Segregation of Uranium Metal from KBasin Sludge: Results from Vendor Testing

A. J. Schmidt
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September 2004

Prepared for
the U.S. Department of Energy
under Contract DE-AC06-76RL01830

Pacific Northwest National Laboratory
Richland, Washington 99352
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\(^{(a)}\) Fluor Corporation Mining & Minerals
Summary and Conclusions

Under contract to Fluor Hanford (FH), Pacific Northwest National Laboratory (PNNL) directed laboratory, bench-scale, and pilot-scale vendor testing to evaluate the use of commercial gravity mineral concentration technology to remove and concentrate uranium metal from Hanford K Basin sludge. Uranium metal in the sludge corrodes by reacting with water to generate heat and hydrogen gas, and may constrain shipment and disposal of the sludge to the Waste Isolation Pilot Plant (WIPP) as remote-handled transuranic (RH-TRU) waste.

To meet the hydrogen gas concentration criterion specified for shipment to WIPP, the uranium metal in the sludge must be removed, oxidized, or otherwise rendered unreactive. These other options may include removing the water from the sludge or preventing the uranium metal from coming in contact with the water.

Separating dense uranium metal from the K Basin sludge is expected to be similar to some physical gold recovery operations. Consequently, the capabilities of commercial gravity mineral concentration technologies were assessed for their applicability to K Basin sludge streams. The testing discussed here was conducted (in air) separately by two vendors (Gekko Systems, Ballarat, Victoria, Australia, and Knelson Concentrators, Vancouver, BC, Canada) using a sludge simulant developed by PNNL in collaboration with FH Sludge Retrieval and Disposal staff and supplied by PNNL especially for uranium metal segregation testing. While not typically deployed in radiological or underwater environments, the equipment evaluated is designed for rugged, low-maintenance applications, and underwater deployment appears technically feasible. The test results and key conclusions from this study are summarized below.

Key Conclusions

Overall, the vendor testing demonstrated the technical feasibility of using commercial gravity concentration equipment to separate the K Basin sludge into a high-volume uranium metal-depleted stream and a low-volume uranium metal-rich stream. In test systems, more than 96% of the uranium metal surrogate was concentrated into 10 to 30% of the sludge mass (7 to 24% of the sludge volume). With more prototypical equipment and stream recycle, higher recoveries may be achieved.

For the K East (KE) Basin sludge (containing KE floor, pit, and canister sludge), the test results indicate that gravity concentration in conjunction with packaging in a tailored matrix can generate a uranium metal-depleted stream for which waste loading (for disposal to WIPP) will not be limited by the rate of hydrogen generation from the uranium metal-water reaction. Once the uranium metal is segregated, waste loading then will be limited by physical volume or fissile gram equivalent (FGE) constraints.

For the K West (KW) canister sludge stream (a major component of the settler tank sludge), it appears gravity concentration will yield a depleted stream for which the remaining uranium metal will be slightly more constraining than the FGE limit for disposal of the sludge to WIPP.

Without extensive stream recycle, the gravity concentration equipment tested will not produce a high-purity uranium metal concentrate stream (e.g., >98%). If it desirable to process the separated uranium metal using the same process as used for fuel elements (e.g., cold vacuum drying), then further upgrading of the concentrate stream (i.e., further removal of non-uranium metal components) will be necessary. Alternatively, for WIPP disposal, the concentrate stream could be processed to oxidize the uranium metal.
Gravity concentration processing of Knockout Pot (KOP) sludge is not recommended, since the KOP sludge is expected to have a high uranium metal content (i.e., greater than 20 wt%) without additional processing. It is unlikely that the equipment evaluated in this study could produce a high-purity uranium metal stream (e.g., >98%) from the KOP sludge.

K Basin Sludge Segregation Simulant

The sludge simulant developed for this testing is conservative with respect to actual sludge containing uranium metal, in that achieving a given separation factor for the simulant should be more difficult than for the actual sludge. Conservative features of the simulated sludge include:

- Use of cobalt-cemented tungsten carbide (referred to as “W/Co”) fragments (250 to 1500 µm, with average size of 580 µm) as the surrogate for uranium metal fragments. The lower particle density of the W/Co (14.5-15.0 g/cm³) compared to uranium metal (19 g/cm³) increases the recovery challenge.

- Use of spherical-shaped stainless steel powder (less than 120 µm, average ~60 µm) with a particle density of ~7.8 g/cm³, as the surrogate for uranium oxyhydrate species. The average particle size and particle density of the stainless steel powder appear to be greater than those of uranium oxyhydrate species, increasing the challenge to separate it from the W/Co in the simulant.

- Use of various grades of Kleen Blast (a sand blasting product, particle density ~2.8 g/cm³) as a surrogate for larger-diameter non-uranium sludge particles to create a simulant that contains a higher fraction of greater-than-500-µm particles than found in KE floor and canister sludge.

Batches of this simulant were prepared at PNNL and sent to the vendors for the separations testing using their process equipment.

Gekko Test Results

Gekko Systems performed two types of testing: 1) shaker table testing (tabling), in which slurry is fed to the upper edge of a sloping table (riffle-decked in a horizontal plane) to evaluate metal segregation and recovery, and 2) spinner testing, in which a centrifugal concentrator is used to evaluate upgrading the concentrate stream from the tabling by further removal of the non-uranium metal components.

Shaker Table Testing

Three shaker table tests were conducted at Gekko with ~20 kg (dry basis) of simulant in each test. In the tabling tests, simulant slurry feed was segregated into a concentrate fraction or fractions containing the W/Co-enriched stream and a tails fraction containing a W/Co-depleted stream. The testing was designed to evaluate the overall applicability of Gekko’s gravity concentration equipment for processing the simulant. Gekko uses the results from shaker table testing to predict the performance of their full-scale InLine Pressure Jig, a mineral concentrating device applied to recover various minerals from alluvial and crushed hard rock ores. The test results (Table S.1) show that the shaker table recovered about 97 to 99% of the tungsten into about 30 to 33% of the sample mass (21 to 24 vol%). At the same time, only about 20 to 25% of the stainless steel powder reported to the concentrate, indicating good separation of the W/Co (uranium metal surrogate) from the stainless steel (uranium oxyhydrate surrogate).
Table S.1. Summary of Gekko Test Results

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Stream Split (a)</th>
<th>Percent Tungsten (W) Recovered to Concentrate (based on analyses)</th>
<th>Overall Analytical Mass Balance Check (c)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Concentrate</td>
<td>Tails</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Wt% of Feed [W]</td>
<td>Wt% of Feed [W]</td>
<td></td>
</tr>
<tr>
<td></td>
<td>[W] Wt% (e)</td>
<td>[W] Wt%</td>
<td>[W] Wt%</td>
</tr>
<tr>
<td></td>
<td>Overall</td>
<td>Plus 500 µm Minus 500 µm</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Shaker Table Tests 1, 2, and 3 conducted with simulant feed containing ~2.78 wt% tungsten</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Shaker Table 1</td>
<td>30.6</td>
<td>8.5</td>
<td>69.4</td>
</tr>
<tr>
<td>Shaker Table 2</td>
<td>31.5</td>
<td>9.3</td>
<td>68.5</td>
</tr>
<tr>
<td>Shaker Table 3</td>
<td>32.3</td>
<td>8.3</td>
<td>67.7</td>
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<tr>
<td>Spinner Upgrade Tests 1 and 2 conducted with concentrate from Shaker Table testing used as feed</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Spinner 1(d)</td>
<td>52.8</td>
<td>7.6</td>
<td>47.2</td>
</tr>
<tr>
<td>Spinner 2(e)</td>
<td>33.1</td>
<td>20.3</td>
<td>66.9</td>
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<td>Combined results from Shaker Table Tests and Spinner Upgrade Tests</td>
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<tr>
<td>Shaker Table 3 + Spinner 2 (actual results)</td>
<td>10.7</td>
<td>20.3</td>
<td>89.3</td>
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<tr>
<td>Shaker Table 3 + Spinner 2 (theoretical(f) results)</td>
<td>10.7</td>
<td>23.3(f)</td>
<td>89.3</td>
</tr>
<tr>
<td>Shaker Table 3 + Spinner 1 (theoretical(f) results)</td>
<td>17.1</td>
<td>16.0(f)</td>
<td>82.9</td>
</tr>
</tbody>
</table>

(a) Feed stream was segregated into tungsten-enriched stream (concentrate) and tungsten-depleted stream (tails). Stream split (mass percent) and tungsten concentration (by assay) in each stream are provided.
(b) Tungsten-containing particles in feed ranged from 250 to 1500 µm. Subsamples from testing were sieved at 500 µm and split into two fractions to examine tungsten recovery as a function of particle size. Overall recovery is combined recovery of plus 500-µm and minus 500-µm splits. Recoveries are based on tungsten measured (assayed) in concentrate and tails.
(c) Check on measured (assayed) tungsten in product streams vs. quantity used to prepare test material. Value = 100%× [mass of W in concentrate (by assay) + mass of W in tails (by assay)] divided by mass of W in feed (mass was measured).
(d) Concentrate from Shaker Table 2 used as feed to Spinner 1. Overall analytical mass balance check on tungsten for this test was only 43%, most likely due to an inhomogeneous splitting of Shaker Table 2 concentrate.
(e) Concentrate from Shaker Table 3 used as feed to Spinner 2.
(f) Tungsten concentrations in concentrate and tails were calculated based on 2.78 wt% tungsten in feed, mass splits as measured, recoveries as measured, and 100% mass balance.
Samples from the shaker table tests were passed through a 500-µm screen to produce plus (greater than) and minus (less than) 500-µm subsamples. Tungsten recovery results were similar for the two size fractions; 97.6 to 99.5% of the plus 500-µm and 95.2 to 97.7% of the minus 500-µm tungsten was recovered into the concentrate. The tungsten measured in all test samples by chemical assay versus the tungsten known to be added to the simulant during feed preparation compared favorably with mass balance closures ranging from 96 to 109%.

The tungsten concentration in the simulant feed was 2.78 wt% (dry basis) and ranged from 8.3 to 9.3 wt% in the concentrate stream (~30 wt% of the sample). In the tails stream (about 70 wt% of the test sample mass), the tungsten concentration ranged from 0.05 to 0.13 wt% (dry basis). If the uranium metal content in nominal KE canister sludge (2.95 wt% dry basis) was reduced to a similar level (e.g., 0.1 wt%) and the resulting sludge packaged in a tailored matrix, waste loading for WIPP disposal would no longer be constrained by hydrogen generation rate limits, but by FGE limits.

**Spinner Testing**

Two concentrate upgrading tests also were conducted at Gekko (Table S.1). In these tests, the primary concentrate generated from the shaker table testing was further concentrated using the InLine Spinner, a centrifugal concentrator. In the first spinner tests, 52.8 wt% of the spinner feed stream was collected as upgraded concentrate (i.e., a “mass pull” of 52.8%). This upgraded concentrate contained 99.5 wt% of the total recovered tungsten in the concentrate plus tails streams. However, the mass balance was low; the total tungsten in the two streams in this test only accounted for 43% of the tungsten expected (i.e., likely due to a problem in sub-sampling to provide the initial feed stream material). In the second spinner test, 33.1% of the spinner feed was collected as upgraded concentrate, with 89.7 wt% of the tungsten reporting to the concentrate stream. Reasonable closure of the mass balance (89%) was achieved in the second spinner test.

