am dissolutions were low (less than 3 percent) for ESW and C/D.

The parallel behaviors of manganese and plutonium in C/D processing were shown again. While ESW dissolved less than 1 percent of the manganese and plutonium, C/D dissolved approximately 42 percent of the manganese and approximately 77 percent of the $^{239,240}Pu$. Oxidative dissolution of manganese and plutonium is at work. Further tests to determine the joint and separate behaviors of manganese and plutonium in C/D processing are described in later sections.

Figure 4-7. Dissolution of Radioactive Components in ESW and C/D Processing of Tank 102-SY Waste.



4.1.7.2 Comparison of ESW and ESW/Peroxide for Tank 102-SY Waste. Leaching results were compared for ESW and ESW/peroxide tests on waste from tank 102-SY. Enhanced solubilization of chromium was the goal of adding peroxide to the ESW alkaline leach step. For tank 102-SY waste, the amount of chromium dissolved decreased from the 21 percent found by ESW to approximately 16 percent by the combined ESW/peroxide treatment. Aluminum dissolution also decreased: 71 percent dissolved by ESW and 66 percent dissolved by the ESW/peroxide treatment. Uranium dissolutions were equivalent (3 to 4 percent) by both and ESW/peroxide. Plutonium dissolutions also were similar and were less than 1 percent for both techniques.

The ESW/peroxide results from wastes of tanks 101-SY and 102-SY indicate no benefit is gained by adding peroxide. The solubilization of chromium(III) hydroxide by the reference ESW flowsheet remains limited by its low solubility compared with the concentrations available in many wastes (including wastes from tanks 101-SY and 102-SY). Oxidative dissolution remains the only viable way of achieving reasonable $\text{Cr}(\text{OH})_3$ solubilization. Further tests of other possible $\text{Cr}(\text{OH})_3$ oxidants (such as hypochlorite) are proposed for future investigations.

4.2 PLUTONIUM REMOVAL FROM C/D PRODUCT SOLUTIONS

Previous discussions of C/D results for wastes in tanks 104-S, 101-SY, and 102-SY and discussions in earlier reports on C/D testing have shown that manganese and plutonium dissolution behaviors are similar in C/D processing. Similar behaviors are thought to be caused by oxidation forming soluble anions [manganate, MnO_4^{2-} , and plutonium(VI) or (V) species, $\text{PuO}_2(\text{OH})_4^{2-}$ or $\text{PuO}_2(\text{OH})_4^{3-}$, respectively]. The dissolution of plutonium, and its reporting to the LLW solution phase in pretreatment processing, is not desired. If plutonium reports to the dissolved LLW stream, a secondary step must be used to remove plutonium from solution.

Reduction of manganate to form insoluble manganese dioxide (MnO_2), the corollary reduction of the solubilized plutonate [Pu(VI) or Pu(V)] to form plutonium dioxide (PuO_2), and coprecipitation of the product oxides offer a promising approach to remove the solubilized plutonium from the LLW stream. Experiments were conducted to test reduction/coprecipitation with C/D product solutions originating from genuine tank 102-SY waste and plutonium- and neptunium-spiked Hanford Site tank composite simulant waste. The reductants tested, hydroxylamine hydrochloride ($\text{NH}_2\text{OH}\cdot\text{HCl}$) and hydrogen peroxide (H_2O_2), are volatile at glass forming temperatures and have the advantage of not adding bulk to the vitrified waste form. Experimental data are summarized in Appendix E.

4.2.1 Coprecipitation Tests with Tank 102-SY Waste C/D Solution

Reduction and coprecipitation experiments were conducted with three separate product solutions from C/D testing of tank 102-SY waste. Although the solutions had been stored in the laboratory for up to 100 days following the C/D experiments, the continuing presence of dissolved manganese was evident by the intense green solution coloration caused by manganate (MnO_4^{2-}) for two solutions and maroon caused by permanganate (MnO_4^-) for the remaining solution.

Each of the three stored solutions were centrifuged, and the supernatant solutions were withdrawn. Aliquots of solutions were analyzed to determine bulk metal concentrations and concentrations of dissolved radionuclides. One molar solutions of the hydroxylamine and peroxide reductants were prepared from reagent chemicals and Q water.

Five mL aliquots of the clarified solutions were drawn. In separate tests, reductants were added by drop (approximately 0.05 mL per drop) to the 5 mL test aliquots, then the solutions were agitated, and the solution's appearances were noted.

In all cases, reactions were rapid and vivid. Introduction of one drop of reductant was sufficient in all cases to cause solution color to change instantly from dark green (caused by manganate) or maroon (caused by permanganate in the solution having lower hydroxide concentration), to brown, then to bright yellow. After a few seconds, as the brown color was supplanted by the yellow, small dark brown solid particles formed. These observations are consistent with the precipitation of brown manganese(IV) dioxide reduced from soluble manganate or permanganate. The residual yellow solution color was caused by chromate (CrO_4^{2-}).

Adding peroxide resulted in some bubbling in the solution; hydroxylamine addition gave a small amount of white precipitate at the point of introduction. The precipitate [probably $\text{Al}(\text{OH})_3$ precipitated by the acidic hydrochloride salt] dissolved rapidly with mixing.

The suspensions were centrifuged and the supernatant solutions were analyzed for bulk metal and radionuclide concentrations. The results of the analyses for one manganate test solution are shown in Figures 4-8 and 4-9. Similar results were obtained for the other two test solutions.

Test data indicate that hydroxylamine and peroxide removed manganese (see Figure 4-8) and plutonium (see Figure 4-9) from solution. Concentration reductions for both metals with each reagent were approximately a factor of three hundred. Significantly, chromium solution concentrations did not decrease by reduction of chromate to the poorly soluble $\text{Cr}(\text{OH})_3$. Thus, the beneficial effect of chromium solubilization by C/D was not negated by the plutonium/manganese reductive coprecipitation. Some coprecipitation of ^{90}Sr also occurred. Concentrations of other metals or radionuclides were not affected by using the reductants.

Figure 4-8. Concentration of Bulk Waste Constituents in Tank 102-SY C/D Solution Before and After Treatment with Peroxide and Hydroxylamine.

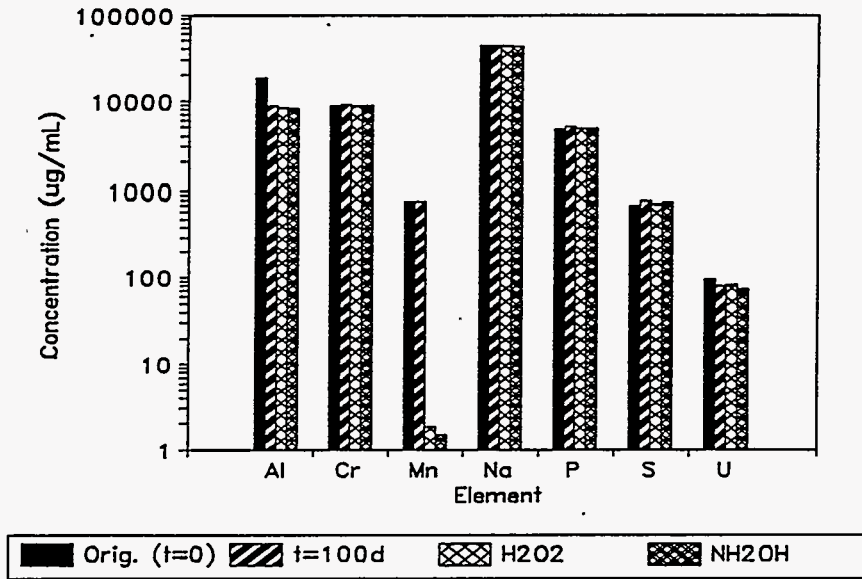
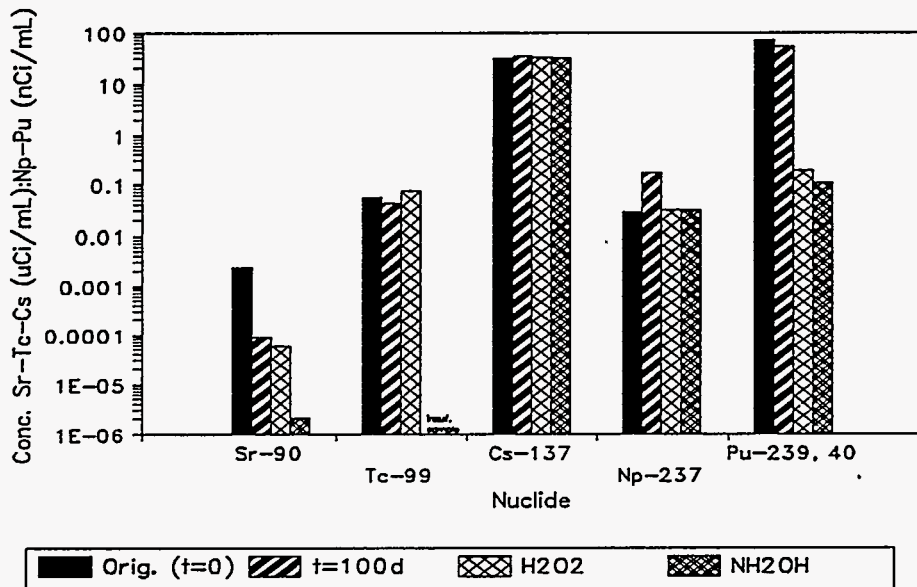


Figure 4-9. Concentrations of Radionuclides in Tank 102-SY C/D Solution Before and After Treatments with Peroxide and Hydroxylamine.



Results from initial scoping tests for coprecipitation indicate that plutonium solubilized by C/D may be rapidly and efficiently removed from solution. However, analytical results for neptunium were near the analytical detection limit and were difficult to interpret. Further tests using neptunium-spiked simulant waste were performed to clarify the behavior of neptunium.

4.2.2 Coprecipitation Tests with Neptunium- and Plutonium-Spiked Simulant

The behavior of neptunium in C/D processing has been difficult to determine because of its low concentration in tested Hanford Site tank wastes. Some solubilization of neptunium by C/D is expected based on the similarity of its chemistry to that of plutonium. Neptunium is easier to oxidize in alkaline solution than plutonium (Peretrukhin et al. 1995); therefore, it would be more susceptible to oxidative dissolution by C/D processing.

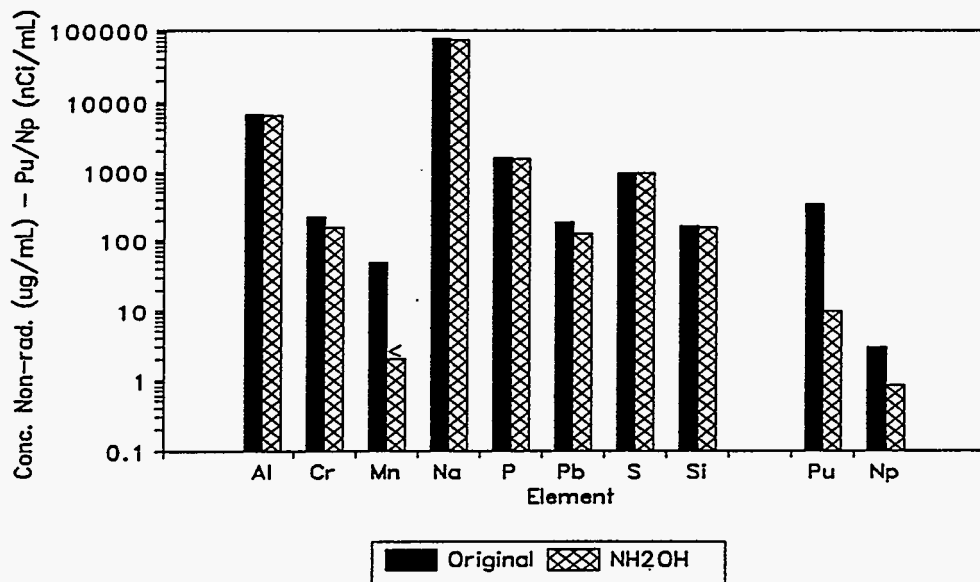
The composition of a comprehensive Hanford Site tank waste simulant containing the most prevalent 27 chemical components of the tank waste (over 99.99 mole percent) has been described (Delegard et al. 1994a). Nitric acid solutions of plutonium(IV) and neptunium(V) spike were added to an aliquot of the comprehensive simulant and the spiked simulant processed by C/D.

The C/D product solution was separated for analysis, and a 5 mL aliquot of the clarified solution was treated with 3 M hydroxylamine hydrochloride. Upon adding a drop of the hydroxylamine, the color changed immediately from blue-green to yellow. No precipitate appeared even after 30 minutes. The treated solution was allowed to stand about 80 hours after which a fine brown precipitate appeared. The original C/D product and the hydroxylamine-treated supernatant solutions were analyzed.

Equal concentrations (approximately 0.004 g/L or 1.8×10^{-5} M) of plutonium and neptunium were found in the C/D product solution. The equal concentrations give evidence for similar solubility-limiting phases for these two neighboring actinides.

The results of the solution analyses before and after hydroxylamine treatment show manganese and plutonium solution concentrations dropped approximately a factor of thirty by treatment with hydroxylamine; neptunium concentrations dropped approximately a factor of three (see Figure 4-10). Chromium solution concentrations also decreased approximately 20 percent and lead decreased approximately 30 percent. The slowness of the precipitation reaction may be caused by the low initial concentration of the dissolved manganese or by complexation by excess hydroxylamine. Further tests must be conducted to determine the cause of slowness of the precipitation reaction, whether heating improves the rate of reduction/coprecipitation and to confirm whether hydroxylamine causes the undesired precipitation of chromium.

Figure 4-10. Concentrations of Bulk Constituents and Radionuclides in Simulant C/D Solution Before and After Treatment with Hydroxylamine.