The overall effect of combining Shaker Table Test 3 results with Spinner Test 2 results is also shown in Table S.1. The last two rows in Table S.1 show theoretical combined results, using the measured tungsten recoveries and assuming 100% closure of the material balances. Figure S.1 illustrates the combined Shaker Table Test 3 and Spinner Test 1 results. For this combination (Figure S.1), 98.2% of the tungsten is recovered into 17.1% of the feed mass. The combined test results do not consider the potential performance improvements that could be achieved by recycling the InLine Spinner tails back as feed to the InLine Pressure Jig.

Based on the testing results with the Gekko gravity concentration equipment, a grinding or prescreening unit operation will not be needed to achieve a high uranium metal recovery and clean-up; i.e., the Gekko equipment can process feed that contains particles up to 25,000 µm.

**Knelson Test Results**

At Knelson Concentrators, laboratory centrifugal concentration testing was followed by pilot-scale testing with a continuous variable discharge concentrator. Results from the Knelson tests are reported in Table S.2. In the laboratory test, 4 kg (dry basis) of simulant was processed as a slurry in five passes through a small-scale centrifugal batch concentrator. The concentrate collected in the first four passes constituted about 20 wt% of the test sample mass (~13 vol% of the test sample). This concentrate contained about 96.5% of the tungsten originally present in the feed and only about 25% of the stainless steel was recovered.
steel powder. The tungsten concentration in the tails (depleted) stream, constituting about 80 wt% of the feed (or 87 vol%) of the test sample mass, was 0.16 wt%. A mass balance over-recovery for tungsten (124%) was determined for this test.

For the pilot-scale testing, PNNL provided 219 kg (dry basis) of K Basin segregation simulant. Due to test system limitations, such as the inability to suspend large particles in the feed tank, the piping layout for the pilot test system, and the valve sizes in the concentrator, the simulant first had to be screened by Knelson to remove particles greater than 1180 µm. This prescreening left 160.5 kg of the simulant for testing but only removed a very small fraction (less than 1%) of the tungsten. The screened simulant was processed through the concentrator in two passes, with the tails from the 1st pass used as feed for the 2nd pass. The combined concentrate from the 1st and 2nd passes contained 98.4 wt% of the recovered uranium metal surrogate (W/Co) in 10.5 vol% (18 wt%) of the screened feed material quantity. The tungsten measured in the concentrate and tails (by assay) accounted for 90% of the tungsten known to be in the feed. A flowsheet for the pilot testing is provided in Figure S.2.
### Table S.2. Summary of Knelson Centrifugal Concentrator Test Results

<table>
<thead>
<tr>
<th>Test</th>
<th>Stream Split&lt;sup&gt;(a)&lt;/sup&gt;</th>
<th>Percent Tungsten (W) Recovered to Concentrate (based on analyses)</th>
<th>Overall Analytical Mass Balance Check&lt;sup&gt;(b)&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Concentrate (enriched stream)</td>
<td>Tails (depleted stream)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Wt% of Feed</td>
<td>[W] Wt%</td>
<td>Wt% of Feed</td>
</tr>
<tr>
<td>Laboratory scale concentrator test conducted with simulant feed containing ~2.78 wt% tungsten</td>
<td>20.2&lt;sup&gt;(c)&lt;/sup&gt;</td>
<td>17.8</td>
<td>79.8</td>
</tr>
<tr>
<td>Pilot-scale concentrator test conducted with simulant feed containing ~3.79 wt% tungsten&lt;sup&gt;(d)&lt;/sup&gt;</td>
<td>17.9&lt;sup&gt;(c)&lt;/sup&gt;</td>
<td>18.7</td>
<td>82.1</td>
</tr>
</tbody>
</table>

(a) Feed stream was segregated into tungsten-enriched stream (concentrate) and tungsten-depleted stream (tails). Stream split (mass percent) and tungsten concentration (by assay) in each stream are provided. Check on measured (assayed) W in product streams vs. quantity used to prepare test material. For the pilot-scale test, values are based on the feed after sieving; see footnote (d).

(b) Value = 100% × [mass of W in concentrate (by assay) + mass of W in tails (by assay)] divided by mass of W in feed (mass was measured).

(c) Combined concentrate from four sequential passes through concentrator (e.g., tails from 1<sup>st</sup> pass used as feed for 2<sup>nd</sup> pass).

(d) Original feed exhibited a particle size distribution that ranged from submicron to ~4000 µm and contained ~2.78 wt% tungsten. To address particle size constraint associated with the pilot-scale test system, the feed was sieved to remove all particles greater than 1180 µm, resulting in a feed that contained ~3.79 wt% tungsten.

(e) Combined concentrate from two sequential passes through concentrator (e.g., tails from 1<sup>st</sup> pass used as feed for 2<sup>nd</sup> pass).

The technical specifications for the Knelson concentrator recommend that the feed stream contain no particles greater than 1/8 in. (3180 µm). Knelson technical staff stated that the concentrator should be able to handle particles up to 2500 µm. While it is possible the concentrator could be modified to accept larger-size feed material, it is most likely that a prescreening step followed by a grinding step would be necessary for processing the K Basin sludge, which can contain particles up to 6350 µm. For KE floor and KE canister sludge, only about 5 wt% of the sludge particles are greater than 2500 µm.
Mass 219 Kg  
W* 6.1 Kg  
W* 2.78 %  

W* = determine from feed make up  
W** = determined by chemical assay  

Mass 58.5 Kg  
W* 0.018 Kg  
W* 0.03 %  

Mass 160.5 Kg  
W* 6.08 Kg  
W* 3.79 %  

Mass 18.5 Kg  
W** 4.92 Kg  
W** 26.7 %  

Mass 142.0 Kg  
W** 1.16 Kg  
W** 0.8 %  

Mass 10.3 Kg  
W** 0.45 Kg  
W** 4.33 %  

Mass 131.7 Kg  
W** 0.09 Kg  
W** 0.07 %  

Mass 28.8 Kg  
W** 5.37 Kg  
W** 18.7 %  

Balance  
W  0.62 Kg  [W in 1st Pass Feed - (W in Concentrate + W in Tails)]  

Figure S.2. Flowsheet for Pilot-Scale Testing of Knelson Continuous Variable Discharge Concentrator
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Acknowledgments

The authors gratefully acknowledge and appreciate the support of the following individuals:

Jennifer Abols, Vice President, Canadian Operations, Gekko Systems, who coordinated the testing, data interpretation, and reporting for the tests conducted by Gekko Systems.

Iswinder Grewal, Manager, Knelson Research & Technology Centre, who coordinated the testing, data interpretation, and reporting for the tests conducted by Knelson Concentrators.

Todd Hart, Pacific Northwest National Laboratory, who performed the sample preparation and inductively coupled plasma analysis on samples from the Knelson testing. This analysis work was performed at the W. R. Wiley Environmental Molecular Sciences Laboratory, a national scientific user facility sponsored by the U.S. Department of Energy’s Office of Biological and Environmental Research and located at PNNL.

We also thank Sue Gano, Pacific Northwest National Laboratory, for her diligent editorial and document production support.
### Acronyms and Abbreviations

<table>
<thead>
<tr>
<th>Acronym</th>
<th>Description</th>
</tr>
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<tr>
<td>CVD</td>
<td>continuous variable discharge</td>
</tr>
<tr>
<td>FGE</td>
<td>fissile gram equivalent</td>
</tr>
<tr>
<td>FH</td>
<td>Fluor Hanford</td>
</tr>
<tr>
<td>ICP</td>
<td>inductively coupled plasma</td>
</tr>
<tr>
<td>ICP-OES</td>
<td>inductively coupled plasma-optical emission spectrometry</td>
</tr>
<tr>
<td>IPJ</td>
<td>InLine Pressure Jig</td>
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<tr>
<td>IPS</td>
<td>InLine Spinner</td>
</tr>
<tr>
<td>KE</td>
<td>K East</td>
</tr>
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<td>KOP</td>
<td>Knockout Pot</td>
</tr>
<tr>
<td>KW</td>
<td>K West</td>
</tr>
<tr>
<td>MCO</td>
<td>multi-canister overpack</td>
</tr>
<tr>
<td>MT</td>
<td>metric tons</td>
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<tr>
<td>NIST</td>
<td>National Institute of Standards and Technology</td>
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<td>NLOP</td>
<td>North Loadout Pit</td>
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<td>PNNL</td>
<td>Pacific Northwest National Laboratory</td>
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<tr>
<td>PoP</td>
<td>proof of principle</td>
</tr>
<tr>
<td>PSD</td>
<td>particle size distribution</td>
</tr>
<tr>
<td>RH</td>
<td>remote handled</td>
</tr>
<tr>
<td>SNF</td>
<td>spent nuclear fuel</td>
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<td>TRU</td>
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<tr>
<td>W</td>
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</table>
1.0 Introduction

The current plan for the majority of the Hanford K Basin sludge is disposition to the Waste Isolation Pilot Plant (WIPP) as remote-handled (RH) transuranic (TRU) waste. Uranium metal fuel particles in the sludge constitute a challenge to disposal operations, because metallic uranium can corrode in water to generate hydrogen gas. Because the hydrogen evolution rate is strictly limited within the RH-TRU 55-gal drums during shipment to WIPP, the sludge loading (i.e., volume of sludge per drum) must be limited so as to not exceed the mandated limit. This constraint in sludge loading thus drives the number of containers and shipments required for disposal to WIPP.

A technology assessment (Mellinger et al. 2004) concluded that to meet the WIPP hydrogen gas concentration criterion, a uranium metal removal operation and some reduction of the reaction rate by a solidification matrix likely will be required. However, testing was required to establish the technical feasibility of segregation processes and to determine the extent to which the uranium metal could be removed. Testing was also required to determine the effects of solidification matrices on mitigation of the uranium metal-water reaction rate.

Under contract to Fluor Hanford (FH), Pacific Northwest National Laboratory (PNNL) directed laboratory, bench-scale, and pilot-scale vendor testing to evaluate the use of commercial gravity mineral concentration technology to remove uranium metal from K Basin sludge streams and concentrate it to a smaller volume fraction. During the initial assessment of the technology, all of the vendors contacted indicated that bench-scale testing using shaker tables or other small-scale prototype equipment would be necessary to confirm the technical viability of this application and to provide estimates of the expected uranium metal removal efficiency.

From discussions with mineral processing experts and vendor technical staff, two internationally recognized vendors were selected to conduct tests using a K Basin sludge simulant provided by PNNL: Gekko Systems (Ballarat, Victoria, Australia) and Knelson Concentrators (Vancouver, BC, Canada). Preliminary results (laboratory and bench scale) were given in an interim report (Schmidt and Elmore 2004). Final results from this study, including pilot-scale testing, are presented here. Section 1.1 provides background information on the sludge streams and disposal management; Section 1.2 summarizes an initial assessment of the uranium removal requirement; and Section 1.3 gives an overview of a conceptual uranium metal segregation process. Section 2.0 describes the K Basin sludge segregation simulant developed by PNNL. Section 3.0 discusses the Gekko testing and results, and Section 4.0 discusses the Knelson testing and results. Section 5.0 outlines deployment considerations. The appendices contain reports provided by the vendors.

Separate testing was conducted (Delegard et al. 2004) to understand the effects of hydraulic grout solidification matrices on decreasing the rate of the uranium metal-water reaction to produce hydrogen. In this testing, the rate of hydrogen gas generation was measured for uranium metal particles of known surface area in water, in simulated K West (KW) Basin canister sludge, and in simulated KW canister sludge immobilized in grout. Four grouts based on Portland cement and two grouts based on magnesium phosphate cement were tested. Most of the cements were formulated to minimize the water component but provide workable mixes. Although none of the tested grouts radically decreased the uranium metal corrosion rate, the lowest overall hydrogen generation rate, about 22% (i.e., a 4-fold decrease) of the rate specified by the Spent Nuclear Fuel (SNF) Project Databook (Duncan2001), was observed for Portland
cement containing bentonite clay. Bentonite also was found to decrease the rate of hydrogen gas generation for the magnesium phosphate cement.

The combined results from the Delegard et al. (2004) tests and the testing discussed here are used in Section 1.2 to assess the uranium removal requirements for various sludge streams.

1.1 Background on Sludge Streams and Disposal Management

Two water-filled concrete pools, K East (KE) and K West (KW) Basins, in the 100K Area of the Hanford Site contained over 2100 metric tons of N Reactor fuel elements stored in aluminum or stainless steel canisters. The fuel currently is being cleaned and removed from the K Basins. During the time the fuel has been stored (mid 1970s to present), approximately 52 m³ of heterogeneous solid material (sludge) has accumulated in the canisters, as well as on the floor and in the associated pits, along with historic sludge materials resulting from earlier operation of the Hanford K East Reactor. The K Basin sludge consists of various proportions of metallic and oxidized fuel particles, canister corrosion products (i.e., various iron and aluminum hydrous oxides and hydroxides), sand filter backwash, windblown material, spalled concrete from uncoated basin walls, and miscellaneous constituents such as ion exchange material (both organic and inorganic) and paint chips (Makenas et al. 1996-99). The Spent Nuclear Fuel Project defines sludge as material that is less than 0.25 in. in any two dimensions.

The KE Basin floor and pit sludge (40.1 m³) contains a large quantity of fuel corrosion products as a result of the open tops, and in many cases open-screened bottoms, of the fuel storage canisters. In contrast, because the fuel stored in the KW Basin was first placed in closed and sealed canisters, most of the corrosion products were kept from the KW Basin floor and pits. Consequently, the volume of sludge buildup in the KW Basin is much smaller than in KE Basin. The small quantity of sludge that was present on the floor of the KW Basin before fuel cleaning and fuel transfer operations began was assumed to be primarily in-blown dust and sediment.

Since the start-up of fuel cleaning and transfer operations, some high-uranium canister-like sludge has been introduced to certain areas of the KW Basin floor. However, only one of the pits, North Loadout Pit (NLOP), in the KW Basin contains a significant amount of sludge (nominally 3.64 m³). This sludge is likely to consist of a mix of fuel corrosion products and sand generated from the backwash of the sand filters from the main basin water treatment system.

Under the current sludge management plan, the KE North Loadout Pit (KE NLOP) sludge (nominally 6.3 m³), which contains very little uranium metal, will be retrieved and transported to another facility, where it will be immobilized in grout and packaged in 55-gal drums for disposal to WIPP as contact-handled transuranic waste. The remaining KE floor and pit sludge (nominally 33.8 m³) and the KE canister sludge (nominally 2.5 m³) will be moved from the floor and pits and consolidated in large, free-standing containers located in the pits. The resulting containerized sludge thus will be isolated from the rest of the KE Basin. Subsequently, the KE containerized sludge will be transferred (via hose-in-hose transfer line) to similar free-standing containers in the KW Basin. The KW Basin containers also will be used to collect the sludge from the KW floor and pits.

Once the material from KE Basin is transferred to the KW Basin, there will be three sludge streams in the KW Basin:
• Knockout Pot sludge (nominally, 0.42 m$^3$) – The KOP sludge arises from fuel element cleaning and is expected to consist primarily of metallic uranium fuel pieces ranging in size from about 500 µm to less than 0.25 in. (6350 µm).

• Settler Tank sludge (nominally 2.8 m$^3$) – The settler tank sludge is material generated from fuel element cleaning that escapes the Knockout Pots, and passes through a 500-µm screen. Its composition is expected to be similar to that of KW canister sludge and contain high concentrations of uranium oxyhydrates, small fuel particles, iron and aluminum oxides, and lower concentrations of other materials.

• Containerized sludge (nominally 40.9 m$^3$) – The containerized sludge includes the containerized sludge from the KE Basin (36.3 m$^3$) plus smaller volumes of sludge from the KW NLOP (3.64 m$^3$) and the KW floor and pits (~1 m$^3$). The overall composition of the containerized sludge collected in the KW Basin thus will be similar to that of the KE floor and canister sludge.

The current plan for the containerized sludge and the settler tank sludge is disposition to WIPP as RH-TRU waste. The KOP sludge may be dispositioned as fuel, i.e., packaged and dried in multi-canister overpacks (MCOs) by cold vacuum drying and stored at the Canister Storage Building, or it may be treated and packaged as RH-TRU for disposition to WIPP. An engineering evaluation of these options, performed by BNFL for Fluor Hanford (Erpenbeck and Miska 2004), favored treatment and packaging in a grouted RH-TRU waste form for disposal of the KOP sludge to WIPP.

1.2 Uranium Removal Requirement

To prepare the sludge to meet WIPP waste acceptance criteria and to increase the quantity of sludge that can be loaded into each container, the reaction of uranium metal with the associated water must be diminished. Methods to achieve this goal include:

• removing the uranium metal
• oxidizing the uranium metal
• removing the water (and preventing its return into the vented storage drums)
• coating (e.g., microencapsulating) the uranium metal to prevent uranium metal-water contact.

This section provides an initial assessment of the impact of uranium metal removal (96.6% removal to 15% of the original sludge volume) combined with final packaging in a tailored matrix. The initial assessment (summarized in Table 1.1) includes estimates of the quantities of various K Basin sludge streams that may be loaded to a 55-gal drum for disposal to WIPP as RH-TRU. The drum loading limits are fixed, based on hydrogen generation rate and $^{239}$Pu fissile gram equivalent (FGE) constraints. The assessment helps show how much uranium metal must be removed from various K Basin sludge streams (design-basis) to eliminate the hydrogen generation rate as the constraint for waste loading. For most sludge streams, the 200-gram WIPP $^{239}$Pu FGE limit becomes the most constraining criterion (i.e., the waste loading per 55-gal drum does not exceed the $^{239}$Pu FGE limit of 200 grams) if the uranium metal content can be decreased sufficiently. For the KE floor and pit sludge, the physical volume is likely to be more limiting to drum loading than the FGE content.
The first several rows in Table 1.1 give the maximum uranium metal mass that can be loaded into a RH-TRU drum without exceeding the per-drum hydrogen gas generation rate limit of $3.65 \times 10^{-8}$ moles hydrogen per second.\(^{(a)}\) Based on the assumptions and parameters given in Table 1.1, the quantity of uranium metal per drum of KE floor, pit, and canister sludge is limited to 13.8 grams, assuming no mitigation occurs in the rate of reaction between uranium metal and oxygen-free water and assuming no credit is taken for consumption (reaction) of uranium metal during storage prior to drum shipment to WIPP. If it is assumed that tailored grout will reduce the uranium metal reaction rate by a factor of 4 (as was demonstrated for grout prepared with Portland cement and bentonite, Delegard et al. 2004), then $4 \times 13.8 = 55.2$ grams of uranium metal can be loaded into each drum without exceeding the hydrogen gas generation rate limit. Because the effective particle size of uranium metal in the settler tank sludge (375 µm) is smaller than that of the KE floor and canister sludge (780 µm), the uranium metal mass limit for the settler tank sludge without solidification matrix rate inhibition is only 6.63 grams. The KOP sludge has a higher effective uranium metal particle size (2300 µm) and, therefore, has a higher (i.e., 40.7 grams) metal mass limit without solidification matrix rate inhibition.

The design-basis uranium metal concentrations in each sludge type, provided in Table 1.1, can be combined with the uranium metal mass limits to calculate the maximum volume of sludge that can be loaded in a drum based on the hydrogen gas generation rate. As shown in Table 1.1, the maximum KE Floor & Pit sludge quantity, taking no credit for attenuation of rate by the solidification matrix or uranium metal removal, is

$$13.8 \text{ g U metal} \times \frac{\text{cm}^3}{0.004 \text{ g U metal}} \times \frac{\text{liter}}{1000 \text{ cm}^3} = 3.4 \text{ liters}.$$\

By similar calculations, the per-drum loading for KE canister sludge is 0.34 liters; the loading for KW Canister & Settler Tank sludge is 0.12 liters; and the loading for KOP sludge is 0.0043 liters.

The next section of Table 1.1 shows the maximum loading of settled sludge (liters per drum) that can be achieved with 4-fold tailored grout rate inhibition and with 96.6 wt% uranium metal segregation to 15% of the original sludge volume to obtain a 25-fold increase in sludge loading for the uranium metal-depleted fraction. The combination of tailored grout and uranium segregation provides a $4 \times 25 = 100$-fold increase in sludge loading. With 96.6% uranium removal and a 4-fold decrease in the hydrogen generation rate, the uranium metal no longer constrains drum loading for KE Floor & Pit sludge, because the sludge quantity (345 liters) exceeds the available drum volume (177 liters) by nearly a factor of 2.

The next portion of Table 1.1 addresses the limits to drum loading based on the $^{239}\text{Pu}$ FGE limit of 200 grams.\(^{(b)}\) It is important to note that, because of measurement and process uncertainties, the practical limit for $^{239}\text{Pu}$ FGE likely will be much less than the 200-gram limit used in this evaluation. For purposes of disposition to WIPP, two standard deviations must be added to the actual FGE measurement to calculate the FGE content of a drum. For example, at a more realistic FGE limit of 150 grams per drum,

\(^{(a)}\) The value of $3.65 \times 10^{-8}$ moles H$_2$ per second was obtained from guidance provided by David DeRosa, Fluor Hanford. A similar value of $3.8391 \times 10^{-8}$ moles H$_2$ per second per RH-TRU drum is given in Table 5.2 of Mellinger et al. (2004). The more conservative (lower) value is used in the present evaluation.