4.3 DETERMINATION OF PLUTONIUM AND NEPTUNIUM SOLUTION SPECIES

The solubilization of plutonium occurring in the C/D processing of genuine tank waste has been attributed to the formation of soluble anionic oxidized species. Similar solubilities of plutonium and neptunium, found in the C/D processing of spiked simulant wastes, imply that similar chemistries exist for these two neighboring actinides. On this basis, tests were performed to identify plutonium and neptunium solution species created in the C/D of simulant waste materials.

Because spectrophotometric techniques are noninvasive and do not disturb the solution chemistry, they are ideally suited to identify dissolved species. Separate plutonium- and neptunium-spiked simulant wastes were prepared, subjected to C/D processing, and the product solution was analyzed spectrophotometrically (using a spectrophotometer with a lower detection limit of approximately 0.0001 absorbance units for a 1-cm cell).

To facilitate observation of suspected actinide species, which have low concentrations and low molar absorptivities, simulant wastes with no strongly absorbing chromophores were prepared. A Hanford Site tank waste composite sludge was created according to the published recipe (Delegard et al. 1994a) but from which iron, chromium, nickel, manganese, and bismuth had been omitted. A surrogate waste simulating the composition of tank 102-SY waste and including added NaNO₃ (but with iron, chromium, and manganese omitted) also was created. The compositions of the two simulant waste types are given in Table 4-3.

Table 4-3. Compositions of Waste Simulants Used in Speciation Tests.

Constituent	Concentration (moles/kg)	
	Composite Simulant	Tank 102-SY Simulant with added NaNO ₃
Al	0.52	0.58
B	0.00074	--
Ca	0.011	0.023
Ce	0.0053	--
K	0.073	0.038
Mg	0.00069	--
Na	8.91	5.66
Pb	0.0067	--
Si	0.056	0.026
Sr	0.0013	--
Cl ⁻	0.038	0.037
F ⁻	0.18	0.018
NO ₂	0.65	0.38
NO ₃	5.34	3.69
PO ₄ ³⁻	0.29	0.085
SO ₄ ²⁻	0.065	0.21
CO ₃ ²⁻	0.14	0.28
OH ⁻	1.4	0.55
TOC*	0.20	0.36
H ₂ O	10.9	26.2

Note:

* Added as acetate.

The absorption spectra of the C/D product solutions were gathered for both simulant waste types. The neptunium-spiked tank waste composite sludge had a dissolved neptunium concentration of 0.034 g/L (equivalent to 1.4×10^{-4} M or approximately 20 nCi/g) and

showed a spectrum matching a published spectrum of Np(V) (Cohen and Fried 1969). The corresponding plutonium-spiked material had a dissolved plutonium concentration of approximately 0.028 g/L (1.2×10^{-4} M or approximately 1600 nCi/g). The spectrum was less distinct but resembled a published spectrum of Pu(V) (Bourges 1972).

A non-spiked sample of the tank 102-SY/NaNO₃ simulant also underwent C/D processing. Colorless bubbles were observed in the product solution. The neptunium-spiked tank 102-SY/NaNO₃ C/D product solution had approximately 0.26 g/L dissolved neptunium. The solution was tea colored and had an intense absorption in the spectrum below approximately 700 nm wavelength. Over several days, the solution color faded and the characteristic absorption caused by Np(V) emerged. A quantity of white precipitate also appeared in the sample vial.

Based on the bubbling observed in the non-spiked C/D solution and the intense absorption in the lower wave lengths of the neptunium-spiked C/D solution, peroxide complexes of neptunium were suspected. To confirm this, a drop of 1 M H₂O₂ was added directly into the spectrophotometric cuvette containing approximately 3 mL of the clarified Np(V) solution. The solution immediately changed to the original tea color with a spectrum matching the original spectrum, except for intensity. Adding 100 μ L of 1 M H₂O₂ to the 10 mL sample containing the neptunium C/D product almost instantly produced the original dark tea color, dissolved the previously observed white precipitate, and gave the original spectrum. The assignment of the tea-colored neptunium spectrum to a Np(V) peroxide complex was corroborated by spectra and other experimental results found in a published report (Musikas 1976).

The plutonium-spiked tank 102-SY/NaNO₃ simulant gave a dissolved plutonium concentration of 0.009 g/L. No readily identifiable spectrum was observed. The work of Musikas (1976) indicates that plutonium(VI) and (V) in alkaline solution form peroxide complexes of only fleeting existence and are rapidly reduced to form Pu(IV) precipitates.

The speciation coprecipitation test data reported in Section 4.2.2 substantiate the use of chemical reductants, such as peroxide or hydroxylamine, to effect reduction and (co)precipitation of plutonium. The coprecipitation of neptunium is more difficult and complicated by the formation of relatively stable peroxide complexes. In analogy with the well-known reactions of peroxide with actinides in acid media, the plutonium and neptunium peroxide complexes may be destroyed readily by heating.

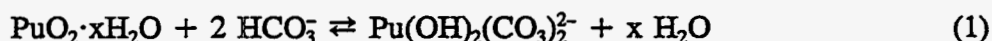
4.4 CARBONATE COMPLEXES OF PLUTONIUM IN ALKALINE SOLUTION

Quantitative evidence of the formation of carbonate complexes of Pu(IV) in alkaline solution has recently been presented in the technical literature (Yamaguchi et al. 1994). The evidence consists of enhanced solution concentration of plutonium as carbonate or bicarbonate concentrations increase.

In the experiments of Yamaguchi et al. (1994), plutonium valence was controlled to the (IV) state by use of NaNO_2 . Tests showed the solid phase contained no carbonate; on this basis, the solid phase $\text{PuO}_2 \cdot x\text{H}_2\text{O}$ was inferred though no spectrophotometry was performed to identify the valence of the dissolved plutonium species, plutonium in the clarified (filtered, at 450 nm and 3 nm pore size) solutions was identified as being in the (IV) state by TTA (thenoyltrifluoroacetone) extraction.

The plutonium solution concentration increased in the presence of carbonate and bicarbonate, and the plutonium concentration was proportional to the square of the carbonate or bicarbonate concentration. Two regimes of increasing plutonium concentration were identified: in the range pH 9.4 to 10.1 (where bicarbonate, HCO_3^- , is predominant) and in the range pH 12 to 13 (where carbonate, CO_3^{2-} , is predominant).

On the basis of the pH dependencies and the observed dependencies of the plutonium solution concentration on (bi)carbonate concentration, two reactions were proposed:



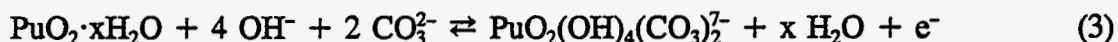
in bicarbonate (pH 9.4 to 10.1), $\log K = -2.7 \pm 0.5$; and



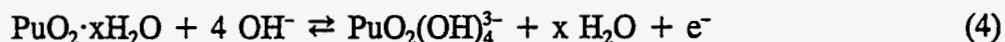
in carbonate (pH 12 to 13), $\log K = -4.98 \pm 0.31$.

Thus, the solubility of the plutonium solid phase ($\text{PuO}_2 \cdot x\text{H}_2\text{O}$) was found to increase in proportion to the square of the bicarbonate or carbonate concentrations.

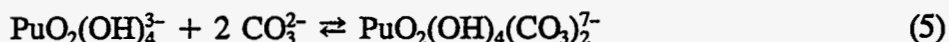
The solubility of $\text{PuO}_2 \cdot x\text{H}_2\text{O}$ in the system $\text{NaOH}/\text{Na}_2\text{CO}_3$ has been reported previously (Delegard 1985). In this study, Pu(V) species had been observed in concentrated NaOH solutions having no added carbonate; therefore, the solubilities observed in mixed $\text{NaOH}/\text{Na}_2\text{CO}_3$ solutions were modeled on the net reaction (3) involving Pu(V):



where reaction (3) is a combination of reactions (4)



$\log K = -8.93 \pm 0.16$; and (5)



$\log K = 1.13 \pm 0.20$.

Reaction (4) was observed in pure concentrated (1 to 15 M) NaOH aqueous systems. The solid phase (poorly crystalline PuO₂) was identified by X-ray diffraction, and the solution species was identified as Pu(V) by spectrophotometric techniques. Reaction (5) was suggested by the enhanced solubility shown when large concentrations of Na₂CO₃ (0.25 to 1.06 M) were included in 2.90 to 4.94 M NaOH solutions. No spectrophotometric or other check of the valence of the dissolved plutonium species was performed for the carbonate experiments. On the basis of the experiments in the "pure" NaOH solution, Pu(V) species were assumed in the NaOH/Na₂CO₃ solutions.

Because of the large negative charge (-7) on the PuO₂⁺ central species and because PuO₂⁺ has the lowest tendency towards complexation in aqueous media of any plutonium valence, the existence of the proposed mixed hydroxide/carbonate complex described in reactions (3) and (5) is unlikely. Spectrophotometric studies of Pu(V) in concentrated (1 to 2 M) Na₂CO₃ (Varlashkin et al. 1984) show that as NaOH concentration increases stepwise from 0.04 to 1.04 to 1.75 to 2.29 M, the Pu(V) spectra change to resemble closely but not identically the spectra observed in 4 M NaOH (Bourges 1972) and 15 M NaOH (Delegard 1985) having no added carbonate. Therefore, the observations of Varlashkin et al. (1984) also give evidence for the decreasing stability of possible Pu(V) carbonate complexes as NaOH concentrations increase.

Based on the research of Yamaguchi et al. (1994), the original experimental data of Delegard (1985) were re-examined. The enhanced solubility of plutonium observed in the presence of carbonate was calculated to be the difference between the total measured plutonium solution concentration and the concentration expected solely by hydrolysis reaction (4).

Solubility was observed to change as a function of time (Delegard 1985); therefore, the solubility caused by reaction (4) in the pure NaOH system was calculated based on the data taken at 153 days' equilibration. This equilibration time closely matched the 156-day equilibration time allowed for the NaOH/Na₂CO₃ system. Using data taken from Table A-1 of Delegard (1985), the plutonium solution concentration caused by the Pu(V) hydroxide complex was calculated. The calculated solubility caused by reaction (4) can be described accurately (R² = 0.996) by the function:

$$\log [\text{Pu}] = 1.997 \log (\text{NaOH}) - 7.543 \quad (6)$$

where [] indicates molal concentrations and () indicates chemical activities.

The data in Table 4-4 are derived from Table A-6 of Delegard (1985). The contribution of the Pu(V) hydroxide complex concentration (designated [PuOH] in Table 4-4) to the total solution concentration (designated [Pu]) was determined by equation (6). The amount of plutonium solution concentration attributed to the carbonate complex (that is, [PuCO₃]) was

calculated as the difference between the total concentration and the concentration due to the hydroxide complex:

$$[\text{PuCO}_3] = [\text{Pu}]_t - [\text{PuOH}] \quad (7)$$

The calculated plutonium carbonate complex concentration can be plotted versus the carbonate chemical activity ($a_{\text{Na}_2\text{CO}_3}$); the slope of the plot should be proportional to the ligand:metal ($\text{CO}_3^{2-}:\text{Pu}$) ratio. Such a plot for the data derived from Delegard (1985) is shown in Figure 4-11. Figure 4-11 also gives data obtained by Yamaguchi et al. (1994). In the case of the Yamaguchi et al. (1994) data, where total ionic strength was limited to 0.1 molal (m), the molar (M) concentrations of plutonium were assumed to be equal to their molal concentrations and the carbonate molar concentrations were assumed to be equal to their activities.

Figure 4-11. Effect of Carbonate on Plutonium Compound Solubility.

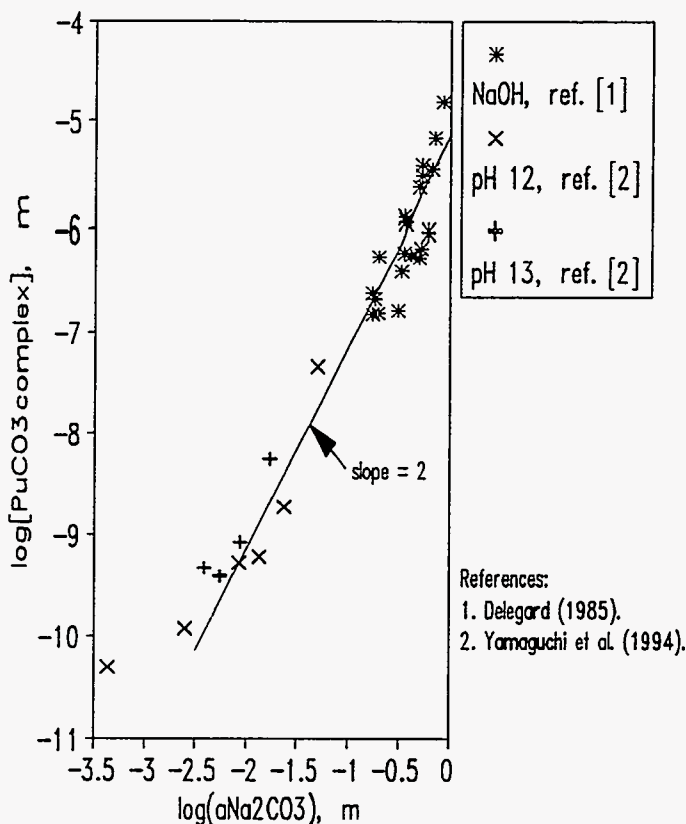


Table 4-4. Experimental and Derived Data for Plutonium Compound Solubility in the NaOH/Na₂CO₃ Aqueous System.