\(^{(b)}\) The volume and FGE content of KE Floor & Pit sludge in Table 1.1 does not include the KW NLOP or KW Floor sludge. It is the intent of this table to show the general impact of mitigation; and not to provide a definitive estimate of the total number of drums needed to handle all K Basin sludge.
Table 1.1. RH-TRU Drum Loading Limits

### Drum Loading Limits Based on H₂ Generation

<table>
<thead>
<tr>
<th>Sludge Treatment</th>
<th>Maximum Quantity Uranium Metal per Drum, g&lt;sup&gt;(a)&lt;/sup&gt;</th>
<th>Maximum Design-Basis Settled Sludge Volume per Drum Based on H₂ Limit, liters&lt;sup&gt;(b)&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>KE Floor &amp; Pit</td>
<td>KE Can</td>
</tr>
<tr>
<td>KE Floor &amp; Pit</td>
<td>6.63/26.52</td>
<td>40.7/162.7</td>
</tr>
<tr>
<td>KE Can</td>
<td>16.63/26.52</td>
<td>40.7/162.7</td>
</tr>
<tr>
<td>KW Can &amp; Settler Tank (KW Can)</td>
<td>6.63/26.52</td>
<td>40.7/162.7</td>
</tr>
<tr>
<td>KOP</td>
<td>13.8 in sludge/55.2 in grout</td>
<td>40.7/162.7</td>
</tr>
</tbody>
</table>

### Drum Loading Limits Based on ²³⁹Pu Fissile Gram Equivalent

<table>
<thead>
<tr>
<th>Sludge Treatment</th>
<th>Maximum Sludge Quantity per Drum Based on 200-gram ²³⁹Pu FGE Limit, liters&lt;sup&gt;(c)&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>KE Floor &amp; Pit</td>
</tr>
<tr>
<td>No Mitigation</td>
<td>379.8 *</td>
</tr>
<tr>
<td>Tailored Grout</td>
<td>564 (at 60 liters sludge/drum)</td>
</tr>
<tr>
<td>Settled Sludge Design-Basis Values</td>
<td>KE Floor &amp; Pit</td>
</tr>
<tr>
<td>U Metal Concentration, g U/cm³</td>
<td>0.004</td>
</tr>
<tr>
<td>²³⁹Pu FGE, g</td>
<td>17800</td>
</tr>
<tr>
<td>Sludge Volume, m³</td>
<td>33.8</td>
</tr>
<tr>
<td>Effective U Metal Particle size, µm</td>
<td>780&lt;sup&gt;(f)&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

* Sludge loading in 177-liter-capacity drums (55-gal drum, 85% full) is not limited under these conditions.
** Uranium metal segregation processing is unlikely to be used for KOP sludge.
(a) Drum gas generation limited to 3.65×10⁻⁸ moles H₂/sec; maximum uranium metal quantity calculated based on effective U metal particle size. Uranium corrodes at 60°C (WIPP transportation maximum) according to the SNF Databook rate (Duncan 2001; 67.72 g U/m²·day) in sludge or at one-fourth of the U metal rate in water for Tailored Grout. No H₂ production by radiolysis of water or organics is considered in this evaluation.
(b) Uranium metal segregation steps remove 96.6% of the U metal with the remaining 4% in 85% of the original sludge volume. The metal-enriched fraction must be further processed.
(c) Maximum sludge quantity per drum, ml = \( \frac{200 \times ²³⁹\text{Pu FGE}}{\text{drum}} \times \frac{\text{Sludge volume, ml}}{\text{Sludge } ²³⁹\text{Pu FGE, g}} \). Measurement and process uncertainties likely will limit the ²³⁹Pu FGE to much less than 200 g (e.g., 120-150 g).
(d) Design-basis parameter from Schmidt (2004).
(e) ²³⁹Pu FGE values taken from Mellinger et al. (2004). The ²³⁹Pu FGE was calculated based on fissile isotope concentrations (²³³U, ²³⁷Np, ²³⁹Pu, ²₄₃Am, ²₄₅Cm, and ²₄₉Cf) given for various sludge types (Schmidt 2004) and defined isotopic quality factors for criticality safety.
(f) Effective particle diameter is average of values provided in Table A-2, Appendix A, of Plys and Schmidt (2003), based on SNF Databook reaction rate.
the Settler Tank (KW Canister) sludge loading would be limited to 10.9 liters (rather than 14.4 liters as stated in Table 1.1), and the drum loading based on the hydrogen gas generation rate of the Settler Tank sludge (12 liters) would no longer limit waste loading.

It is seen in Table 1.1 that the sludge loading limits based on hydrogen gas generation rate after accounting for tailored grout and uranium segregation are close to those projected based on the $^{239}$Pu FGE. However, the KOP sludge, and the concentrate streams (uranium metal-rich fractions) arising from the uranium metal segregation process, will contain uranium metal at concentrations that will limit the sludge volume drum loading far below the $^{239}$Pu FGE limit. Consequently, these streams will need to be treated (e.g., oxidize the uranium metal) in order to increase the waste loading to the $^{239}$Pu FGE limit.

1.3 Conceptual Uranium Metal Segregation Process

Because of the high density of the target uranium metal, the separation of uranium metal from K Basin sludge is expected to be very similar to some gold recovery operations. Therefore, the capabilities of commercial gravity concentration technologies were initially assessed by contacting a number of equipment vendors, retailers, and mineral processing experts. The vendors were given information on the processing objectives, and K Basin sludge physical properties (e.g., particle size distribution, particle densities). A pre-conceptual process flow diagram (Figure 1.1) was also provided, assuming a grouted product waste form.

According to the process flow diagram, sludge would be mobilized and pumped to the process feed tank. The feed tank contents also could be agitated (via agitator or slurry mix pump), as required, to maintain the sludge as a uniform slurry. The level of agitation could potentially be set at a sufficiently high rate to break up large agglomerated sludge constituents, such as the friable aluminum hydroxide nodules found in the sludge from aluminum canister corrosion.

**Uranium metal depletion step (primary loop).** From the process feed tank, the slurry will be fed to a gravity concentration unit. Commercially available devices that may be applicable for this operation include pulsed jigging devices and centrifugal concentrators.

The tails stream, depleted in uranium metal from the concentration device, would be recycled back to the feed tank and then back through the device to achieve higher uranium metal removal efficiency. When the batch is completed, the tails would be pumped to the grout feed tank.

The concentrate stream, enriched in uranium metal, would be staged in a separate tank for additional processing. Although not shown in Figure 1.1, a water input stream will be required for operation of the uranium metal concentrator. Treatment (e.g., settling or filtration) to remove excess water may also be required.

**Uranium metal concentrate cleaning (upgrading) step (secondary loop).** The uranium metal-rich concentrate stream will contain residual non-uranium metal material, such as aluminum hydroxide nodules, iron hydroxides, and some uranium oxyhydrates. Most of these tramp hydroxides/hydrates must be removed from the concentrate stream if the uranium metal particles are dispositioned to MCOs. The cold vacuum drying conditions are not sufficient to decompose the hydroxides and remove the water from the crystal hydrates. Commercial centrifugal concentrators are capable of increasing the uranium metal concentration and decreasing the volume of the concentrate stream. However, it is unlikely that the resulting uranium metal concentration will be sufficiently high to allow upgraded concentrate to be processed through the cold vacuum drying system without further upgrading/cleaning steps.
The concentrated uranium metal stream from this operation then would be loaded into a container for subsequent processing. The tails from the upgrading process would be recycled back to the process feed tank.

**Figure 1.1.** Process Flow Diagram for Conceptual K Basin Sludge Uranium Metal Segregation
2.0  K Basin Sludge Segregation Simulant

A sludge simulant was developed specifically to test the viability and performance of vendor equipment for separating uranium metal particles out of sludge. Because the segregation simulant was used to test equipment that employs gravity or density separation methods, the formulation was developed considering characteristics critical to these separation methods (particle size distribution, particle density, and physical behavior). Of particular note is the use of a cobalt-cemented tungsten carbide (referred to as “W/Co”) particulate to simulate the uranium metal fragments in the sludge. The W/Co has similar particle size and shape, and approaches the density of the uranium metal. With the lower density of the W/Co (14.5 to 15.0 g/cm³) compared to uranium metal (19 g/cm³), recovery of the W/Co from the simulant is expected to provide a greater challenge than recovery of uranium metal from the actual sludge. Stainless steel powder was selected to represent uranium oxides and hydrates. The other simulant components, flyash, silica powder, goethite (Fe₂O₃·H₂O), and “Kleen Blast,” were chosen to represent the non-uranium remainder of the sludge components (e.g., sand and dust, rust, spalled concrete, aluminum oxide/hydroxide, etc.).

The composition of the segregation simulant was largely based on a simulant previously characterized and used to evaluate uranium metal distribution during loading of K Basin sludge into containers (Schmidt and Elmore 2002). The composition of the Schmidt and Elmore (2002) settling simulant was formulated to resemble a 40:60 volume mixture of KE canister and KE floor sludge at safety basis parameters. In turn, this settling simulant was based in part on the K Basin simulant developed for the large-diameter-container proof-of-principle (PoP) test.(a) The PoP simulant was composed of 8 wt% W/Co, 17 wt% flyash (typically between 0.5 and 180 µm), 37.5% Min-U-Sil-40 (a ground silica, with the majority of particles between 5 and 20 µm), and 37.5% foundry sand (majority of particles between 100 and 400 µm). The particle size distribution (PSD) and settling behavior of the PoP simulant were found to be very similar to that of KE Basin floor sludge. Furthermore, the yield strength of the K Basin PoP simulant was found to be 93 Pa after 24 hours, and 106 Pa after 48 hours, which correlates reasonably well with values found for samples of actual floor and canister sludges. The PoP simulant development was largely based on simulant experience related to successful K Basin sludge sampling equipment development since 1995.

The segregation simulant for vendor testing was prepared by PNNL in accordance with the Sludge Retrieval and Disposition Project approved test instruction.(b) The composition and the bases for the simulant component selection are summarized below.

- **W/Co to simulate metallic uranium: 3.2 wt% (dry basis).**

  **Material Description**  Cobalt-cemented tungsten carbide, KENFCRESP, was obtained from Kennametal, Fallon, NV. The composition of W/Co is 6.9 wt% Co, 5.7 wt% C, and 0.20 wt% Ti, with the balance (~87 wt%) being W. The particle density of W/Co is 14.5 to 15.0 g/cm³ with bulk


dry density of \( \sim 10 \text{ g/cm}^3 \). The particle sizes range from 250 to 1500 \( \mu \text{m} \), with an average particle size of \( \sim 580 \mu \text{m} \) (50% -1500 \( \mu \text{m} \), +580 \( \mu \text{m} \); 50% -580 \( \mu \text{m} \), +250 \( \mu \text{m} \)).

The lower particle density of the W/Co (14.5-15.0 g/cm\(^3\)) compared to uranium metal (19 g/cm\(^3\)) gives a decreased density gradient between the uranium metal simulant and the remainder of the simulated sludge components, and increases the recovery challenge.

**Basis** It is expected that most of the uranium fragments in actual K Basin sludge, not including KOP sludge, will be between 250 and 2000 \( \mu \text{m} \). The effective average uranium metal particle diameter in KE canister sludge is 780 \( \mu \text{m} \) (Plys and Schmidt, 2003, Appendix A, Table A-2). The W/Co particle size and density are smaller than those expected for uranium metal in K Basin sludge and thus act to pose more of a separation challenge.

- **Stainless Steel Powder to simulate non-metallic uranium phases: 32 wt\% (dry basis).**

**Material Description** Spherical-shaped stainless steel powder, A434L, was obtained from Hoeganaes, Cinnaminson, NJ. The composition of A434L is 17 wt\% Cr, 1.0 wt\% Mo, 0.2 wt\% Mn, 0.9 wt\% Si, 0.02 wt\% C, with the balance (~81 wt\%) being Fe. The particle density is 7.8 g/cm\(^3\), and all particles are -120 mesh (less than 125 \( \mu \text{m} \)) with an average particle size (volume basis) of \( \sim 60 \mu \text{m} \).

The average particle density of the non-metallic uranium species in KE floor and canister sludge is estimated to be 7.5 g/cm\(^3\), near that of the stainless steel. However, the average particle size of high-uranium-content KE and KW canister sludge after shearing/sonication is less than 60 \( \mu \text{m} \) (smaller than the stainless steel). The separation gradient between the uranium metal simulant (W/Co) and the uranium oxyhydrate simulant (stainless steel spherical powder) thus is lower than the gradient between uranium metal and uranium oxyhydrate in the actual sludge.

**Basis** During corrosion of uranium metal in water, UO\(_2\) particles on the order of 10 \( \mu \text{m} \) and smaller are generated. Also, the average particle size of high-uranium-content KE and KW canister sludge after shearing and sonicication is less than 60 \( \mu \text{m} \) (Makenas et al. 1997, 1998). UO\(_2\) generated from corrosion will further react to form higher uranium oxides (U\(_4\)O\(_9\), U\(_3\)O\(_8\)) and U(VI) hydrates (e.g., schoepite) with a range of particle densities between about 5 and 11 g/cm\(^3\). Based on analyses developed in Schmidt and Delegard (2002), the average particle density of the non-metallic uranium species in KE floor and canister sludge is estimated to be about 7.5 g/cm\(^3\). Therefore, stainless steel powder (~7.8 g/cm\(^3\)) was selected as a suitable surrogate for the non-metallic uranium.

Note, however, that while the majority of the non-metallic uranium particles are less than 250 \( \mu \text{m} \), non-metallic uranium particles, fragments, and agglomerates larger than 250 \( \mu \text{m} \) are present in K Basin sludge samples. As an example, about 40 wt\% (dry basis) of KE canister sludge sample 96-06 was made up of particles greater than 710 \( \mu \text{m} \) (Makenas et al. 1997). On a dry weight basis, the uranium concentration of 96-06 was about 83 wt\%, which would indicate the larger-diameter (greater than 700 \( \mu \text{m} \)) particles contained an appreciable quantity of uranium. Use of the stainless steel powder to simulate the non-metallic uranium simplifies the simulant.

- **Kleen Blast to simulate larger-diameter non-uranium particles: 29 wt\% (dry basis).**

**Material Description** Kleen Blast (from Kleen Blast Abrasives, Tacoma, WA) is a sand blasting product sold in a range of size distributions, including -6+8 mesh, -8+12 mesh, and -16+30 mesh. Kleen Blast is a homogenous glassy slag mixture, chemically composed of 45 wt\% silica, 23 wt\%
iron oxide, 19 wt% calcium oxide, and 7 wt% aluminum oxide. Its particle density is approximately 2.8 g/cm³. To simulate the desired range of particle sizes, the 29 wt% Kleen Blast component was allocated according to the following distribution:

-6+8 mesh (3360 to 2380 µm) = 10 wt%
-8+12 mesh (2380 to 1680 µm) = 10 wt%
-16+30 mesh (1180 to 600 µm) = 9 wt%

**Note:** The actual particle size range was found to be greater than that indicated by Kleen Blast mesh specifications.

The Kleen Blast simulant component contains a higher fraction of plus (greater than) 500-µm particles than the KE floor and canister sludge non-uranium constituents, e.g., Al(OH)₃. The larger particle size in the simulant for the non-uranium constituents produces a lower separation gradient from the simulant metal than the non-uranium constituents in the actual sludge pose with the uranium metal fragments.

**Basis**  Approximately 25 wt% of the actual KE floor and canister sludge is composed of particles greater than 500 µm. However, very limited data exist on particle density as a function of particle size for actual K Basin sludge (Bredt et al. 1999). For canister sludge, particles between 500 and 1410 µm exhibited a density of 4.63 g/cm³. The particle densities of larger canister sludge particles were 2.89 g/cm³ (1410 to 4000 µm) and 2.23 g/cm³ (4000 to 6350 µm). For floor sludge, the particle densities were 3.14 g/cm³ (<250 µm), 2.78 g/cm³ (250 to 500 µm), and 2.63 g/cm³ (500 to 1410 µm). It is recognized that the use of a single component (Kleen Blast) to simulate all large non-metallic uranium particles is a simplification of the complex composition of the actual K Basin sludge.

The W/Co, stainless steel powder, and Kleen Blast constitute 64.2 wt% of the sludge simulant on a dry weight basis. The remaining 35.8 wt% simulates fine particulate sludge components.

- **Flyash to simulate fine particulate in the sludge: 15.4 wt% (dry basis).**
  
  **Material Description**  Class F Flyash was obtained from Redimix, Pasco, WA. The flyash has a particle density ranging from ~2.2 to 2.5 g/cm³ and a particle size between 0.5 and 180 µm.

  **Basis**  Flyash has been used extensively and successfully in a number of prior K Basin sludge simulants.

- **Min-U-Sil 40 to simulate fine particulate in the sludge: 15.4 wt% (dry basis).**
  
  **Material Description**  Min-U-Sil 40 is a ground high-purity silica obtained from U.S. Silica, Berkeley Springs, WV. Its particle density is 2.65 g/cm³, and its mean particle diameter is 11 µm.

  **Basis**  Min-U-Sil has been used extensively and successfully in a number of prior K Basin sludge simulants.
• Goethite to simulate submicron particulate in the sludge: 5 wt% (dry basis).

**Material Description**  Goethite, FeOOH [iron (III) oxide monohydrate or Fe₂O₃·H₂O, was obtained from Strem Chemical, Inc., Newburyport, MA. Goethite has a particle density of 4.0 g/cm³ and an average (volume percent basis) particle size of 0.199 µm with 99% of all particles less than 1 µm.

**Basis**  Goethite has been identified in a number of K Basin sludge samples. Also, this very fine particulate material increases the viscosity and provides a slimy texture to the simulant. This provides more resistance to the mechanical separation of the surrogate uranium metal (W/Co) particles from the remainder of the simulant sludge.

Figure 2.1 shows the simulant components at their respective mass ratios. Table 2.1 summarizes the simulant composition and provides the component particle densities and component particle size distributions. Figure 2.2 compares the PSD of the simulant to the average PSDs from actual KE floor and canister samples (Schmidt 2004). Because of its importance to this testing, the W/Co fragments were sieved using a large number of screens to obtain a more precise PSD for this component. Table 2.2 provides the detailed W/Co sieving results.

The Spent Nuclear Fuel Project definition of sludge is any particulate material that will pass through a screen with 0.25-in. (6350-µm) openings. In sludge samples from the K Basins, the amount of particles between 4000 and 6350 µm has been found to be relatively small (e.g., 0 to 4%, Schmidt 2004). Consequently, the current uranium metal segregation simulant was developed to provide a reasonably challenging case, with its range of particles being no greater than 4000 µm.

![Figure 2.1. Components of K Basin Sludge Segregation Simulant](image)
Table 2.1. Metal Segregation Simulant Composition and Properties

<table>
<thead>
<tr>
<th>Property</th>
<th>W/Co</th>
<th>SS Powder</th>
<th>Kleen Blast</th>
<th>Flyash</th>
<th>Min-U-Sil-40</th>
<th>Goethite</th>
<th>Cumulative Simulant</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wt Percent, dry</td>
<td>3.2</td>
<td>32</td>
<td>10</td>
<td>10</td>
<td>9</td>
<td>15.4</td>
<td>15.4</td>
</tr>
<tr>
<td>Particle density, g/cm³</td>
<td>14.5 – 15.0</td>
<td>7.8</td>
<td>2.6 – 2.8</td>
<td>2.6 – 2.8</td>
<td>2.6 – 2.8</td>
<td>2.2 – 2.5</td>
<td>2.65</td>
</tr>
<tr>
<td>Particle Size, μm</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4000</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>2380</td>
<td>100</td>
<td>100</td>
<td>74.91</td>
<td>64.61</td>
<td>99.69</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>2000</td>
<td>100</td>
<td>100</td>
<td>26.10</td>
<td>38.46</td>
<td>94</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>1500</td>
<td>100</td>
<td>100</td>
<td>10</td>
<td>8</td>
<td>50</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>1000</td>
<td>83</td>
<td>100</td>
<td>0.5</td>
<td>5</td>
<td>40</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>750</td>
<td>65.7</td>
<td>100</td>
<td>0</td>
<td>2</td>
<td>20</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>500</td>
<td>21.6</td>
<td>100</td>
<td>0</td>
<td>0.5</td>
<td>10</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>250</td>
<td>0.41</td>
<td>100</td>
<td>0</td>
<td>0</td>
<td>1.69</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>106</td>
<td>0</td>
<td>83.6</td>
<td>0</td>
<td>0</td>
<td>0.5</td>
<td>97.8</td>
<td>100</td>
</tr>
<tr>
<td>100</td>
<td>0</td>
<td>80.7</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>97.4</td>
<td>100</td>
</tr>
<tr>
<td>53</td>
<td>0</td>
<td>39.5</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>88</td>
<td>98</td>
</tr>
<tr>
<td>30</td>
<td>0</td>
<td>17.3</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>75</td>
<td>94</td>
</tr>
<tr>
<td>20</td>
<td>0</td>
<td>7.8</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>65</td>
<td>83</td>
</tr>
<tr>
<td>10</td>
<td>0</td>
<td>1.3</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>45.6</td>
<td>46</td>
</tr>
<tr>
<td>5</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>30</td>
<td>26</td>
</tr>
<tr>
<td>1</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>7.8</td>
<td>7</td>
</tr>
</tbody>
</table>

Measured Segregation Simulant Properties:
- Settled density = 2.1 g/cm³.
- Weight percent solids in settled simulant = 71 wt%.
- Volume fraction water in settled simulant = 0.61.
Figure 2.2. Comparison of the Particle Size Distributions of the Uranium Segregation Simulant (U-Seg Simulant) to Average Particle Size Distribution of KE Floor and Canister Sludge Samples

<table>
<thead>
<tr>
<th>Sieve Size, µm</th>
<th>Wt% Less Than</th>
</tr>
</thead>
<tbody>
<tr>
<td>2000</td>
<td>100.00</td>
</tr>
<tr>
<td>1180</td>
<td>99.71</td>
</tr>
<tr>
<td>1000</td>
<td>83.03</td>
</tr>
<tr>
<td>850</td>
<td>72.64</td>
</tr>
<tr>
<td>710</td>
<td>62.98</td>
</tr>
<tr>
<td>600</td>
<td>56.34</td>
</tr>
<tr>
<td>500</td>
<td>21.55</td>
</tr>
<tr>
<td>425</td>
<td>12.91</td>
</tr>
<tr>
<td>355</td>
<td>5.22</td>
</tr>
<tr>
<td>300</td>
<td>1.66</td>
</tr>
<tr>
<td>250</td>
<td>0.41</td>
</tr>
<tr>
<td>212</td>
<td>0.18</td>
</tr>
</tbody>
</table>
3.0 Gekko Systems Testing

This section discusses the gravity concentration testing conducted at Gekko Systems. Two types of tests were performed: shaker table testing to evaluate uranium metal segregation and recovery and spinner testing to evaluate upgrading the concentrate stream from the tabling by further removal of the non-uranium metal components.

3.1 Equipment Description

The Gekko test systems and equipment are briefly described below. Additional information on Gekko Systems mineral concentration equipment, installations, and research services can be found at www.gekkos.com.

3.1.1 Shaker Tables

To conservatively assess the applicability of gravity concentration for uranium metal removal from K Basin sludge, Gekko performed a series of bench-scale shaker table tests (“tabling”). Gekko has found that the separation results achieved from tabling can be used to predict the performance of their InLine Pressure Jig (IPJ) concentrators. Furthermore, tabling can be conducted cost effectively with relatively small feed batches. In shaker table testing, a slurry (typically 25 wt% solids) is fed to the upper edge of a sloping table (riffle-decked in a horizontal plane). The flowing film separates the small dense particles (which quickly move to the lower, slower moving layer of the film) from the coarse, light particles. This effect is enhanced by vibrating the table at a right angle to the water flow in a slow forward stroke and a fast return stroke. The net effect is that particles move diagonally across the table. The suspended solids are held up in pools behind the riffles as the material moves across the table. The shaking action of the table results in size classification and specific gravity stratification (Weiss 1985). Shaker tables are typically operated with particles ranging from 75 to 3000 µm.