[NaOH], M	[Na ₂ CO ₃], M	p _{NaOH} , m	p _{Na2CO3} , m	log[Pu], m	log[PuOH], m	log[PuCO ₃], m	log(a _{Na2CO3}), m
2.90	0.701	2.39	0.416	-6.124	-6.788	-6.230	-0.381
2.95	0.512	2.38	0.310	-6.488	-6.791	-6.787	-0.509
2.95	0.912	2.45	0.522	-6.058	-6.766	-6.153	-0.282
2.95	1.050	2.58	0.610	-5.940	-6.721	-6.019	-0.215
2.96	0.261	2.46	0.172	-6.377	-6.763	-6.607	-0.764
3.24	0.255	2.81	0.172	-6.424	-6.647	-6.820	-0.764
3.47	0.515	3.18	0.335	-6.157	-6.540	-6.389	-0.475
3.47	0.783	3.32	0.496	-6.061	-6.503	-6.256	-0.305
3.94	0.515	3.92	0.347	-5.975	-6.359	-6.207	-0.460
3.94	0.771	4.02	0.500	-5.567	-6.337	-5.648	-0.301
3.99	1.060	4.29	0.673	-5.421	-6.280	-5.486	-0.172
4.00	0.260	3.88	0.185	-6.192	-6.367	-6.670	-0.733
4.43	0.513	4.85	0.362	-5.745	-6.174	-5.947	-0.441
4.52	0.766	5.29	0.531	-5.358	-6.099	-5.445	-0.275
4.69	1.060	5.76	0.712	-5.118	-6.025	-5.175	-0.148
4.75	0.765	5.57	0.527	-5.421	-6.054	-5.536	-0.278
4.80	0.484	5.61	0.354	-5.674	-6.048	-5.913	-0.451
4.85	0.250	5.57	0.194	-5.983	-6.054	-6.805	-0.712
4.89	1.050	6.72	0.852	-4.779	-5.891	-4.814	-0.070
4.94	0.257	5.78	0.201	-5.816	-6.022	-6.239	-0.697

The effect of carbonate on the plutonium solubility observed in the data derived from Delegard (1985) is consistent with the effect carbonate has on plutonium solubility on the pH 12 and 13 data of Yamaguchi et al. (1994) (see Figure 4-11). The data also are

consistent with a 2:1 ratio in the postulated $\text{CO}_3^{2-}:\text{Pu(IV)}$ complex. The coincidence of these results and the carbonate complexation reactions proposed by Yamaguchi et al. (1994) suggest that a similar mechanism and complex operates in the $\text{NaOH}/\text{Na}_2\text{CO}_3$ system.

Further confirmation tests were attempted, based on spectrophotometric verification of Pu(IV) in aqueous $\text{NaOH}/\text{Na}_2\text{CO}_3$ solutions. To ensure initial formation of a Pu(IV) carbonate complex, Pu(IV) -spiked 1.0 M NaHCO_3 solutions were prepared to give approximately 1.7×10^{-4} M Pu final concentration. All the plutonium spike dissolved. The resulting solution spectrum matched spectra of Pu(IV) in 1 M NaHCO_3 previously published (Wester and Sullivan 1983 and Varlashkin et al. 1984). The molar absorptivity of the prominent 487 nm peak was approximately 70 L/cm-mol (versus approximately 90 L/cm-mol found by Wester and Sullivan [1983] and Varlashkin et al. [1984]).

Sufficient 50 percent NaOH solution was added to this solution to make the resulting solution 4.1 M NaOH and 0.80 M Na_2CO_3 . Grass green plutonium solids formed immediately. After mixing and centrifuging for clarification, absorption spectra were gathered. Spectra also were obtained for solutions of 4.7 M NaOH and 1.2 M Na_2CO_3 to which Pu(IV) spike was added. Green plutonium solids precipitated immediately on introduction of the Pu(IV) spike. Finally, spectra were obtained for Pu(IV) -spiked solutions of 4.7 M NaOH and 1.2 M Na_2CO_3 which were made approximately 0.1 M in hydroxylamine (NH_2OH) as a reductant.

The plutonium spectra obtained in all highly alkaline solutions were weak. Assignment of peaks was complicated by the possible existence of several dissolved plutonium species (for example, the Pu(V) -hydroxide complex, possible Pu(V) -carbonate complexes, as well as the Pu(IV) -carbonate complex being sought). Summaries of the spectral observations are given in Table 4-5.

Table 4-5. Spectral Features for Pu in $\text{NaOH}/\text{Na}_2\text{CO}_3$ Solutions.

Solution, [NaOH]/[Na ₂ CO ₃]	Equil. Time	Peak Location (nm) and Absorbance, 1 cm Cell				
		1160-1170	980	950	600-700	490-550
4.1/0.8	10 min.	0.003	0.0015	0.0008	0.003	0.001
4.1/0.8	10 min.	0.005	0.001	0.0005	0.004	0.0008
4.7/1.2	82 days	0.0005	0.0001	ND	ND	ND
4.7/1.2 - NH_2OH	10 min.	0.006	0.0015	0.0008	ND	ND
4.7/1.2 - NH_2OH	30 min.	0.007	0.002	0.001	ND	ND
4.7/1.2 - NH_2OH	2 hours	0.005	0.0015	0.001	ND	ND

Note:

ND: not detectible; <0.0001 absorbance unit.

The most prominent peak, observed in all spectra, occurred around 1170 nm. This peak generally was accompanied by a doublet, observed in the near infrared region and centered around 980 and 950 nm. Broad absorbance peaks were found in the visible region (at 490 to 550 nm and 600 to 700 nm) for the tests in 4.0 M NaOH and 0.8 M Na₂CO₃. The peaks observed around 600 to 700 nm were approximately as intense as the peak at 1170 nm.

For the sample aged 82 days and for all the samples to which hydroxylamine reductant had been added, no spectral features were observed in the visible region (below 800 nm). Because Pu(V) solution species should not exist in the presence of hydroxylamine and the peaks appearing in the visible region disappeared when hydroxylamine was added, the peaks observed in the visible region were probably caused by Pu(V). The absence of peaks in the visible region for the aged sample implies that Pu(V) dissolved species do not contribute significantly to the plutonium solution concentration. According to the solubility data in Table 4-4, the expected concentration of the Pu(V)-hydroxide complex would be approximately 10 percent as much as the plutonium carbonate complex.

In summary, these limited experiments give evidence for the existence, in aged strongly alkaline solution, of Pu(IV)-carbonate complexes but not of Pu(V)-carbonate complexes. Further experiments are required to determine more confidently the existence and behavior of possible Pu(IV)-carbonate complexes in highly alkaline solution. One such experiment would be the determination of Pu^(IV)O₂·xH₂O solubility in mixed NaOH/Na₂CO₃ solutions in the presence of reductants.

4.5 WORK WITH THE UNIVERSITY OF IDAHO PLASMA LABORATORY

Engineering lab scale calcination studies of simulated Hanford Site tank wastes were conducted at the University of Idaho under the direction of Professor Patrick Taylor. Mr. R. S. Goheen of the Pacific National Laboratory coordinated the work. Results of the studies have been reported (Goheen et al. 1995).

In the University of Idaho tests, the kinetics of fundamental calcination chemical reactions; nitrate/nitrite decomposition, conversion of Al(OH)₃ to NaAlO₂, oxidation of Cr(OH)₃ to form CrO₄²⁻, and organic carbon destruction; were investigated as functions of calciner power and residence time. A 50-kilowatt nontransferred plasma torch was used to provide the rapid heating required for the tests. The nontransferred torch means the plasma arc does not pass through the material to be heated. Instead, gas heated by the plasma arc is used to transfer heat to the waste. This heat transfer method is identical to that used in earlier pilot-scale calcination tests of tank 101-SY simulant waste conducted in November 1992 and November 1993 at the Westinghouse Science and Technology Center in Waltz Mill, Pennsylvania (Delegard et al. 1994b).

The principal investigator provided information to the researchers at the University of Idaho on compositions of candidate Hanford Site waste simulants, analytical methods, and goals of C/D processing. He also visited the laboratory at the Idaho campus for discussions with

Professor Taylor and his staff, reviewed the interim and final reports, and arranged a technical seminar by the investigator (on C/D chemistry) and Professor Taylor (on the plasma reactor results) at the Hanford Site for Tank Waste Remediation System program managers and engineers. Tank Waste Remediation System funding of the plasma calciner tests was terminated on 31 January 1995 with the publication of the test results (Goheen et al. 1995).

4.6 TECHNICAL PAPERS

An external release document on laboratory studies of C/D processing of genuine Hanford Site tank wastes from tanks 104-T and 111-T (from the bismuth phosphate process) and tank 102-SY (from the Plutonium Finishing Plant) was prepared and issued in January 1995 (Delegard 1995b). The document also compared the new results with results from earlier studies of the C/D of tank 101-SY (a complexant concentrate), tank 104-S (a REDOX process sludge), and tank 107-BX (another bismuth phosphate process waste).

Contributions were made to a paper on plutonium compound solubility in alkaline solution prepared for the 19th Annual Actinide Separations Conference in June 1995 (Rao et al. 1995).

A paper summarizing the laboratory studies of C/D over the past several years has been accepted for presentation at the Tank Waste Chemistry symposium of the Industrial and Engineering Chemistry Division of the 211th national meeting of the American Chemical Society in March 1996 (Delegard 1995a).

5.0 ACKNOWLEDGEMENTS

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APPENDIX A

**RESULTS OF ESW AND C/D TESTS
OF WASTES FROM TANK 104-S**

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A-3

C/D of 104-S with NaNO3
ESW of 104-S

From WHC-N-656 1; pp. 117 and 118

	Sample	Time	WtCrucB4	WtCrucSyn	Wtw/NO3	Wt129C	Wt850C	WtTube	WtTubeLI	WtCrucAft	CrucWtL	CrAlLoss
104-S ESW	DES104W/J2340	Wash	14.137	19.138				13.979	53.264			
	DES104L/J2454	Leach						14.360	47.160			
	DES104R/J2341	Rinse						14.054	45.010			
104-S/CD	104SCDA/J2350	20	63.727	73.758	80.888	75.273	72.520	14.061	48.978	63.722	0.005	0.003
	104SCDB/J2351							14.061	35.925			
									14.061	25.832		

	Sample	Time	NetWt	FrLoss128	FrLoss850	WtSolids	WtSoln	WtDryTub	WtDrySol	WtAl(OH)3	Soln.Vol.	Soln.Dens	Wt%Solids	g sample	mLsol'n./
104-S/ESW	DES104W/J2340	Wash	4.999				39.285	14.563	0.468		38.5	1.020	9.122	4.451845	
	DES104L/J2454	Leach	4.999				32.770				30.0	1.062		3.46897	
	DES104R/J2341	Rinse	4.999				30.956				31.0	0.999		3.584602	
104-S/CD	104SCDA/J2350	20	10.0110	0.3272	0.4876	8.7930	32.364	14.549	0.250	0.008	27.000	1.199	2.497	1.573335	
	104SCDB/J2351		10.0110				29.600				30.500	0.970		1.777286	

	Sample	Time	NO2 (ug/mL)	NO3 (ug/mL)	OH (ug/mL)	F (ug/mL)	Cl (ug/mL)	PO4 as P (ug/mL)	SO4 as S (ug/mL)	TOC (ug/mL)	TIC (ug/mL)
104-S/ESW	DES104W/J2340	Wash	2.93E+03	1.68E+04	1.30E+03		2.57E+02		1.19E+02	3.31E+02	2.42E+02
	DES104L/J2454	Leach	1.44E+02	3.18E+02	4.37E+04	1.25E+01	1.12E+01		9.52E+01	1.42E+02	
	DES104R/J2341	Rinse	4.33E+01	6.86E+01	2.98E+03		6.09E+00		3.58E+01	8.30E+01	
104-S/CD	104SCDA/J2350	20	7.67E+04	8.58E+04	6.68E+03				9.70E+01		3.96E+02
	104SCDB/J2351		5.44E+03	4.96E+03	6.69E+02		4.18E+01				4.70E+01

	Sample	Time	NO2 (Wt%)	NO3 (Wt%)	OH (Wt%)	F (Wt%)	Cl (Wt%)	PO4 as P (Wt%)	SO4 as S (Wt%)	TOC (Wt%)	TIC (Wt%)	H2O (Wt%)
104-S	DE104SBL/J2364		1.90E+00	1.42E+01	9.36E-01	0.00E+00	1.97E-01	0.00E+00	3.03E+02	7.55E-01	2.78E-01	60.5
Enh.SlWashes			5.67E-01	0.00E+00	4.21E+01	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00
NaNO3			0.00E+00	7.29E+01	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	
104S/ESW	4.999gS&3.6491gNaOH/NO2		1.34E+00	8.21E+00	1.83E+01	0.00E+00	1.14E-01	0.00E+00	1.75E+02	4.36E-01	1.81E-01	
104S/NaNO3	10.010gS&7.150gNaNO3		1.11E+00	3.87E+01	5.46E-01	0.00E+00	1.15E-01	0.00E+00	1.77E+02	4.40E-01	1.62E-01	3.53E+01
104-S/ESW	DES104W/J2340	Wash	1.30E+00	7.48E+00	5.79E-01	0.00E+00	1.14E-01	0.00E+00	5.30E-02	1.47E-01	1.08E-01	
	DES104L/J2454	Leach	5.00E-02	1.10E-01	1.52E+01	4.34E-03	3.89E-03	0.00E+00	0.00E+00	3.30E-02	4.93E-02	
	DES104R/J2341	Rinse	1.55E-02	2.48E-02	1.07E+00	0.00E+00	2.18E-03	0.00E+00	0.00E+00	1.28E-02	2.96E-02	
	Sum Enh.Sl.Wash		1.37E+00	7.81E+00	1.68E+01	4.34E-03	1.20E-01	0.00E+00	5.30E-02	1.93E-01	1.87E-01	
104-S/CD	104SCDA/J2350	20	1.21E+01	1.36E+01	1.06E+00	0.00E+00	0.00E+00	0.00E+00	1.53E-02	0.00E+00	6.26E-02	
	104SCDB/J2351		9.67E-01	8.80E-01	1.19E-01	0.00E+00	7.43E-03	0.00E+00	0.00E+00	0.00E+00	8.35E-03	
	Sum C/D 104-S		1.30E+01	1.43E+01	1.17E+00	0.00E+00	7.43E-03	0.00E+00	1.53E-02	0.00E+00	7.10E-02	
	Fract.Dis. - ESW		1.02E+00	9.28E-01	9.17E-01	ERR	1.06E+00	ERR	3.02E-04	4.43E-01	1.16E+00	
	Fract.Dis. - C/D		1.18E+01	3.71E-01	2.14E+00	ERR	6.48E-02	ERR	8.63E-06	0.00E+00	4.38E-01	