3.1.2 Jigs

A conventional jig separates particles using a pulsating column of water. A slurry is subjected to vertical pulsations that alternately lift and lower the slurry mass. The upward pulse will tend to loosen and expand the particle bed, and the downward pulse will tend to consolidate the bed. The denser particles will migrate progressively towards the bottom of the bed with each pulse cycle. The downward movement of the lighter particles is retarded by the upward pulse, but the lighter particles are not accelerated fast enough to settle by the downward pulse. A bed of dense particles builds up at the bottom, and lighter particles leave the device with the overflow water. To accentuate density differences relative to particle size differences, short jiggling cycles can be used (short, fast strokes). The short cycles allow small denser particles to be affected more by initial acceleration (mass effect) than by the terminal velocity (size effect). Conventional jigs are typically operated with particles ranging from 150 to 3000 µm.

3.1.3 InLine Pressure Jig

The operating principle of the InLine Pressure Jig is described by Gray (1997) and Moony and Gray (1998). The Gekko IPJ was developed for the recovery of coarse, free gold (typically ranging from 40 to
>600 µm) from low-grade ores, and has also been used to recover copper, silver, tantalum, garnets, and diamonds. The IPJ can accept feed particle sizes up to 25 mm (25,000 µm).

Figure 3.1 shows the operating principles and general flow through an IPJ. The Gekko IPJ is a single-hutch circular jig with a movable screen, where the hutch is the large conical central chamber. The internal screen is driven by a hydraulic drive through a central shaft that protrudes through the lower diaphragm. The screen support frame is sealed to the hutch with a flexible rubber diaphragm and is held in position by flexible rubber blocks that oscillate to allow for vertical movement of the frame. The drive shaft is sealed at the base of the hutch with a flexible rubber diaphragm. The unit is encapsulated and fed under pressure. The feed is split, and concentrate and tailings are continuously discharged. The only moving internal parts are the rubber diaphragms and the screen support blocks. The hydraulic drive is external and can be located remotely to the unit.

The feed (typically 30 to 80 wt% solids) is introduced through the feed pipe into the distributor where its direction is reversed, and it then flows out into the deceleration chamber. Due to the large volume of the deceleration chamber, the flow rate drops below the terminal velocity of the suspended solids. The heavy particles drop to the bed and are captured in the slow flowing material in the separation interface on top of the bed. The bed consists of ragging (typically lead shot) located on top of a wedge wire screen. The bed is pulsed through the central drive shaft by the drive ram at the base of the machine. During the down-stroke of the bed, particles are segregated as a result of their relative density, size, and shape. The up-stroke produces a suction phase, which causes the heavier concentrate to be drawn down through the ragging and into the hutch. The lighter material flows to the outer circumference of the bed and overflows the screen support frame to report to the tails outlet, where it is discharged under pressure. Hutch water is added through the hutch water inlet to provide concentrate water and pulse water through the screen.
The advantages of the IPJ cited in the vendor literature include low water consumption, ability to operate with poor hutch water quality (up to 2% solids), high operability/lower maintenance (two moving parts), flexibility to adjust the mass pull to the concentrate from 1 to 20%, and a low pressure drop. For the K Basin sludge, Gekko’s smallest IPJ, Model IPJ600 (pictured in Figure 3.2), would likely be an appropriate unit. Key unit specifications are listed in Table 3.1.

![Figure 3.2](image). Gekko Systems InLine Pressure Jig, Model IPJ600. Capacity of 1 to 2 metric tons per hour (dry solid basis). The unit is compact and sealed, and contains only two moving parts.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight of IPJ600 (dry)</td>
<td>kg</td>
<td>200</td>
</tr>
<tr>
<td>Volume of unit</td>
<td>L</td>
<td>200</td>
</tr>
<tr>
<td>Feed throughput capacity (dry solids basis)</td>
<td>MT/hr</td>
<td>1 to 2</td>
</tr>
<tr>
<td>Solids concentration in feed</td>
<td>wt%</td>
<td>30 to 80</td>
</tr>
<tr>
<td>Maximum feed size</td>
<td>µm</td>
<td>6000-25,000</td>
</tr>
<tr>
<td>Concentrate rate (dry solids basis)</td>
<td>kg/hr</td>
<td>30 to 600</td>
</tr>
<tr>
<td>Hutch water addition rate</td>
<td>Lpm</td>
<td>6 to 30</td>
</tr>
<tr>
<td>Pulse rate</td>
<td>cycles/sec</td>
<td>50 to 200</td>
</tr>
<tr>
<td>Supply power</td>
<td>kW</td>
<td>1.5</td>
</tr>
<tr>
<td>Maximum operating pressure</td>
<td>kPa</td>
<td>180</td>
</tr>
<tr>
<td>Pressure drop</td>
<td>kPa</td>
<td>10</td>
</tr>
</tbody>
</table>

3.3
3.1.4 **InLine Spinner**

The InLine Spinner (ISP), pictured in Figure 3.3, is a fully automated, enclosed centrifugal concentrator, which can be used to upgrade the concentrate stream generated from the IPJ. In the ISP, concentrates are fed in the base of a spinning bowl that operates while full of slurry. The heavy particles are forced to the outside of the bowl and become captured in a series of riffles (concentric grooves). Cutter bars, operating parallel to the inner face of the bowl, create a turbulent zone that enhances the separation potential and the capacity for dense particles to replace the lighter particles caught by the centrifugal force. The continuous slurry feed pushes the lighter material out over the edge of the bowl. At controlled time intervals (dump cycle), feed to the unit is stopped, and the concentrate is removed (washed) from the bowl. Figure 3.4 shows a cut-away diagram.

Advantages cited in the vendor literature include the ability to receive particles up to 4000 µm (and occasional large particles up to 10,000 µm), low water consumption, and low maintenance. For the K Basin sludge, the unit shown in Figure 3.3 (Model ISP02) would likely be an appropriate unit. Key unit specifications are listed in Table 3.2.

![Figure 3.3](image_url) **Figure 3.3.** Gekko Systems ISP02 InLine Spinner. Can process up to 3 metric tons per hour (dry solid basis).
Table 3.2. Specifications for the Gekko Inline Spinner Model ISP02

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight of ISP02 (dry)</td>
<td>kg</td>
<td>250</td>
</tr>
<tr>
<td>Bowl speed</td>
<td>rpm</td>
<td>100</td>
</tr>
<tr>
<td>Feed throughput capacity (dry solids basis)</td>
<td>MT/hr</td>
<td>2 to 3</td>
</tr>
<tr>
<td>Solids concentration in feed</td>
<td>wt%</td>
<td>5 to 20</td>
</tr>
<tr>
<td>Maximum recommended feed size</td>
<td>µm</td>
<td>4000⁶⁰</td>
</tr>
<tr>
<td>Concentrate rate (dry solids basis)</td>
<td>kg/hr</td>
<td>30 to 600</td>
</tr>
<tr>
<td>Water consumption per dump cycle</td>
<td>L</td>
<td>30</td>
</tr>
<tr>
<td>Supply power</td>
<td>kW</td>
<td>2.2</td>
</tr>
</tbody>
</table>

(a) ISP can handle occasional large particles up to 10,000 µm.

3.2 Test Results

The results from the shaker table tests and spinner tests are presented here. Gekko’s test report is provided in Appendix A, and includes graphical and tabular data from each test and a conceptual flowsheet for processing K Basin sludge.
### 3.2.1 Shaker Table Testing

Three shaker table tests were conducted with ~20 kg (dry basis) of simulant each. In the tabling tests, simulant slurry feed was segregated into a concentrate fraction(s) (tungsten-enriched stream) and a tails fraction (tungsten-depleted stream).

For the first tabling test, simulant provided by PNNL was split (dry), and a representative feed sample was obtained. Next, the sample was mixed with water to form a slurry consisting of about 50 to 60 wt% solids. The slurry was then processed on a shaker table to determine the potential for single-pass gravity uranium (represented by W/Co surrogate) recovery. To develop tungsten recovery data as a function of mass pull, the concentrate was collected from the table in four distinct fractions (collected from different table locations), which together constituted about 31 wt% (dry basis) of the total feed mass. The remaining 69 wt% (dry basis) was collected as a single tails fraction. When the tabling was completed, all fractions were dried and then passed through a 500-µm sieve to produce plus and minus 500-µm samples from each sample. Representative subsamples were collected from each fraction, and these subsamples were assayed (by laboratories in Australia) for tungsten, cobalt, iron, nickel and chromium to produce a recovery-yield curve for each element. Table 3.3 summarizes the assay methods and the reported detection limits.

The results from the first shaker table test are summarized in Figure 3.5, which shows the recovery of the simulant components as a function of concentrate mass pull (yield). For example, at a mass pull of about 30%, the tungsten recovery is above 95%. As expected, the cobalt recovery tracks reasonably well with the tungsten recovery, because the W/Co fragments used as a surrogate for uranium metal are about 87 wt% W, 6.9 wt% Co, and 5.7 wt% C, and no other simulant component contains significant cobalt or tungsten.

The iron, nickel, and chromium (mostly present as stainless steel powder) together exhibited similar behavior, with recoveries ranging from 20 to 25% at a mass pull of 30%. The relative behavior of iron, nickel, and chromium compared with tungsten and cobalt is a good indication that uranium oxides/hydrates can effectively be segregated from uranium metal.

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Technique</th>
<th>Detection Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni, Co</td>
<td>Mixed acid digest, including hydrofluoric acid, with atomic absorption spectrometry analysis</td>
<td>2 ppm, 5 ppm</td>
</tr>
<tr>
<td>Cr, Fe</td>
<td>Na₂O₂ fusion, with inductively coupled plasma (ICP)-optical emission spectrometry (OES) analysis</td>
<td>0.1%</td>
</tr>
<tr>
<td>W</td>
<td>Na₂O₂ fusion, with ICP-mass spectrometry analysis</td>
<td>5 ppm</td>
</tr>
<tr>
<td>W</td>
<td>X-ray fluorescence</td>
<td>Not stated.</td>
</tr>
</tbody>
</table>

Table 3.3. Methods and Detection Limit for Element Assays
Figure 3.5. K Basin Segregation Simulant Tabling Recovery-Yield Curve for Shaker Table Test 1. Recoveries of tungsten (W), cobalt (Co), chromium (Cr), iron (Fe), and nickel (Ni) as a function of percent mass recovered to concentrate stream (yield).

Based on the results from the first tabling test, Shaker Table Tests 2 and 3 were conducted at similar concentrate mass pulls (31.5 and 32.3 wt% mass pull to concentrate, respectively). For Tests 2 and 3, the concentrates were collected as single samples rather than as a series of concentrate fractions.

Table 3.4 summarizes the results from the three shaker table tests (and also includes the overall results of the spinner testing). The results from the tabling tests demonstrate consistency and repeatability. Approximately 97 to 99% of the tungsten was recovered into about 30 to 33% of the sample mass (using a shaker table). Based on bulk density measurement of the dried concentrate and tails samples, the concentrate fractions from the shaker table tests comprised 21 to 24% of the sample volume (i.e., bulk density of concentrate sample was generally greater than the bulk density of the tails samples).

The tungsten concentration in the simulant feed was 2.78 wt% (dry basis) and ranged from 8.3 to 9.3 wt% in the concentrate stream (~30 wt% of the sample). In the tails stream (about 70 wt% of the test sample mass), the tungsten concentration ranged from 0.05 to 0.13 wt% (dry basis), a factor of 20 to 55 below that of the feed.

Only about 20 to 25% of the stainless steel powder was recovered in the concentrate (i.e., good separation of the tungsten from the stainless steel).
### Table 3.4. Summary of Shaker Table and Spinner Tests

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Stream Split (a)</th>
<th>Percent Tungsten (W) Recovered to Concentrate (b) (based on analyses)</th>
<th>Overall Analytical Mass Balance Check (c)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Concentrate (enriched stream)</td>
<td>Tails (depleted stream)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Wt% of Feed</td>
<td>[W] Wt% (c)</td>
<td>Wt% of Feed</td>
</tr>
<tr>
<td>Shaker Table Tests 1, 2, and 3 conducted with simulant feed containing ~2.78 wt% tungsten</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Shaker Table 1</td>
<td>30.6</td>
<td>8.5</td>
<td>69.4</td>
</tr>
<tr>
<td>Shaker Table 2</td>
<td>31.5</td>
<td>9.3</td>
<td>68.5</td>
</tr>
<tr>
<td>Shaker Table 3</td>
<td>32.3</td>
<td>8.3</td>
<td>67.7</td>
</tr>
<tr>
<td>Spinner Upgrade Tests 1 and 2 conducted with concentrate from Shaker Table testing used as feed</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Spinner 1 (d)</td>
<td>52.8</td>
<td>7.6</td>
<td>47.2</td>
</tr>
<tr>
<td>Spinner 2 (e)</td>
<td>33.1</td>
<td>20.3</td>
<td>66.9</td>
</tr>
<tr>
<td>Combined results from Shaker Table Tests and Spinner Upgrade Tests</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Shaker Table 3 + Spinner 2 (actual results)</td>
<td>10.7</td>
<td>20.3</td>
<td>89.3</td>
</tr>
<tr>
<td>Shaker Table 3 + Spinner 2 (theoretical (f) results)</td>
<td>10.7</td>
<td>23.3 (f)</td>
<td>89.3</td>
</tr>
<tr>
<td>Shaker Table 3 + Spinner 1 (theoretical (f) results)</td>
<td>17.1</td>
<td>16.0 (f)</td>
<td>82.9</td>
</tr>
</tbody>
</table>

(a) Feed stream was segregated into tungsten-enriched stream (concentrate) and tungsten-depleted stream (tails). Stream split (mass percent) and tungsten concentration (by assay) in each stream are provided.

(b) Tungsten-containing particles in feed ranged from 250 to 1500 µm. Subsamples from testing were sieved at 500 µm and split into two fractions to examine tungsten recovery as a function of particle size. Overall recovery is combined recovery of plus 500-µm and minus 500-µm splits. Recoveries are based on tungsten measured (assayed) in concentrate and tails.

(c) Check on measured (assayed) tungsten in product streams vs. quantity used to prepare test material. Value = 100%× [mass of W in concentrate (by assay) + mass of W in tails (by assay)] divided by mass of W in feed (mass was measured).

(d) Concentrate from Shaker Table 2 used as feed to Spinner 1. Overall analytical mass balance check on tungsten for this test was only 43%, most likely due to an inhomogeneous splitting of Shaker Table 2 concentrate.

(e) Concentrate from Shaker Table 3 used as feed to Spinner 2.

(f) Tungsten concentrations in concentrate and tails were calculated based on 2.78 wt% tungsten in feed, mass splits as measured, recoveries as measured, and 100% mass balance.

As previously noted, all samples were dried and then passed through a 500-µm sieve to produce plus and minus 500-µm sample fractions. Table 3.4 shows that the tungsten recovery results were similar for the two size fractions; 97.6 to 99.5% of the plus 500-µm and 95.2 to 97.7% of the minus 500-µm tungsten was recovered into the concentrate. [From Section 2.0, approximately 22% of the W/Co particles in the segregation simulant are less than 500 µm.]
Mass balance calculations were performed as a measure of quality control over the testing, subsampling, and sample analyses. For these calculations, the mass of tungsten recovered in concentrate and tails samples, as measured by chemical assay, was compared to the mass of tungsten known to be added (measured by weighing) to the segregation simulant during the simulant preparation. As shown in Table 3.4, mass balance closures from the shaker table testing were excellent, ranging from 96 to 109%.

3.2.2 Spinner Testing

In the first spinner test, a fraction of the concentrate from Shaker Table Test 2 was processed through a laboratory spinner, and 52.8 wt% of the feed stream was collected as upgraded spinner concentrate. This upgraded concentrate contained 99.5 wt% of the recovered tungsten. Because the mass balance was low (only 43% of the tungsten in the test was accounted for), a second spinner test was conducted. For the second spinner test, concentrate from Shaker Table Test 3 was used as the feed. In Spinner Test 2, 33.1% of the feed was collected as upgraded concentrate, with 89.7 wt% of the recovered tungsten reporting to this concentrate stream. Reasonable closure of the mass balance (89%) was achieved in the second spinner test. The key results from the spinner testing are provided in Table 3.4.

In Spinner Test 1, the results indicated that the spinner concentrate contained a lower tungsten concentration (7.6 wt%) than the spinner feed (concentrate from Shaker Table Test 2, 9.3 wt% tungsten). This anomaly, which contributes to the poor material balance, most likely resulted from a non-uniform sample split, as only a portion of the concentrate from Shaker Table Test 2 was used for Spinner Test 1. In comparison, the concentrate from Spinner Test 2 contained about 20 wt% tungsten.

The concentration of tungsten in the spinner tails is probably not a critical parameter with respect to applicability of the use of the spinner to process K Basin sludge. This is because the spinner tails most likely would be returned to the jig feed and recycled through the segregation system.

The lower portion of Table 3.4 also shows the effect of combining the Shaker Table 3 results with Spinner Test 2 results for a single-pass process (i.e., no recycle; tails from spinner are combined with tails from jig). For this combination, the final concentrate stream contains ~90% of the tungsten in only 10.7 wt% (6.6 vol%) of the initial total feed. The relatively low tungsten recovery for this combination is an artifact of the relatively low tungsten recovery to the concentrate in Spinner Test 3. If the spinner tails were recycled back through the jig, a significantly greater tungsten recovery for the combined spinner/tabling process would be obtained.

The last two rows in Table 3.4 show theoretical combined results, using the measured tungsten recoveries and assuming 100% closure of the material balances. The calculations reflected in these rows assume a single-pass process (i.e., no recycle of spinner tails). Figure 3.6 depicts the theoretical combination of Shaker Table 3 and Spinner Test 1 results. For this combination, 98.2% of the tungsten is recovered into 17.1% of the feed mass.

3.3 Overall Assessment of Amenability of K Basin Sludge to Gekko Gravity Concentration Equipment

In shaker table testing, greater than 96% of the uranium metal surrogate was concentrated into about 30 wt% of the simulant sludge mass. When combined with a tailored matrix for final packaging, this level of uranium metal removal likely will be sufficient to eliminate the hydrogen gas rate limit as the controlling waste-loading constraint (for disposal to WIPP) for most of the K Basin sludge.
Testing with the InLine Spinner showed the uranium metal surrogate concentration in the concentrate stream could be increased by more than a factor of 2, and the final volume of concentrate stream could be reduced to about 6.6% of the original feed volume. Also, based on the testing results, a grinding or prescreening unit operation would not be needed to achieve a high uranium metal recovery and clean-up, because Gekko equipment can process feed that contains particles up to 25,000 µm.

**Figure 3.6.** Combined Results of Gekko’s Shaker Table 3 Results with Spinner Test 1, Assuming 100% Mass Balance Closure

Overall, the testing demonstrated the technical feasibility of Gekko gravity concentration equipment for segregating uranium metal in K Basin sludge. Gekko has previously examined underwater application of the InLine Pressure Jig, and believes underwater deployment for both the IPJ and ISP (with a few modifications) is technically feasible.
4.0 Knelson Concentrators Testing

This section discusses the laboratory and pilot-scale testing conducted by Knelson Concentrators. Additional information on the Knelson Concentrators mineral concentration equipment, installations, and research services can be found at www.knelson.com. Knelson’s reports on the laboratory and pilot-scale testing are provided in Appendix B. These reports contain additional details on the system parameters and include graphical and tabular data for both tests.

4.1 Laboratory-Scale Testing

Testing was conducted at Knelson to evaluate the performance of their laboratory-scale batch centrifugal concentrator (KC-MD3 – Figure 4.1) with the K Basin segregation simulant. A 4-kg batch of simulant (dry basis) was shipped to Knelson and was processed in five passes through the laboratory concentrator. The full mass of simulant was fed to the concentrator in a single batch, followed by fluidization and segregation. For each pass, a small mass pull, representing about 3 to 5 wt% of the simulant, was removed as the concentrated stream of higher-density components of the simulant. The concentrate and tails were then dried and weighed. A small (~100 gram) sample of the tails was split from the total for later analysis, along with the concentrate. The remainder of the tails was then slurried with water and fed back to the concentrator for the next cycle. Knelson reported a typical, well-behaved separation with the concentrator.

All samples were sent to PNNL for analysis. At PNNL, the samples were split with a riffle-splitter to provide ~10-gram subsamples. In the laboratory, 0.2- to 0.4-gram aliquots were digested in trace metal grade acid (10 ml conc. nitric and 5 ml conc. hydrofluoric), twice for 30 min at 200°C. Next, 35 ml of 4% boric acid was added, and the solution was digested for an additional 30 min at 165°C. The digestions were performed in a CEM microwave extraction system using temperature and pressure control. The solution was then diluted to mark in a 100-ml volumetric flask.

Figure 4.1. Knelson Laboratory-scale Batch Concentrator (KC-MD3) with a 0-100 lb/hr Feed Capacity
The solutions were analyzed in a Perkin Elmer 3000DV ICP-OES. Calibration was made using National Institute of Standards and Technology (NIST)-certified standards. A second set of NIST-certified standards from a different vendor was used to verify the calibration to ±10%.