WHC-EP-0882

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Sample	Time	ESW											
		Na (ug/mL)	Al (ug/mL)	Si (ug/mL)	less than Fe (ug/mL)	Cr (ug/mL)	less than Mn (ug/mL)	less than Ni (ug/mL)	S (ug/mL)	less than Ca (ug/mL)	U (ug/mL)	K (ug/mL)	
104-S/ESW	DES104W/J2340	Wash	9.75E+03	5.29E+02	1.41E+01	2.00E-01	2.00E+02	2.00E-01	4.00E-01	3.53E+01	2.00E-01	<.000037	2.91E+01
	DES104L/J2454	Leach	6.03E+04	6.15E+03	7.54E+01	2.00E-01	6.03E+00	2.00E-01	4.00E-01		2.00E-01	4.48E-01	
	DES104R/J2341	Rinse	4.23E+03	3.96E+02	8.50E+00	2.00E-01	2.64E-01	2.00E-01	4.00E-01	1.20E+00	2.00E-01	3.30E-02	
104-S/CD	104SCDA/J2350	20	8.72E+04	2.17E+04	3.88E+02	2.00E-01	5.43E+02	8.34E+01	4.00E-01	9.63E+01	2.00E-01	9.51E+01	
	104SCDB/J2351		5.65E+03	1.72E+03	1.36E+01	2.00E-01	3.63E+01	3.52E+00	4.00E-01	6.72E+00	2.00E-01	7.52E+00	3.97E+00
Sample	Time	Na (Wt%)	Al (Wt%)	Si (Wt%)	less than Fe (Wt%)	Cr (Wt%)	less than Mn (Wt%)	less than Ni (Wt%)	S (Wt%)	less than Ca (Wt%)	U (Wt%)	K (Wt%)	
104-S	DE104SBL/J2364	7.00E+00	6.78E+00	2.08E-01	1.77E-01	1.57E-01	7.95E-02	4.89E-01	2.69E-02	4.89E-02	2.48E-01		
Enh.SLWashes		5.73E+01	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	
NaNO3		2.71E+01	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	
104S/ESW	4.999gS&3.6491gNaOH/NO2	2.82E+01	3.92E+00	1.19E-01	1.02E-01	9.08E-02	4.60E-02	2.83E-01	1.55E-02	2.83E-02	1.42E-01	0.00E+00	
104S/NaNO3	10.010gS&7.150gNaNO3	1.54E+01	3.98E+00	1.20E-01	1.03E-01	9.16E-02	4.64E-02	2.85E-01	1.57E-02	2.85E-02	1.44E-01	0.00E+00	
104-S/ESW	DES104W/J2340	Wash	4.34E+00	2.36E-01	6.28E-03	8.90E-05	8.90E-02	8.90E-05	1.78E-04	1.57E-02	8.90E-05	0.00E+00	1.30E-02
	DES104L/J2454	Leach	2.09E+01	2.13E+00	2.82E-02	6.94E-05	2.09E-03	6.94E-05	1.39E-04	0.00E+00	6.94E-05	1.55E-04	0.00E+00
	DES104R/J2341	Rinse	1.52E+00	1.42E-01	3.05E-03	7.17E-05	9.48E-05	7.17E-05	1.43E-04	4.30E-04	7.17E-05	1.18E-05	0.00E+00
Sum Enh.SLWash		2.68E+01	2.51E+00	3.55E-02	2.30E-04	9.12E-02	2.30E-04	4.60E-04	1.61E-02	2.30E-04	1.67E-04	1.30E-02	
Percent Dissolved ESW		2.79E+01	1.84E+00	2.65E-02	1.03E-04	8.78E-02	1.29E-04	1.29E-03	1.55E-02	3.25E-04	7.78E-05	0.00E+00	
104-S/CD	104SCDA/J2350	20	1.37E+01	3.41E+00	6.10E-02	3.15E-05	8.54E-02	1.31E-02	6.29E-05	1.52E-02	3.15E-05	1.50E-02	0.00E+00
	104SCDB/J2351		1.00E+00	3.06E-01	2.42E-03	3.55E-05	6.45E-03	6.28E-04	7.11E-05	1.19E-03	3.55E-05	1.34E-03	7.06E-04
Sum C/D 104-S		1.47E+01	3.72E+00	6.35E-02	6.70E-05	9.19E-02	1.37E-02	1.34E-04	1.63E-02	6.70E-05	1.63E-02	7.06E-04	
Percent Dissolved C/D		1.52E+01	3.60E+00	4.81E-02	3.53E-05	9.16E-02	8.15E-03	7.83E-04	1.57E-02	9.03E-05	4.27E-02	0.00E+00	
Fract.Diss. - ESW		9.49E-01	6.41E-01	2.98E-01	2.25E-03	1.01E+00	5.01E-03	1.63E-03	1.04E+00	8.14E-03	1.18E-03	ERR	
Fract.Diss. - C/D		9.59E-01	9.41E-01	5.28E-01	6.49E-04	1.00E+00	2.98E-01	4.70E-04	1.04E+00	2.35E-03	1.14E-01	ERR	
Sample	Time	Na	Al	Si	Fe	Cr	Mn	Ni	S	Ca	U		
104-S	Wash	18.0	4.4	3.9	0.0	94.4	0.1	0.2	97.3	0.4	0.0		
	Leach	77.1	40.0	16.4	0.0	2.2	0.1	0.1	0.0	0.3	0.1		
	Rinse	5.6	2.7	1.9	0.0	0.1	0.1	0.1	2.7	0.4	0.0		
Sample	Time	Na (ug/g)	Al (ug/g)	Si (ug/g)	less than Fe (ug/g)	Cr (ug/g)	Mn (ug/g)	Ni (ug/g)	S (ug/g)	Ca (ug/g)	U (ug/g)	K (ug/g)	
Residues													
104-S/ESW	DESWS1/J2344	0	3.81E+04	3.10E+05	1.36E+04	2.51E+04	3.40E+02	9.17E+03	1.10E+04	0.00E+00	2.17E+03	3.35E+04	
104-S/CD	DE104CD1/J2345	20	6.33E+04	1.47E+05	3.61E+04	7.65E+04	1.10E-01	2.58E+04	1.95E+04	0.00E+00	8.45E+03	1.54E+04	
Sample	Time	Na	Al	Si	Fe	Cr	Mn	Ni	S	Ca	U	K	
104-S/ESW	Wt.Fraction	0											
	Solution		2.68E-01	2.51E-02	3.55E-04	2.30E-06	9.12E-04	2.30E-06	4.60E-06	1.61E-04	2.30E-06	1.67E-06	
	Residue		3.48E-03	2.83E-02	1.24E-03	2.29E-03	3.10E-05	8.36E-04	1.00E-03	0.00E+00	1.98E-04	3.06E-03	
104-S/CD	Solution	20	1.47E-01	3.72E-02	6.35E-04	6.70E-07	9.19E-04	1.37E-04	1.34E-06	1.63E-04	6.70E-07	1.63E-04	
	Residue		1.58E-03	3.67E-03	9.51E-04	1.96E-03	2.75E-09	6.44E-04	4.87E-04	0.00E+00	2.11E-04	3.85E-04	
Sample	Time	Na	Al	Si	Fe	Cr	Mn	Ni	S	Ca	U	K	
Fract.Diss.-ESW		9.87E-01	4.70E-01	2.22E-01	1.00E-03	9.67E-01	2.74E-03	4.57E-03	1.00E+00	1.15E-02	5.47E-04	1.00E+00	
Fract.Diss.-C/D		9.89E-01	9.10E-01	4.00E-01	3.42E-04	1.00E+00	1.76E-01	2.74E-03	1.00E+00	3.17E-03	2.98E-01	1.00E+00	

Cr(VI) (ug/mL)	Pb (ug/mL)	less than Mg (ug/mL)	Sr (ug/mL)	Ni (ug/mL)	wash/rinse						Less than	
					Sr-90 (uCi/mL)	Tc-99 (uCi/mL)	Cs-137 (uCi/mL)	Pu-239/40 (nCi/mL)	Am-241 (nCi/mL)	Np-237 (nCi/mL)		
1E+02					1.91E-03	1.78E-03	4.40E+00	3.55E-03	2.85E-02	<0.016		
7E+01					1.17E-01	3.14E-05	3.67E-01	7.39E-03	3.85E-02	<0.016		
5E+00					2.19E-02	<7.15e-6	1.39E-02	4.59E-03	2.32E-02	<0.0221		
5E+02					7.66E-04	4.76E-04	9.30E+00	1.93E+00	2.42E-02	6.67E-02		
1E+01	1.29E+00				1.30E-03	7.76E-05	6.87E-01	2.90E-02	2.46E-02	<.003		
Cr(VI) (Wt%)	Pb (Wt%)	Mg (Wt%)	Sr (Wt%)	Ni (Wt%)	Sr-90 (uCi/g)	Tc-99 (uCi/g)	Cs-137 (uCi/g)	Pu-239/40 (nCi/g)	Am-241 (nCi/g)	Np-237 (nCi/g)	Sample	
35E-01	4.85E-02		2.11E-02	4.89E-01	1.54E+02	1.20E-02	3.85E+01	1.99E+02	6.10E+01	<13	104-S DE104SBL/	
0E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	Enh.SLWashes	
0E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	NaNO3	
4E-02	2.80E-02	0.00E+00	1.22E-02	2.83E-01	8.90E+01	6.94E-03	2.23E+01	1.15E+02	3.53E+01	0.00E+00	104S/ESW 4.999gS&3.	
3E-02	2.83E-02	0.00E+00	1.23E-02	2.85E-01	8.98E+01	7.00E-03	2.25E+01	1.16E+02	3.56E+01	0.00E+00	104S/NaNO3 10.010gS&	
2E-01	0.00E+00	0.00E+00	0.00E+00	0.00E+00	8.50E-03	7.92E-03	1.96E+01	1.58E-02	1.27E-01	0.00E+00	104-S DES104W/J	
6E-03	0.00E+00	0.00E+00	0.00E+00	0.00E+00	4.06E-01	1.09E-04	1.27E+00	2.56E-02	1.34E-01	0.00E+00	DES104L/J	
2E-03	0.00E+00	0.00E+00	0.00E+00	0.00E+00	7.85E-02	0.00E+00	4.98E-02	1.65E-02	8.32E-02	0.00E+00	DES104R/J	
3E-01	0.00E+00	0.00E+00	0.00E+00	0.00E+00	4.93E-01	8.03E-03	2.09E+01	5.79E-02	3.44E-01	0.00E+00	Sum Enh.SI	
4E-02	0.00E+00	ERR	0.00E+00	0.00E+00	2.66E-01	4.47E-03	2.13E+01	3.48E-02	1.86E-01	ERR		
5E-02	0.00E+00	0.00E+00	0.00E+00	0.00E+00	1.21E-03	7.49E-04	1.48E+01	3.04E+00	3.81E-02	1.05E-01	104-S/NaNO3 104SCDA/	
2E-03	2.29E-04	0.00E+00	0.00E+00	0.00E+00	2.31E-03	1.38E-04	1.22E+00	5.15E-02	4.37E-02	0.00E+00	104SCDB/	
4E-01	2.29E-04	0.00E+00	0.00E+00	0.00E+00	3.52E-03	8.87E-04	1.59E+01	3.09E+00	8.18E-02	1.05E-01	Sum C/D 1	
3E-02	1.57E-03	0.00E+00	0.00E+00	0.00E+00	1.44E-03	1.39E-03	2.21E+01	2.07E+00	3.62E-02	0.00E+00		
8E+00	0.00E+00	ERR	0.00E+00	0.00E+00	5.54E-03	1.16E+00	9.40E-01	5.03E-04	9.74E-03	ERR	Fract.Diss. -	
8E+00	8.10E-03	ERR	0.00E+00	0.00E+00	3.91E-05	1.27E-01	7.06E-01	2.66E-02	2.30E-03	ERR	Fract.Diss. -	
					Sr-90	Tc-99	Cs-137	Pu-239/40	Am-241	Np-237		
					0.01	63.57	89.46	0.01	0.19	ERR		
					0.25	0.87	5.81	0.01	0.20	ERR		
					0.05	0.00	0.23	0.01	0.13	ERR		
Cr(VI) (ug/g)	Pb (ug/g)	Mg (ug/g)	Sr (ug/g)	Ni (ug/g)	Sr-90 (uCi/g)	Tc-99 (uCi/g)	Cs-137 (uCi/g)	Pu-239/40 (nCi/g)	Am-241 (nCi/g)	Np-237 (nCi/g)	Residues	Sample
1E+03	1.01E+03	0.00E+00	2.43E+03	1.10E+04	1.80E+03	4.86E-02	1.08E+01	2.10E+03	7.11E+02	<18.2	104-S ESW DESWS1/J	
1E+03	1.56E+03	2.29E+03	1.16E+04	1.95E+04	8.81E+03	1.43E-01	1.09E+01	6.81E+03	3.22E+03	<28.1	104-S C/D DE104CD1/	
Cr(VI)	Pb	Mg	Sr	Ni	Sr-90	Tc-99	Cs-137	Pu-239/40	Am-241	Np-237	Wt.Fraction	
23E-03	0.00E+00	0.00E+00	0.00E+00	0.00E+00	4.93E-01	8.03E-03	2.09E+01	5.79E-02	3.44E-01	0.00E+00	104-S ESW Solution	
0E+00	9.21E-05	0.00E+00	2.22E-04	1.00E-03	1.84E+02	4.43E-03	9.85E-01	1.92E+02	6.49E+01	0.00E+00	Residue	
04E-03	2.29E-06	0.00E+00	0.00E+00	0.00E+00	3.52E-03	8.87E-04	1.59E+01	3.09E+00	8.18E-02	1.05E-01	104-S C/D Solution	
0E+00	3.90E-05	5.72E-05	2.90E-04	4.87E-04	2.20E+02	3.57E-03	2.72E-01	1.70E+02	8.04E+01	0.00E+00	Residue	
Cr(VI)	Pb	Mg	Sr	Ni	Sr-90	Tc-99	Cs-137	Pu-239/40	Am-241	Np-237	Fract.Diss.-ESW	
0E+00	0.00E+00	ERR	0.00E+00	0.00E+00	2.99E-03	6.44E-01	9.55E-01	3.02E-04	5.27E-03	ERR	Fract.Diss.-ESW	
0E+00	5.56E-02	0.00E+00	0.00E+00	0.00E+00	1.60E-05	1.99E-01	9.83E-01	1.78E-02	1.02E-03	1.00E+00	Fract.Diss.-C/D	

APPENDIX B

**RESULTS OF ESW/PEROXIDE TESTS OF SIMULANT
TANK 101-SY WASTE**

WHC-EP-0882

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Chromium Hydroxide Dissolution from Sim. 101-SY: Cr(VI) Solution Measurements

Notebook WHC-N-656 1; pp. 121-122

Standard calibration curve for Beer-Lambert relationship for chromate in 0.5 M NaOH @ 372 nm.

Conc. (mM)	Absorbance	Fit	Res. (Abs-Fit)	Regression Output:	
9.99E-01		4.999		Constant	0.000703
7.50E-01	3.755	3.751	4.31E-03	Std Err of Y Est	0.001278
5.00E-01	2.490	2.499	-9.28E-03	R Squared	0.999978
2.50E-01	1.258	1.248	1.01E-02	No. of Observations	9
1.50E-01	0.742	0.747	-5.32E-03	Degrees of Freedom	7
1.00E-01	0.492	0.497	-5.04E-03		
7.50E-02	0.372	0.372	9.85E-05	X Coefficient(s)	0.199776
5.00E-02	0.250	0.247	3.24E-03	Std Err of Coef.	0.000356
3.00E-02	0.145	0.147	-1.65E-03	Conc.(mM) = 0.199776 * (Absorb.) + 0.000703	
7.03E-04		0.000			
0.00E+00	0.000				

Leach tests from tank 101-SY simulant; results

	Series A	Series B	Series C	Series D
Wt. sample	17.065	17.129	17.192	17.116
Total Cr (g)	6.997E-02	7.023E-02	7.049E-02	7.018E-02
Tot Cr(mol)	1.346E-03	1.351E-03	1.356E-03	1.350E-03
[Cr]diss.(M)	1.324E-01	1.322E-01	1.303E-01	1.261E-01
[Cr]cuvet(mM)	1.104E-03	1.101E-03	1.086E-03	1.051E-03
H2O2add(uL)	60	120	300	600

Leach solution measurements

	Time (h)	ABSORBANCE				CONCENTRATION (mM)			
		Series A	Series B	Series C	Series D	Series A	Series B	Series C	Series D
First Contact	0.5	0.207	0.230	0.359	0.503	4.21E-02	4.67E-02	7.24E-02	1.01E-01
	1	0.257	0.342	0.572	0.842	5.20E-02	6.90E-02	1.15E-01	1.69E-01
	3	0.423	0.578	1.088	1.474	8.52E-02	1.16E-01	2.18E-01	2.95E-01
	25.75	0.735	1.054	1.748	2.180	1.48E-01	2.11E-01	3.50E-01	4.36E-01
	139.25	0.845	1.204	1.786	2.245	1.70E-01	2.41E-01	3.58E-01	4.49E-01
	307.75	0.836	1.209	1.795	2.293	1.68E-01	2.42E-01	3.59E-01	4.59E-01
	361	0.863	1.229	1.862	2.366	1.73E-01	2.46E-01	3.73E-01	4.73E-01
2nd Contact	431	1.526	2.095	2.798	3.126	3.06E-01	4.19E-01	5.60E-01	6.25E-01
	70	0.663	0.866	0.936	0.760	1.33E-01	1.74E-01	1.88E-01	1.53E-01

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APPENDIX C

**RESULTS OF ESW AND C/D TESTS OF
WASTES FROM TANK 101-SY**

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ESW of 101-SY
ESW/PER of 101-SY

From WHC-N-656 1; pp. 119 and 123

Sample	Time	WtCrucB4	WtCrucSyn	WtW/NO3	Wt128C	Wt850C	WtTube	WtTubeLJ	WtCrucAft	CrucWtL	CrAlLoss		
101-SY ESW	DE101SYW/J2456 Wash	13.995	19.078				13.638	61.278					
	DE101SYL/J2458 Leach						14.028	42.448					
	DE101SYR/J2457 Rinse						14.061	48.821					
101-SY ESW/PE	1SYPERW/J2637 Wash	14.195	19.000				14.039	57.580					
	1SYPERL1/J2641 Leach 1						14.372	32.150					
	1SYPERL2/J2645 Leach 2						13.999	30.234					
	1SYPERR/J2647 Rinse						13.707	42.886					
Sample	Time	NetWt	FrLoss128	FrLoss850	WtSolids	WtSoln	WtDryTub	WtDrySol	WtAl(OH)	Soln.Vol	Soln.Dens	Wt%Solid	mLsol'n/ g sample
101-SY ESW	DE101SYW/J2456 Wash	5.083				47.640	14.062	0.057		45.0	1.059	1.121	5.153
	DE101SYL/J2458 Leach	5.083				28.420				26.0	1.093		2.978
	DE101SYR/J2457 Rinse	5.083				34.740				35.0	0.993		4.008
101-SY ESW/PE	1SYPERW/J2637 Wash	4.805				43.541	15.336	1.141		45.0	0.968	23.746	5.323
	1SYPERL1/J2641 Leach 1	4.805				17.778				15.0	1.185		1.774
	1SYPERL2/J2645 Leach 2	4.805				16.235				15.0	1.082		1.774
	1SYPERR/J2647 Rinse	4.805				29.179				30.0	0.973		3.549
Sample	Time	NO2 (ug/mL)	NO3 (ug/mL)	OH (ug/mL)	F (ug/mL)	Cl (ug/mL)	PO4 as P (ug/mL)	SO4 as S (ug/mL)	TOC (ug/mL)	TIC (ug/mL)			
101-SY ESW	DE101SYW/J2456 Wash1	1.14E+04	1.45E+04	2.39E+03		8.27E+02	2.40E+02	1.23E+02	1.25E+03	6.39E+02			
	DE101SYL/J2458 Wash2	1.15E+04	1.41E+04	5.26E+03		8.60E+02	2.35E+02	1.22E+02	1.24E+03	6.55E+02			
	Avg. 1&2	1.15E+04	1.43E+04	3.83E+03		8.44E+02	2.38E+02	1.23E+02	1.25E+03	6.47E+02			
	101SYLCH/J2488 Leach			5.31E+04					6.44E+01				
	DE101SYR/J2457 Rinse		5.07E+00	6.94E+03					2.97E+01	3.95E+01			
101-SY ESW/PE	1SYPERW/J2637 Wash	1.97E+04	1.81E+04	2.13E+03		9.95E+02	3.36E+02	1.66E+02	1.68E+03	1.10E+03			
	1SYPERL1/J2641 Leach 1			4.50E+04					8.25E+01	9.05E+01			
	1SYPERL2/J2645 Leach 2			4.85E+04					6.00E+01	1.26E+02			
	1SYPERR/J2647 Rinse			7.53E+02					0.00E+00	5.90E+01			
Sample	Time	NO2 (Wt%)	NO3 (Wt%)	OH (Wt%)	F (Wt%)	Cl (Wt%)	P (Wt%)	S (Wt%)	TOC (Wt%)	TIC (Wt%)	H2O (Wt%)		
101-SY	WINDOW E REPORT	1.05E+01	1.17E+01	2.47E+00	3.00E-02	7.90E-01	2.09E-01	1.33E-01	1.53E+00	6.30E-01	35.5		
	Corrected for %H2O	1.24E+01	1.38E+01	2.92E+00	3.54E-02	9.33E-01	2.47E-01	1.57E-01	1.81E+00	7.44E-01	23.8		
Enh.SI.Washes		5.67E-01	0.00E+00	4.21E+01	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00		
101-SY/ESW	5.083gS&3.6491gNaOH/NO2	7.46E+00	8.04E+00	1.93E+01	2.06E-02	5.43E-01	1.44E-01	9.17E-02	1.05E+00	4.33E-01			
101-SY ESW	101SYW/R/J2456, Avg. 1&2	5.90E+00	7.37E+00	1.97E+00	0.00E+00	4.35E-01	1.22E-01	6.31E-02	6.42E-01	3.33E-01			
	101SYLCH/J2488 Leach	0.00E+00	0.00E+00	1.58E+01	0.00E+00	0.00E+00	0.00E+00	0.00E+00	1.92E-02	0.00E+00			
	DE101SYR/J2457 Rinse	0.00E+00	2.03E-03	2.78E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	1.19E-02	1.58E-02			
	Sum Enh.SI.Wash	5.90E+00	7.37E+00	2.06E+01	0.00E+00	4.35E-01	1.22E-01	6.31E-02	6.73E-01	3.49E-01			
	Fract.Diss. - ESW	7.91E-01	9.16E-01	1.07E+00	0.00E+00	8.00E-01	8.53E-01	6.89E-01	6.39E-01	8.06E-01			
101-SY/ESWPER	4.805gS&3.6491gNaOH/NO2	7.06E+00	7.60E+00	1.92E+01	1.95E-02	5.13E-01	1.36E-01	8.67E-02	9.94E-01	4.09E-01			
101-SY ESW/PE	1SYPERW/J2637 Wash	1.05E+01	9.63E+00	1.13E+00	0.00E+00	5.30E-01	1.79E-01	8.85E-02	9.94E-01	5.86E-01			
	1SYPERL1/J2641 Leach 1	0.00E+00	0.00E+00	7.98E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	1.46E-02	1.61E-02			
	1SYPERL2/J2645 Leach 2	0.00E+00	0.00E+00	8.61E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	1.06E-02	2.24E-02			
	1SYPERR/J2647 Rinse	0.00E+00	0.00E+00	2.67E-01	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	2.09E-02			
	Sum ESWPER	1.05E+01	9.63E+00	1.80E+01	0.00E+00	5.30E-01	1.79E-01	8.85E-02	9.20E-01	6.45E-01			
	Fract.Diss.-ESWPER	1.41E+00	1.20E+00	8.32E-01	0.00E+00	9.75E-01	1.25E+00	9.66E-01	8.74E-01	1.49E+00			

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Sample	Time	less than												
		Na (ug/mL)	Al (ug/mL)	Si (ug/mL)	Fe (ug/mL)	Cr (ug/mL)	Ca (ug/mL)	P (ug/mL)	S (ug/mL)	U (ug/mL)	K (ug/mL)	Cr(VI) (ug/mL)		
101-SY ESW	DE101SYW/J2456	Wash1	2.04E+04	2.94E+03	2.64E+01	2.00E-01	4.39E+00	1.40E+01	2.58E+02	1.51E+02	1.47E+00	2.89E+02	1.28E+01	6.00E-01
	DE101SYL/J2458	Wash2	2.02E+04	2.92E+03	2.64E+01	2.00E-01	4.34E+00	1.44E+01	2.58E+02	1.51E+02	1.36E+00	2.90E+02	1.32E+01	5.00E-01
		Avg.1&2	2.03E+04	2.93E+03	2.64E+01	2.00E-01	4.37E+00	1.42E+01	2.58E+02	1.51E+02	1.42E+00	2.90E+02	1.30E+01	5.00E-01
	101SYLCH/J2488	Leach	6.94E+04	2.02E+02	1.07E+02	2.00E-01	4.97E+01	2.00E-01			1.61E+00		5.07E+01	
	DE101SYR/J2457	Rinse	9.05E+03	9.24E+00	2.79E+00	2.00E-01	8.63E+00	2.00E-01		1.20E+00	1.66E+00		1.53E+01	
101-SY/ESWPER	1SYPERW/J2637	Wash	2.83E+04	3.96E+03	3.54E+01	2.00E-01	5.66E+00	2.00E+01	3.51E+02	2.06E+02	9.01E-01	3.79E+02		
	1SYPERL1/J2641	Leach 1	5.91E+04	3.18E+02	8.58E+01	2.00E-01	2.21E+02				1.17E+01			
	1SYPERL2/J2645	Leach 2	6.15E+04	6.35E+01		2.00E-01	1.78E+02				2.06E+00			
	1SYPERR/J2647	Rinse	1.01E+03	8.10E+00	7.59E-01	3.60E+00	4.59E+01				6.09E-02			
Sample	Time	Na (Wt%)	Al (Wt%)	Si (Wt%)	Fe (Wt%)	Cr (Wt%)	Ca (Wt%)	P (Wt%)	S (Wt%)	U (Wt%)	K (Wt%)	Cr(VI) (Wt%)		
101-SY	WINDOW E REPORT	2.07E+01	3.20E+00	4.58E-02	2.80E-02	4.10E-01	2.30E-02	2.09E-01	1.33E-01	6.30E-03	3.26E-01	2.00E-03		
	Corrected for %H2O	2.44E+01	3.78E+00	4.58E-02	3.31E-02	4.84E-01	2.72E-02	2.47E-01	1.57E-01	7.44E-03	3.85E-01	2.36E-03		
Enh.SI.Washes		5.73E+01	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00		
101-SY/ESW	5.083gSY&3.6491gNaOH/NO	3.82E+01	2.20E+00	2.67E-02	1.93E-02	2.82E-01	1.58E-02	1.44E-01	9.17E-02	4.33E-03	2.24E-01	1.38E-03		
101-SY ESW	101SYW,R/J2456, Avg.1&2	1.05E+01	1.51E+00	1.36E-02	1.03E-04	2.25E-03	7.32E-03	1.33E-01	7.78E-02	7.29E-04	1.49E-01	6.70E-03		
	101SYLCH/J2488	Leach	2.07E+01	6.01E-02	3.19E-02	5.96E-05	1.48E-02	5.96E-05	0.00E+00	0.00E+00	4.79E-04	0.00E+00		
	DE101SYR/J2457	Rinse	3.63E+00	3.70E-03	1.12E-03	8.02E-05	3.46E-03	8.02E-05	0.00E+00	4.81E-04	6.65E-04	0.00E+00		
	Sum Enh.SI.Wash	3.48E+01	1.57E+00	4.66E-02	2.43E-04	2.05E-02	7.46E-03	1.33E-01	7.83E-02	1.87E-03	1.49E-01	2.79E-02		
	% Total Diss. ESW	3.81E+01	2.17E+00	2.49E-02	2.02E-04	1.46E-02	8.31E-03	1.44E-01	9.17E-02	3.16E-03	2.24E-01	1.38E-03		
101-SY/ESWPER	4.805gS&3.6491gNaOH/NO2	3.74E+01	2.08E+00	2.52E-02	1.82E-02	2.66E-01	1.49E-02	1.36E-01	8.67E-02	4.09E-03	2.12E-01	1.30E-03		
	1SYPERW/J2637	Wash	1.51E+01	2.11E+00	1.88E-02	1.06E-04	3.01E-03	1.06E-02	1.87E-01	1.10E-01	4.80E-04	2.02E-01		
	1SYPERL1/J2641	Leach 1	1.05E+01	5.64E-02	1.52E-02	3.55E-05	3.92E-02	0.00E+00	0.00E+00	0.00E+00	2.08E-03	0.00E+00		
	1SYPERL2/J2645	Leach 2	1.09E+01	1.13E-02	0.00E+00	3.55E-05	3.16E-02	0.00E+00	0.00E+00	0.00E+00	3.66E-04	0.00E+00		
	1SYPERR/J2647	Rinse	3.58E-01	2.87E-03	2.69E-04	1.28E-03	1.63E-02	0.00E+00	0.00E+00	0.00E+00	2.16E-05	0.00E+00		
	Sum ESWPER	3.68E+01	2.18E+00	3.43E-02	1.45E-03	9.01E-02	1.06E-02	1.87E-01	1.10E-01	2.94E-03	2.02E-01	0.00E+00		
	% Total Diss. ESWPER	3.71E+01	1.93E+00	6.81E-03	2.87E-04	2.19E-02	8.28E-04	1.36E-01	8.66E-02	3.87E-03	2.12E-01	ERR		
101-SY C/D	% Total Diss. C/D	2.07E+01	3.19E+00	4.52E-02	1.31E-03	4.10E-01	4.12E-03	2.06E-01	1.33E-01	5.67E-03	3.26E-01	ERR		
	Fract.Diss. - ESW	9.10E-01	7.15E-01	1.75E+00	1.26E-02	7.27E-02	4.72E-01	9.26E-01	8.54E-01	4.33E-01	6.66E-01	2.03E+01		
	Fract.Diss - ESWPER	9.85E-01	1.05E+00	1.36E+00	7.99E-02	3.38E-01	7.12E-01	1.38E+00	1.27E+00	7.19E-01	9.52E-01	0.00E+00		
		Na	Al	Si	Fe	Cr	Ca	P	S	U				
Percent Removed	Wash	30.0	94.8	27.3	0.4	0.6	51.5	100.0	99.4	28.4				
101-SY ESW	Leach	59.3	3.8	63.8	0.3	3.7	0.4	0.0	0.0	18.7				
	Rinse	10.4	0.2	2.2	0.3	0.9	0.6	0.0	0.6	25.9				
Percent Removed	Wash	40.6	90.0	15.0	0.1	0.3	5.5	100.0	100.0	15.4				
101-SY ESWPER	Leach 1	28.3	2.4	12.2	0.0	3.6	0.0	0.0	0.0	66.6				
	Leach 2	29.4	0.5	0.0	0.0	2.9	0.0	0.0	0.0	11.7				
	Rinse	1.0	0.1	0.2	1.4	1.5	0.0	0.0	0.0	0.7				
Residues	Sample	Time	Na (ug/g)	Al (ug/g)	Si (ug/g)	Fe (ug/g)	Cr (ug/g)	Ca (ug/g)	P (ug/g)	S (ug/g)	U (ug/g)	K (ug/g)	Cr(VI) (ug/g)	
101-SY ESW	DE101SYS/J2461	0	6.33E+04	1.65E+04	2.98E+03	2.04E+04	3.36E+05	6.01E+03	5.00E-01	5.00E-01	6.17E+02			
101-SY ESWPER	1SYPER/J2651		1.15E+04	6.88E+03	3.83E+03	3.83E+03	4.24E+04	7.65E+03	5.00E-01	5.00E-01	7.32E+00			
WtFraction		0	Na	Al	Si	Fe	Cr	Ca	P	S	U	K	Cr(VI)	
101-SY ESW	Solution		3.48E-01	1.57E-02	4.66E-04	2.43E-06	2.05E-04	7.46E-05	1.33E-03	7.83E-04	1.87E-05	1.49E-03	2.79E-04	
	Residue		7.10E-04	1.85E-04	3.34E-05	2.29E-04	3.77E-03	6.74E-05	5.61E-09	5.61E-09	6.92E-06	0.00E+00	0.00E+00	
101-SY ESWPER	Solution		3.68E-01	2.18E-02	3.43E-04	1.45E-05	9.01E-04	1.06E-04	1.87E-03	1.10E-03	2.94E-05	2.02E-03	0.00E+00	
	Residue		2.73E-03	1.63E-03	9.09E-04	9.09E-04	1.01E-02	1.82E-03	1.19E-07	1.19E-07	1.74E-06	0.00E+00	0.00E+00	
101-SY C/D	Solution	20	1.98E-01	3.00E-02	3.93E-03	1.14E-05	3.89E-03	2.90E-05	2.76E-03	1.63E-03	7.24E-05	2.92E-03		
WHC-EP-0766	Residue		3.89E-05	5.19E-05	5.06E-05	2.33E-04	1.66E-06	1.33E-04	3.92E-05	5.80E-07	8.00E-06	0.00E+00	ERR	
			Na	Al	Si	Fe	Cr	Ca	P	S	U	K	Cr(VI)	
	Fract.Diss -ESW		9.98E-01	9.88E-01	9.33E-01	1.05E-02	5.16E-02	5.25E-01	1.00E+00	1.00E+00	7.30E-01	1.00E+00	1.00E+00	
	Fract.Diss -ESWPER		9.93E-01	9.30E-01	2.74E-01	1.57E-02	8.21E-02	5.54E-02	1.00E+00	1.00E+00	9.44E-01	1.00E+00	ERR	
	Fract.Diss -C/D		1.00E+00	9.98E-01	9.87E-01	4.66E-02	1.00E+00	1.79E-01	9.86E-01	1.00E+00	9.00E-01	1.00E+00	ERR	

less than				less than				less than				Less than
Sr	Mn	Ni	Sr-90	Tc-99	Cs-137	Pu-239/40	Am-241	Np-237				Less than
(ug/mL)	(ug/mL)	(ug/mL)	(uCi/mL)	(uCi/mL)	(uCi/mL)	(nCi/mL)	(nCi/mL)	(nCi/mL)				thans
00	2.00E-01	1.38E+01	4.55E-01	8.00E-03	3.01E+01	5.82E-03	3.26E-02	2.16E-02				
00	2.00E-01	1.30E+01	4.33E-01	8.40E-03	2.96E+01	4.08E-03	3.31E-02	2.52E-02				
00	2.00E-01	1.34E+01	4.44E-01	8.20E-03	3.00E+01	4.95E-03	3.29E-02	2.34E-02				
	2.00E-01	4.00E-01	2.24E-03	9.47E-04	2.70E-01	8.86E-02	2.63E-01	3.04E-02	Np			
	2.00E-01	4.00E-01	1.48E-02	3.30E-02	8.87E-03	4.72E-03	3.09E-02	2.02E-02				
	2.00E-01	1.87E+01	6.47E-01	1.53E-02	3.90E+01	5.00E-02	7.77E-01	2.06E-02	less than Pu,Am,Np			
	2.00E-01		8.77E-03	7.02E-04	6.55E-01	7.18E-03	5.02E-02	2.52E-02	less than Pu,Am			
	2.00E-01		9.41E-03	3.48E-04	2.77E-02	2.77E-01	9.97E-01	1.87E-02				
	9.57E-01	3.08E-01	1.76E-01	1.88E-05		1.31E-01	1.47E+00	2.90E-02	less than Np			
				less than								
Sr	Mn	Ni	Sr-90	Tc-99	Cs-137	Pu-239/40	Am-241	Np-237				Sample
(Wt%)	(Wt%)	(Wt%)	(uCi/g)	(uCi/g)	(uCi/g)	(nCi/g)	(nCi/g)	(nCi/g)				WINDOW E REPO
03	1.69E-04	6.82E-03	2.10E+01	2.10E-01	3.50E+02	6.70E+00	1.10E+02	2.80E-02	101-SY			Corrected for %H2
03	1.69E-04	6.82E-03	2.48E+01	2.38E-01	3.96E+02	7.59E+00	1.25E+02	3.17E-02				
00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	Enh SI.Washes			
03	9.84E-05	3.97E-03	1.44E+01	1.38E-01	2.31E+02	4.42E+00	7.25E+01	1.85E-02	101-SY/ESW			5.083gSY&3.6491
03	0.00E+00	1.03E-04	2.29E+00	4.23E-02	1.54E+02	2.55E-02	1.69E-01	1.21E-01	101-SY ESW			101SYW,RJ2456,
00	0.00E+00	5.96E-05	6.67E-03	2.82E-03	8.04E-01	2.64E-01	7.83E-01	9.05E-02				101SYLCH/J2488
00	0.00E+00	8.02E-05	5.93E-02	1.32E-01	3.56E-02	1.89E-02	1.24E-01	8.10E-02				DE101SYR/J2457
03	0.00E+00	2.43E-04	2.35E+00	1.77E-01	1.55E+02	3.08E-01	1.08E+00	2.92E-01				Sum Enh.SI.Wash
03	ERR	1.41E-04	2.08E+00	1.28E-01	2.31E+02	1.06E-01	8.03E-01	1.54E-03				% Total Diss. ESW
03	9.30E-05	3.75E-03	1.36E+01	1.31E-01	2.18E+02	4.17E+00	6.85E+01	1.74E-02				
00	0.00E+00	1.06E-04	3.44E+00	8.14E-02	2.08E+02	2.66E-01	4.14E+00	1.10E-01				
00	0.00E+00	3.55E-05	1.56E-02	1.25E-03	1.16E+00	1.27E-02	8.91E-02	4.47E-02				
00	0.00E+00	3.55E-05	1.67E-02	6.17E-04	4.91E-02	4.91E-01	1.77E+00	3.32E-02				
00	0.00E+00	3.40E-04	6.25E-01	6.67E-05	0.00E+00	4.65E-01	5.22E+00	1.03E-01				
00	0.00E+00	5.17E-04	4.10E+00	8.34E-02	2.09E+02	1.24E+00	1.12E+01	2.90E-01				
RR	ERR	8.04E-05	1.86E-04									
RR	2.35E-05	0.00E+00	ERR									
00	0.00E+00	6.12E-02	1.63E-01	1.28E+00	6.73E-01	6.98E-02	1.48E-02	1.58E+01	Fract.Diss. - ESW			
00	0.00E+00	1.38E-01	3.00E-01	6.37E-01	9.58E-01	2.96E-01	1.64E-01	1.66E+01				
				Sr-90	Tc-99	Cs-137	Pu-239/40	Am-241	Np-237			
Wash				14.0	22.1	99.4	0.2	0.2	3.4			
Leach				0.0	1.5	0.5	2.1	0.8	2.6			
Rinse				0.4	69.2	0.0	0.1	0.1	2.3			
less than				less than								
Sr	Mn	Ni	Sr-90	Tc-99	Cs-137	Pu-239/40	Am-241	Np-237	Residues			Sample
(ug/g)	(ug/g)	(ug/g)	(uCi/g)	(uCi/g)	(uCi/g)	(nCi/g)	(nCi/g)	(nCi/g)				
01	5.90E+03	1.24E+04	1.25E+03	1.22E+00	1.00E+01	1.12E+03	8.57E+03	2.86E+02				
	9.94E+02	2.18E+04	2.00E+02	2.49E-01	2.13E-01	1.20E+02	1.64E+03	3.70E+01				
				Sr-90	Tc-99	Cs-137	Pu-239/40	Am-241	Np-237	Wt Fraction		
05	0.00E+00	2.43E-06	2.35E+00	1.77E-01	1.55E+02	3.08E-01	1.08E+00	2.92E-01	Solution			
09	0.00E+00	6.62E-05	1.40E+01	1.37E-02	1.12E-01	1.26E+01	9.61E+01	3.21E+00	Residue			
00	0.00E+00	5.17E-06	4.10E+00	8.34E-02	2.09E+02	1.24E+00	1.12E+01	2.90E-01				
00	0.00E+00	2.36E-04	4.75E+01	5.91E-02	5.06E-02	2.85E+01	3.89E+02	0.00E+00				
	2.08E-07	0.00E+00	1.45E+00	3.65E-01	3.44E+02	8.03E+00	2.24E+00	4.41E-02	104-S C/D		Solution	
	1.29E-06	5.85E-05	0.00E+00	1.85E+01	2.22E-05	8.97E-01	2.33E+00	7.68E+01	2.77E-03		Residue	
				Sr-90	Tc-99	Cs-137	Pu-239/40	Am-241	Np-237	Fract.Diss.-ESW		
00	ERR	3.54E-02	1.44E-01	9.28E-01	9.99E-01	2.40E-02	1.11E-02	8.35E-02	Fract.Diss.-ESW			
01	ERR	2.14E-02	7.95E-02	5.85E-01	1.00E+00	4.15E-02	2.80E-02	1.00E+00				
RR	1.39E-01	0.00E+00	ERR	7.27E-02	1.00E+00	9.97E-01	7.75E-01	2.83E-02	9.41E-01		Fract.Diss -C/D	

APPENDIX D

**RESULTS OF ESW AND C/D TESTS
OF WASTES FROM TANK 102-SY**

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ESW of 102-SY
ESW/PER of 102-SY

From WHC-N-656 1; pp. 120 and 124

Sample	Time	WtCrucB4	WtCrucSyn	Wt.w/NO3	W128C	W1850C	WtTube	WtTubeLJ	WtCrucAft	CrucWIL	CrALoss		
102-SY ESW	102SYWWSH/J2484 Wash	14.057	19.347				14.383	61.861					
	102SYLCH/J2485 Leach						14.038	47.523					
	102SYRIN/J2486 Rinse						14.027	44.110					
102-SY ESWPER	2SYPERW/J2638 Wash	13.563	18.405				14.049	55.016					
	2SYPERL1/J2642 Leach 1						14.077	32.580					
	2SYPERL2/J2646 Leach 2						14.111	30.418					
	2SYPERR/J2648 Rinse						14.007	42.440					
Sample	Time	NetWt	FrLoss128	FrLoss650	WtSolids	WtSoln	WtDryTub	WtDrySol	WtAl(OH)	Soln.Vol.	Soln.Dens	Wt%Solid	mLsol'n/ g sample
102-SY ESW	102SYWWSH/J2484 Wash	5.290				47.478	14.463	0.406		47.5	1.000	7.675	5.313734
	102SYLCH/J2485 Leach	5.290				33.485				31.0	1.060		3.467911
	102SYRIN/J2486 Rinse	5.290				30.083				30.0	1.003		3.356043
102-SY ESWPER	2SYPERW/J2638 Wash	4.842				40.967	16.784	3.221		40.0	1.024	66.522	4.710815
	2SYPERL1/J2642 Leach 1	4.842				18.503				15.0	1.234		1.768556
	2SYPERL2/J2646 Leach 2	4.842				16.305				15.0	1.087		1.768556
	2SYPERR/J2648 Rinse	4.842				28.433				30.0	0.948		3.533111
Sample	Time	NO2 (ug/mL)	NO3 (ug/mL)	OH (ug/mL)	F (ug/mL)	Cl (ug/mL)	PO4 as P (ug/mL)	SO4 as S (ug/mL)	TOC (ug/mL)	TIC (ug/mL)			
102-SY ESW	102SYWWSH/J2484 Wash	2.30E+03	4.81E+03	6.58E+02	3.22E+02	1.45E+02	1.23E+03	9.53E+01	7.18E+02	2.73E+02			
	102SYLCH/J2485 Leach	0.00E+00	0.00E+00	4.51E+04		0.00E+00	5.09E+02	0.00E+00	1.32E+02	9.45E+01			
	102SYRIN/J2486 Rinse			3.56E+03			2.86E+01		4.07E+01	2.20E+01			
102-SY ESWPER	2SYPERW/J2638 Wash	6.67E+03	4.11E+03	2.20E+03	7.09E+02	1.09E+02	2.61E+03		5.21E+02	3.59E+02			
	2SYPERL1/J2642 Leach 1			4.04E+04					1.06E+02	9.30E+01			
	2SYPERL2/J2646 Leach 2			4.83E+04					1.06E+02	1.64E+02			
	2SYPERR/J2648 Rinse			2.03E+03					2.97E+01	7.80E+01			
Sample	Time	NO2 (Wt%)	NO3 (Wt%)	OH (Wt%)	F (Wt%)	Cl (Wt%)	P (Wt%)	S (Wt%)	TOC (Wt%)	TIC (Wt%)	H2O (Wt%)		
102-SY	WHC-EP-0632	2.44E+00	5.71E+00	9.60E-01	5.60E-02	1.80E-01	4.11E-01	1.39E+00	5.40E-01	3.92E-01	62.5		
Enh.SLWashes		5.67E-01	0.00E+00	4.21E+01	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00		
102-SY/ESW	5.290gS&3.6491gNaOH/NO2	1.68E+00	3.38E+00	1.78E+01	3.31E-02	1.07E-01	2.43E-01	8.23E-01	3.20E-01	2.32E-01			
102-SY/ESWPER	4.842gS&3.6491gNaOH/NO2	1.64E+00	3.26E+00	1.87E+01	3.19E-02	1.03E-01	2.34E-01	7.93E-01	3.08E-01	2.24E-01			
102-SY ESW	102SYWWSH/J2484 Wash	1.22E+00	2.56E+00	3.50E-01	1.71E-01	7.70E-02	6.54E-01	5.07E-02	3.82E-01	1.45E-01			
	102SYLCH/J2485 Leach	0.00E+00	0.00E+00	1.56E+01	0.00E+00	0.00E+00	1.77E-01	0.00E+00	4.58E-02	3.28E-02			
	102SYRIN/J2486 Rinse	0.00E+00	0.00E+00	1.19E+00	0.00E+00	0.00E+00	9.58E-03	0.00E+00	1.37E-02	7.38E-03			
	Sum Enh.SLWash	1.22E+00	2.56E+00	1.72E+01	1.71E-01	7.70E-02	8.40E-01	5.07E-02	4.41E-01	1.85E-01			
102-SY ESWPER	2SYPERW/J2638 Wash	3.14E+00	1.94E+00	1.04E+00	3.34E-01	5.13E-02	1.23E+00	0.00E+00	2.45E-01	1.69E-01			
	2SYPERL1/J2642 Leach 1	0.00E+00	0.00E+00	7.14E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	1.87E-02	1.64E-02			
	2SYPERL2/J2646 Leach 2	0.00E+00	0.00E+00	8.53E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	1.91E-02	2.90E-02			
	2SYPERR/J2648 Rinse	0.00E+00	0.00E+00	7.17E-01	0.00E+00	0.00E+00	0.00E+00	0.00E+00	1.05E-02	2.78E-02			
	Sum ESWPER	3.14E+00	1.94E+00	1.74E+01	3.34E-01	5.13E-02	1.23E+00	0.00E+00	2.94E-01	2.42E-01			
	Fract.Diss. - ESW	7.29E-01	7.56E-01	9.67E-01	5.16E+00	7.23E-01	3.45E+00	6.16E-02	1.38E+00	7.96E-01			
	Fract.Diss. - ESWPER	1.92E+00	5.95E-01	9.34E-01	1.05E+01	5.00E-01	5.24E+00	0.00E+00	9.54E-01	1.08E+00			
102-SY C/D	WHC-EP-0632	4.08E-01	4.08E-01	7.46E-01									

D-3

WHC-EP-0882

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Sample	Time	Na	Al	Si	less than	Cr	less than	less than	P	S	U	K	Cr(VI)	
					Fe		Mn	Ca						
		(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	
102-SY ESW	102SYWWSH/J2484	Wash	8.81E+03	2.32E+02	5.00E-01	2.00E-01	3.94E+02	2.00E-01	1.00E-01	1.18E+03	1.00E+02	7.08E-01	1.52E+02	3.85E+02
	102SYLCH/J2485	Leach	6.34E+04	3.85E+03	4.97E+01	2.00E-01	1.02E+02	2.00E-01	1.00E-01	5.73E+02	1.00E+00	1.49E+01	5.00E+00	1.02E+02
	102SYRIN/J2486	Rinse	5.28E+03	2.50E+02	1.14E+01	2.00E-01	5.56E+00	2.00E-01	1.00E-01	3.07E+01	1.00E+00	1.39E-01	5.00E+00	5.53E+00
102-SY ESWPER	2SYPERW/J2638	Wash	1.33E+04	2.50E+02		2.00E-01	4.88E+02	2.00E-01	1.00E-01	2.50E+03	9.74E+01	2.19E+00	1.37E+02	
	2SYPERL1/J2642	Leach 1	5.74E+04	6.45E+02	6.75E+01	2.00E-01	7.12E+01	2.00E-01		1.83E+03		1.61E+01		
	2SYPERL2/J2646	Leach 2	6.15E+04	8.52E+02	5.49E+01	2.00E-01	1.04E+02					1.87E+01		
	2SYPERR/J2648	Rinse	2.70E+03	5.17E+01	5.08E+00	1.23E+01	1.20E+01	2.60E+00	1.88E+00	1.22E+01		1.43E+00		
Sample	Time	Na	Al	Si	Fe	Cr	Mn	Ca	P	S	U	K	Cr(VI)	
		(Wt%)	(Wt%)	(Wt%)	(Wt%)	(Wt%)	(Wt%)	(Wt%)	(Wt%)	(Wt%)	(Wt%)	(Wt%)	(Wt%)	
102-SY	WHC-EP-0632	2.07E+01	3.20E+00	4.58E-02	2.80E-02	4.10E-01	6.82E-03	2.30E-02	2.09E-01	1.33E-01	6.30E-03	3.26E-01	2.00E-03	
Enh.Sl.Washes		5.73E+01	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	
102-SY/ESW	5.290gS&3.6491gNaOH/NO2	3.68E+01	1.89E+00	2.71E-02	1.66E-02	2.43E-01	4.04E-03	1.36E-02	1.24E-01	7.89E-02	3.73E-03	1.93E-01	1.18E-03	
102-SY/ESWPER	4.842gS&3.6491gNaOH/NO2	3.64E+01	1.82E+00	2.61E-02	1.60E-02	2.34E-01	3.89E-03	1.31E-02	1.18E-01	7.60E-02	3.59E-03	1.86E-01	1.14E-03	
102-SY ESW	102SYWWSH/J2484	Wash	4.68E+00	1.23E-01	2.06E-04	1.06E-04	2.09E-01	1.06E-04	5.31E-05	6.27E-01	5.31E-02	3.76E-04	8.08E-02	2.05E-01
	102SYLCH/J2485	Leach	2.20E+01	1.34E+00	1.72E-02	6.94E-05	3.54E-02	6.94E-05	3.47E-05	1.99E-01	3.47E-04	5.17E-03	1.73E-03	3.54E-02
	102SYRIN/J2486	Rinse	1.77E+00	8.39E-02	3.83E-03	6.71E-05	1.87E-03	6.71E-05	3.36E-05	1.03E-02	3.36E-04	4.66E-05	1.68E-03	1.88E-03
	Sum Enh.Sl.Wash		2.84E+01	1.54E+00	2.13E-02	2.43E-04	2.47E-01	2.43E-04	1.21E-04	8.36E-01	5.38E-02	5.59E-03	8.42E-02	2.42E-01
102-SY ESWPER	2SYPERW/J2638	Wash	6.27E+00	1.18E-01	0.00E+00	9.42E-05	2.30E-01	9.42E-05	4.71E-05	1.18E+00	4.59E-02	1.03E-03	6.45E-02	0.00E+00
	2SYPERL1/J2642	Leach 1	1.01E+01	1.14E-01	1.19E-02	3.53E-05	1.26E-02	3.53E-05	0.00E+00	3.23E-01	0.00E+00	2.84E-03	0.00E+00	0.00E+00
	2SYPERL2/J2646	Leach 2	1.09E+01	1.51E-01	9.70E-03	3.53E-05	1.84E-02	0.00E+00	0.00E+00	0.00E+00	0.00E+00	3.30E-03	0.00E+00	0.00E+00
	2SYPERR/J2648	Rinse	9.54E-01	1.83E-02	1.79E-03	4.35E-03	4.24E-03	9.50E-04	6.64E-04	4.31E-03	0.00E+00	5.05E-04	0.00E+00	0.00E+00
	Sum ESWPER		2.82E+01	4.00E-01	2.34E-02	4.51E-03	2.65E-01	1.08E-03	7.11E-04	1.51E+00	4.59E-02	7.68E-03	6.45E-02	0.00E+00
	% Total Diss. ESW		3.50E+01	1.35E+00	5.35E-03	3.85E-05	5.12E-02	3.18E-05	1.24E-05	1.16E-01	7.89E-02	1.40E-04	1.93E-01	1.18E-03
	% Total Diss. ESWPER		3.50E+01	1.20E-01	1.88E-03	4.91E-05	3.79E-02	9.95E-05	1.31E-02	1.19E-01	7.60E-02	1.25E-04	1.86E-01	ERR
102-SY C/D		% Total Diss. C/D	2.00E+01	3.03E+00	2.31E-02	1.09E-04	4.07E-01	2.89E-03	4.81E-04	1.90E-01	1.33E-01	6.57E-04	3.26E-01	2.00E-03
	Fract.Diss. - ESW		7.96E-01	8.14E-01	7.87E-01	1.47E-02	1.02E+00	6.01E-02	8.92E-03	6.76E+00	6.82E-01	1.50E+00	4.36E-01	2.04E+00
	Fract.Diss. - ESWPER		7.75E-01	2.19E-01	6.97E-01	2.82E-01	1.13E+00	2.78E-01	5.42E-02	1.26E+01	6.03E-01	2.14E+00	3.47E-01	0.00E+00
	Percent Removed	Wash	16.2	5.7	0.2	0.0	17.9	0.0	0.0	70.3	96.7	0.3		
	102-SY	Leach	75.9	61.7	16.0	0.0	3.0	0.0	0.0	22.3	0.6	3.5		
		Rinse	6.1	3.9	3.5	0.0	0.2	0.0	0.0	1.2	0.6	0.0		
		less than PER								less than				
Residues	Sample	Time	Na	Al	Si	Fe	Cr	Mn	Ca	P	S	U	K	Cr(VI)
			(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)
102-SY ESW	102SYSOL/J2481	0	6.64E+04	8.08E+04	1.13E+04	1.36E+05	1.20E+05	4.01E+04	1.73E+04	7.32E+03	5.00E-01	1.86E+04		
102-SY ESWPER	2SYPERW/J2652		1.67E+04	8.57E+04	4.54E+03	2.20E+04	2.06E+04	6.33E+03				3.20E+03		
102-SY ESW	Wt.Fraction	0	Na	Al	Si	Fe	Cr	Mn	Ca	P	S	U	K	Cr(VI)
	Solution		2.84E-01	1.54E-02	2.13E-04	2.43E-05	2.47E-03	2.43E-05	1.21E-05	8.36E-03	5.38E-04	5.59E-05	8.42E-04	2.42E-03
	Residue		5.10E-03	6.20E-03	8.67E-04	1.04E-02	9.21E-03	3.06E-03	1.33E-03	5.62E-04	3.64E-05	1.43E-03	0.00E+00	0.00E+00
102-SY ESWPER	Solution		2.82E-01	4.00E-03	2.34E-04	4.51E-05	2.65E-03	1.08E-05	7.11E-05	1.51E-02	4.59E-04	7.68E-05	6.45E-04	0.00E+00
	Residue		1.11E-02	5.70E-02	3.02E-03	1.46E-02	1.37E-02	4.21E-03	0.00E+00	0.00E+00	0.00E+00	2.13E-03	0.00E+00	0.00E+00
102-SY C/D	Solution	20	7.58E-02	2.81E-02	5.24E-04	3.81E-05	1.34E-02	1.20E-03	2.54E-05	1.08E-02	1.04E-03	1.35E-04	1.35E-04	1.45E-03
WHC-EP-0632	Residue		2.79E-03	1.57E-03	5.15E-04	9.72E-03	1.07E-04	1.63E-03	1.19E-03	1.04E-03	6.38E-07	1.16E-03	0.00E+00	0.00E+00
	Fract.Diss.-ESW		9.82E-01	7.13E-01	1.97E-01	2.33E-04	2.11E-01	7.88E-04	9.13E-04	9.37E-01	1.00E+00	3.77E-02	1.00E+00	1.00E+00
	Fract.Diss.-ESWPER		9.82E-01	6.56E-02	7.20E-02	3.07E-03	1.62E-01	2.56E-03	1.00E+00	1.00E+00	1.00E+00	3.48E-02	1.00E+00	ERR
	Fract.Diss.-C/D		9.84E-01	9.47E-01	5.04E-01	3.90E-03	9.92E-01	4.24E-01	2.09E-02	9.12E-01	9.99E-01	1.04E-01	1.00E+00	1.00E+00

Pb	Sr	Ni	less than			Less than	Sample		
(ug/mL)	(ug/mL)	(ug/mL)	Sr-90 (uCi/mL)	Tc-99 (uCi/mL)	Cs-137 (uCi/mL)	Pu-239/40 (nCi/mL)	Am-241 (nCi/mL)	Np-237 (nCi/mL)	
0E+00	2.00E-01	2.02E+00	3.63E-02	6.73E-03	4.41E+00	2.78E-02	2.63E-01	2.44E-02	102SYWSH/J2484
0E+00	2.00E-01	5.00E-01	3.54E-03	1.05E-03	4.48E-01	1.93E-01	2.68E-02	3.57E-02	102SYLCH/J2485
0E+00	2.00E-01	5.00E-01	4.34E-04	2.27E-04	1.84E-02	8.31E-02	7.73E-02	3.87E-02	102SYRIN/J2486

1.53E+00

Sr-90	Tc-99	Cs-137	Pu-239/40	Am-241	Np-237	Less than
2.39E-02	6.11E-03	7.27E+00	6.17E-01	1.97E+00	2.44E-02	Np
7.22E-03	2.49E-04	6.41E-01	1.16E+00	2.32E-01	1.95E-02	
8.34E-03	1.35E-04	3.48E-01	2.45E+00	4.51E-01	3.21E-02	Np
6.54E-02	5.53E-04	2.35E-02	4.65E+00		2.45E-02	Np

Pb (Wt%)	Sr (Wt%)	Ni (Wt%)	Sr-90 (uCi/g)	Tc-99 (uCi/g)	Cs-137 (uCi/g)	Pu-239/40 (nCi/g)	Am-241 (nCi/g)	Np-237 (nCi/g)	Sample
48E-03	1.69E-04	1.50E-02	2.10E+01	2.10E-01	3.50E+02	6.70E+00	1.10E+02	2.80E-02	102-SY WHC-EP-0632
00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	Enh.SLWashes
65E-03	1.00E-04	8.88E-03	1.24E+01	1.24E-01	2.07E+02	3.96E+00	6.51E+01	1.66E-02	102-SY/ESW 5.290gS&3.6491g
55E-03	9.64E-06	8.56E-03	1.20E+01	1.20E-01	2.00E+02	3.82E+00	6.27E+01	1.60E-02	

Pb	Sr	Ni	Sr-90	Tc-99	Cs-137	Pu-239/40	Am-241	Np-237	Sample
31E-04	1.06E-04	1.07E-03	1.93E-01	3.68E-02	2.34E+01	1.48E-01	1.40E+00	1.30E-01	102-SY ESW 102SYWSH/J2484
47E-04	6.94E-05	1.73E-04	1.23E-02	3.64E-03	1.55E+00	6.69E-01	0.29E-02	1.24E-01	102SYLCH/J2485
36E-04	6.71E-06	1.68E-04	1.48E-03	7.62E-04	6.18E-02	2.79E-01	2.59E-01	1.30E-01	102SYRIN/J2486
21E-03	2.43E-04	1.41E-03	2.07E-01	4.02E-02	2.50E+01	1.10E+00	1.75E+00	3.83E-01	Sum Enh.SLWash % Total Diss. ESW

Pb	Sr	Ni	Sr-90	Tc-99	Cs-137	Pu-239/40	Am-241	Np-237
00E+00	0.00E+00	7.21E-04	1.13E-01	2.88E-02	3.42E+01	2.91E+00	9.28E+00	1.15E-01
00E+00	0.00E+00	0.00E+00	1.28E-02	4.40E-04	1.13E+00	2.05E+00	4.10E-01	3.44E-02
00E+00	0.00E+00	0.00E+00	1.47E-02	2.38E-04	6.15E-01	4.33E+00	7.97E-01	5.67E-02
00E+00	0.00E+00	0.00E+00	2.31E-01	1.95E-03	8.30E-02	1.64E+01	0.00E+00	8.66E-02
00E+00	0.00E+00	7.21E-04	3.71E-01	3.14E-02	3.61E+01	2.57E+01	1.05E+01	2.93E-01

Pb	Sr	Ni	Sr-90	Tc-99	Cs-137	Pu-239/40	Am-241	Np-237
70E-06	5.55E-08	1.17E-04	3.71E-02	3.52E-04	1.97E+02	1.11E-03	8.63E-03	3.41E-03
ERR	ERR	8.55E-03	3.66E-02	3.56E-03	1.93E+02	1.83E-02	2.78E-02	1.12E-04
58E-04	0.00E+00	0.00E+00	1.53E+00	2.10E-01	3.49E+02	5.19E+00	3.12E+00	2.63E-02

Pb	Sr	Ni	Sr-90	Tc-99	Cs-137	Pu-239/40	Am-241	Np-237	Fract.Diss. - ESW
58E-01	2.43E+00	1.59E-01	1.66E-02	3.23E-01	1.21E-01	2.76E-01	2.69E-02	2.31E+01	
00E+00	0.00E+00	8.43E-02	3.10E-02	2.62E-01	1.81E-01	6.73E+00	1.67E-01	1.83E+01	

Pb	Sr	Ni	Sr-90	Tc-99	Cs-137	Pu-239/40	Am-241	Np-237	Residues	Sample
77E+03	5.38E+02	1.38E+04	8.99E+02	1.84E+02	1.62E+01	5.12E+04	1.72E+05	1.93E+01	102-SY ESW	102SYSOL/J2481
			1.82E+02	1.54E+00	1.79E+00	8.03E+03	3.55E+04	6.22E+01	102-SY ESWPER	

Pb	Sr	Ni	Sr-90	Tc-99	Cs-137	Pu-239/40	Am-241	Np-237	Wt.Fraction
21E-05	2.43E-06	1.41E-05	2.07E-01	4.02E-02	2.50E+01	1.10E+00	1.75E+00	3.83E-01	102-SY ESW Solution
73E-04	4.13E-05	1.06E-03	6.90E+01	1.41E+01	1.24E+00	3.93E+03	1.32E+04	1.48E+00	Residue
00E+00	0.00E+00	7.21E-06	3.71E-01	3.14E-02	3.61E+01	2.57E+01	1.05E+01	2.93E-01	102-SY ESWPER
00E+00	0.00E+00	0.00E+00	1.21E+02	1.02E+00	1.19E+00	5.34E+03	2.36E+04	4.14E+01	
78E-05	0.00E+00	0.00E+00	1.45E+00	3.65E-01	3.44E+02	8.03E+00	2.24E+00	4.41E-02	102-SY C/D Solution
95E-04	3.65E-05	1.29E-03	1.65E+01	2.22E-05	8.97E-01	2.33E+00	7.68E+01	2.77E-03	WHC-EP-0632 Residue

Pb	Sr	Ni	Sr-90	Tc-99	Cs-137	Pu-239/40	Am-241	Np-237	Fract.Diss.-ESW
77E-02	5.55E-02	1.32E-02	2.99E-03	2.84E-03	9.53E-01	2.79E-04	1.33E-04	2.06E-01	
ERR	ERR	1.00E+00	3.06E-03	2.96E-02	9.68E-01	4.79E-03	4.44E-04	7.02E-03	
02E-01	0.00E+00	0.00E+00	7.27E-02	1.00E+00	9.97E-01	7.75E-01	2.83E-02	9.41E-01	Fract.Diss.-C/D

APPENDIX E

**RESULTS OF TESTS TO REMOVE PLUTONIUM
FROM C/D SOLUTIONS**

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Testing of Reductants for Plutonium and Manganese in C/D Products
 Notebook WHC-N-656 1; p. 79

Sample											insuf.sample			
	Al (ug/mL)	Cr (ug/mL)	Mn (ug/mL)	Na (ug/mL)	P (ug/mL)	S (ug/mL)	U (ug/mL)	Si (ug/mL)	Sr-90 (uCl/mL)	Tc-99 (uCl/mL)	Cs-137 (uCl/mL)	Np-237 (nCl/mL)	Pu-239,40 (nCl/mL)	
J1858	1.07E+04	4.11E+03	5.92E+02	5.14E+04	2.64E+03	5.23E+02	2.04E+02	2.63E+02	1.58E-03	6.72E-02	1.28E+01		1.06E+03	
1858B/J2148	1.06E+04	4.50E+03	5.60E+02	5.13E+04	3.00E+03	5.87E+02	2.17E+02	1.13E+02	1.92E-04	8.47E-03	1.29E+01	2.11E-01	9.88E+02	
1858PER/J2149	1.03E+04	4.40E+03	1.62E+00	5.14E+04	2.98E+03	5.66E+02	2.14E+02	1.94E+03	7.21E-05	2.08E-02	1.26E+01	3.10E-02	1.20E+01	
1858HN/J2150	1.05E+04	4.45E+03	1.86E+00	5.14E+04	3.07E+03	5.79E+02	2.27E+02	1.47E+03	5.75E-05	4.50E-02	1.25E+01	9.44E-02	3.75E+00	
J2121	1.83E+04	8.64E+03	7.72E+02	4.36E+04	4.72E+03	6.93E+02	9.66E+01	3.64E+02	2.41E-03	5.66E-02	3.03E+01	2.81E-02	6.90E+01	
2121B/J2151	8.66E+03	9.20E+03	7.61E+02	4.41E+04	5.01E+03	7.77E+02	7.97E+01	2.22E+02	8.72E-05	4.32E-02	3.26E+01	1.73E-01	5.15E+01	
2121PER/J2152	8.33E+03	8.59E+03	1.83E+00	4.32E+04	4.80E+03	7.26E+02	8.47E+01	1.68E+03	5.77E-05	7.84E-02	3.14E+01	3.19E-02	1.90E-01	
2121HN/J2154	8.22E+03	8.72E+03	1.51E+00	4.20E+04	4.82E+03	7.48E+02	7.37E+01	1.95E+02	2.07E-06		3.03E+01	3.13E-02	1.04E-01	
J2122	4.12E+03	2.06E+03	1.88E+02	1.66E+04	3.78E+03	1.35E+02	1.13E+01	5.50E+01	2.43E-03	1.33E-02	7.01E+00	7.93E-02	4.04E+01	
2122B/J2155	3.18E+03	1.78E+03	1.27E+02	1.33E+04	3.20E+03	1.16E+02	1.10E+01	3.95E+01	1.44E-04		7.48E+00	2.81E-02	4.42E+01	
2122PER/J2156	3.92E+03	2.17E+03	1.00E+00	1.67E+04	4.04E+03	1.45E+02	1.10E+01	4.09E+01	2.81E-06		7.18E+00	2.74E-02	1.21E-01	
2122HN/J2157	3.89E+03	2.10E+03	1.00E+00	1.66E+04	3.97E+03	1.43E+02	1.10E+01	3.87E+01	1.88E-05		7.33E+00	1.84E-02	5.02E-01	

PUPPTRES/CDelegard

C/D of Np and Pu Spiked Simulated HLW Composite; Solution Analyses Before and After Addition of Hydroxylamine
 Notebook WHC-N-656 1; p. 116

Sample #										nCl/mL	
	Al	Cr	Mn	Na	P	Pb	S	Si		Pu	Np
Initial DEHLW1/J2347	6.59E+03	2.23E+02	4.84E+01	7.50E+04	1.60E+03	1.85E+02	9.47E+02	1.59E+02		3.43E+02	2.92E+00
At NH2OH DEHLW02/J2348	6.47E+03	1.57E+02	2.00E+00	7.34E+04	1.53E+03	1.24E+02	9.35E+02	1.56E+02		9.60E+00	8.42E-01

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