The results from the first series of tests conducted at Knelson are summarized in Table 4.1. Table 4.1 includes results from concentrates and tails from each pass through the concentrator. Typically, a tails

Table 4.1. Results from Lab Concentrator Test (5 Passes) with K Basin Segregation Simulant

<table>
<thead>
<tr>
<th>Sample #</th>
<th>Name</th>
<th>Description</th>
<th>Sample Fractions</th>
<th>Tungsten</th>
<th>Cobalt</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Reported Mass, g</td>
<td>Wt% in Sample</td>
<td>Mass in Fraction, g</td>
</tr>
<tr>
<td>9406</td>
<td>Ai Concentrate</td>
<td>First Pass Concentrate</td>
<td>220.0</td>
<td>5.60</td>
<td>36.46</td>
</tr>
<tr>
<td>9407</td>
<td>Ai MD3 Tails</td>
<td>First Pass Tails subsample</td>
<td>98.1</td>
<td>2.50</td>
<td>7.08</td>
</tr>
<tr>
<td>9408</td>
<td>Ai Concentrate</td>
<td>Second Pass Concentrate</td>
<td>182.0</td>
<td>4.63</td>
<td>13.14</td>
</tr>
<tr>
<td>9409</td>
<td>Ai MD3 Tails</td>
<td>Second Pass Tails subsample</td>
<td>109.7</td>
<td>2.79</td>
<td>0.88</td>
</tr>
<tr>
<td>9410</td>
<td>Ai Concentrate</td>
<td>Third Pass Concentrate</td>
<td>150.2</td>
<td>3.83</td>
<td>8.97</td>
</tr>
<tr>
<td>9411</td>
<td>Ai MD3 Tails</td>
<td>Third Pass Tails subsample</td>
<td>100.5</td>
<td>2.56</td>
<td>0.84</td>
</tr>
<tr>
<td>9412</td>
<td>Av Concentrate</td>
<td>Fourth Pass Concentrate</td>
<td>156.0</td>
<td>3.97</td>
<td>2.64</td>
</tr>
<tr>
<td>9413</td>
<td>Av MD3 Tails</td>
<td>Fourth Pass Tails subsample</td>
<td>112.1</td>
<td>2.85</td>
<td>0.24</td>
</tr>
<tr>
<td>9414</td>
<td>Av Concentrate</td>
<td>Fifth Pass Concentrate</td>
<td>150.9</td>
<td>3.84</td>
<td>0.11</td>
</tr>
<tr>
<td>9415</td>
<td>Av MD3 Tails</td>
<td>Fifth Pass Tails subsample</td>
<td>117.2</td>
<td>2.98</td>
<td>0.16</td>
</tr>
<tr>
<td>9416</td>
<td>Av MD3 Tails Bulk</td>
<td>Fifth Pass Tails bulk final tails</td>
<td>2530.0</td>
<td>64.43</td>
<td>0.16</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Total</td>
<td>3926.70</td>
<td>100.00</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Wt% in all fractions</td>
<td>3.44</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Sample #</th>
<th>Description</th>
<th>Iron</th>
<th>Chromium</th>
<th>Nickel</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Wt% in Sample</td>
<td>Mass in Fraction, g</td>
<td>Wt% of Total</td>
</tr>
<tr>
<td>9406</td>
<td>First Pass Concentrate</td>
<td>28.28</td>
<td>62.22</td>
<td>6.25</td>
</tr>
<tr>
<td>9407</td>
<td>First Pass Tails subsample</td>
<td>21.61</td>
<td>21.20</td>
<td>2.13</td>
</tr>
<tr>
<td>9408</td>
<td>Second Pass Concentrate</td>
<td>31.56</td>
<td>57.44</td>
<td>5.77</td>
</tr>
<tr>
<td>9409</td>
<td>Second Pass Tails subsample</td>
<td>23.09</td>
<td>25.33</td>
<td>2.54</td>
</tr>
<tr>
<td>9410</td>
<td>Third Pass Concentrate</td>
<td>31.99</td>
<td>48.05</td>
<td>4.83</td>
</tr>
<tr>
<td>9411</td>
<td>Third Pass Tails subsample</td>
<td>26.22</td>
<td>26.35</td>
<td>2.65</td>
</tr>
<tr>
<td>9412</td>
<td>Fourth Pass Concentrate</td>
<td>26.21</td>
<td>40.89</td>
<td>4.11</td>
</tr>
<tr>
<td>9413</td>
<td>Fourth Pass Tails subsample</td>
<td>24.04</td>
<td>26.95</td>
<td>2.71</td>
</tr>
<tr>
<td>9414</td>
<td>Fifth Pass Concentrate</td>
<td>38.09</td>
<td>57.48</td>
<td>5.77</td>
</tr>
<tr>
<td>9415</td>
<td>Fifth Pass Tails subsample</td>
<td>23.79</td>
<td>27.88</td>
<td>2.80</td>
</tr>
<tr>
<td>9416</td>
<td>Fifth Pass Tails bulk final tails</td>
<td>23.79</td>
<td>601.90</td>
<td>60.45</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Total</td>
<td>995.89</td>
<td>100.00</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Wt% in all fractions</td>
<td>25.36</td>
<td>2.14</td>
</tr>
</tbody>
</table>
sample is not collected until the last pass, as the intermediate tails samples contain some of the component of interest (W/Co) that would otherwise be recovered in the concentrate in subsequent passes. However, in this test series, intermediate tails samples were collected to provide additional data to better understand the concentrator performance. Almost 60% of the W/Co was collected in the 1st pass, which constituted about 5.6 wt% of the total test sample mass. About 28% of the W/Co was recovered in the 2nd and 3rd pass concentrates. In the 4th pass, only about 4% of the W/Co was recovered. Table 4.1 shows that very little additional W/Co was collected in the concentrate in the 5th pass.

Figure 4.2 shows the recovery of the simulant components as a function of concentrate mass (yield). To construct Figure 4.2, the concentrations of analytes in all intermediate tails samples (samples 9407, 9409, 9411, and 9413) were assumed to be at the concentrations of the final tails sample (9415). The concentrate collected in the first four passes constituted about 18 wt% of the test sample mass. This concentrate contained about 96% of the W/Co originally present in the feed and only about 25% of the stainless steel powder. Based on the concentration of W/Co in the feed material (3.2 wt%), the W/Co

![Recovery Yield Curve](image)

**Figure 4.2.** Recovery-Yield Curve from Lab Concentrator Test (5 Passes) with K Basin Segregation Simulant. Recoveries (%) of tungsten (W), cobalt (Co), chromium (Cr), iron (Fe), and nickel (Ni) as a function of percent mass recovered to concentrate stream (yield, %).
concentration in the tails stream (i.e., depleted stream that constituted 82 wt% of the test sample mass) is 0.17 wt% (by calculation). By assay, the tails stream from the 5th pass was found to contain ~0.18 wt% W/Co, in excellent agreement with the calculated value. Overall, a mass balance over-recovery for tungsten was measured for this test (i.e., 24% more tungsten was measured by assay in the concentrate plus tails than was present in the feed. A similar tungsten over-recovery was observed for one of the calibration check standards processed with the samples.

From Figure 4.2, about 93% of the tungsten (and 95% of the cobalt) was recovered in the concentrate from the first three passes (which make up 14 wt% of the sample mass).

To estimate the volume of the concentrates and final tails, the bulk dry densities of selected samples were measured. The bulk densities of the concentrates from the first four passes (samples 9406, 9408, 9410 and 9412) are 3.5, 3.0, 2.6, and 2.2 g/cm³, respectively. The bulk density of the final tails sample (9415) is 1.9 g/cm³. Based on these bulk densities, the first three concentrates constitute 9.3 vol% of the total sample volume, and the first four concentrates constitute 13 vol% of the total sample volume. Likewise, the W/Co-depleted stream (tails) from this testing constitutes 87 to 91 vol% of the total sample.

### 4.2 Pilot-Scale Testing

The results from the laboratory testing demonstrated the technical feasibility of using the Knelson centrifugal concentrator for processing K Basin sludge simulants. Therefore, pilot-scale testing with a continuous centrifugal concentrator was performed to obtain data to better predict the performance of a full-scale system. A portion of this testing was observed by Roger Nendick, Vice President, Operations, Fluor Corporation Minerals & Mining, who provided information and photographs used in this discussion.

The pilot-scale testing was conducted using the same sludge simulants as were used for the laboratory-scale tests. Approximately 220 kg of simulants was shipped to Knelson for this testing. Figure 4.3 shows the continuous centrifugal concentrator unit (CVD-6).

In Figure 4.4, the concentrator is seen in the lower part of the “right-hand” tower, below the feed tank at the top. The tailings from the tests are pumped to the tank on top of the “left-hand” tower. Figure 4.5 shows a closer view of the concentrator with the tailings pump in the foreground.

After some initial system testing with the full simulant, Knelson found it necessary to prescreen the simulant to remove oversize particles that could plug the feed line to the concentrator and potentially plug valves within the concentrator. The necessity of the prescreening was primarily due to a limitation of the feed system for the pilot-scale unit rather than a limitation of the technology. Initially the feed simulant was screened at 12 mesh (1680 µm), and processing was attempted. However, due to the specific gravity and particle size of some of the feed constituents, the pilot unit agitation and feed system was not able to effectively supply a homogeneous feed. Specifically, the higher density and coarser particles preferentially settled to the bottom of the feed tank. These settled materials would subsequently plug the feed line. Testing was discontinued, and the feed sample was re-screened at 16 mesh (1180 µm) prior to reloading into the feed system. This minus 16-mesh feed was then used for testing. The prescreening steps reduced the simulant feed mass from ~220 kg to 160.5 kg. However, based on characterization of the simulant components (Section 2.0), 99.71 wt% of the W/Co will pass through a 1180-µm sieve. Therefore, very little of the W/Co was removed by prescreening.
Figure 4.3. Knelson Pilot-Scale Continuous Centrifugal Concentrator (CVD-6)

Figure 4.4. Knelson Pilot-Scale Test Stand
For the testing, prescreened simulant was processed through the CVD-6 twice (two passes). The tails from the 1\textsuperscript{st} pass were used as feed for the 2\textsuperscript{nd} pass. The feed was processed as a \( \sim 30 \) wt\% (solids) slurry at a rate of about 280 kg (solids) per hour. The CVD-6 operating parameters were set as follows: a fluidization water flow rate of 8 gpm, cone RPM corresponding to an acceleration of 60 G; pinch valve settings were 0.04 sec open for both passes and 10 sec closed and 20 sec closed, respectively, for the 1\textsuperscript{st} pass and the 2\textsuperscript{nd} pass.

Figure 4.6 shows a close-up view of the concentrator bowl during operation. Feed is introduced down the center pipe, and the heavy material is held against the rotating bowl by centrifugal force. The lighter tailings material can be seen rising up the bowl and overflowing the top of the bowl.

Figure 4.7 shows the bucket collecting the concentrate (center), with the tailings pump sump in the top left. Note the color difference between the tailings (yellow) and the concentrate (gray), indicating essentially all the yellowish iron oxide monohydrate was separated from the tungsten-rich concentrate.

For the 1\textsuperscript{st} pass, the simulant was processed through the CVD-6 with the entire concentrate generated being drawn off and collected as a single sample (1\textsuperscript{st} pass concentrate). The recovered concentrate sample was dried, weighed, and packaged. For the 2\textsuperscript{nd} pass, the tailings were reprocessed through CVD-6 to scavenge any remaining recoverable W/Co from the tails. A final tails sample was collected from the tanks, dried, and packaged. All samples were returned to the PNNL for assaying.

Figure 4.8 shows the partially dismantled concentrator. The concentrate collection riffle can be seen at the midpoint of the bowl wall and the eight concentrate release valves around the periphery. Figure 4.9 shows a closer view of the concentrate collection rings during clean-up at the end of the test. Some W/Co particles can be seen in the rings.
Figure 4.6. Center Pipe and Bowl of CVD-6 Pilot-Scale Concentrator During Operation with K Basin Simulant. Note the yellow lighter fraction rising up the center.

Figure 4.7. Tungsten-rich Concentrate Collected from CVD-6 Pilot-scale Test
Figure 4.8. Partially Dismantled Concentrator Showing the Concentrate Collection Riffle in the Center of the Bowl Wall Surrounded by Eight Concentrate Release Valves (Pinch Valves)

Figure 4.9. Closer View of Concentrate Collection Riffle During Clean-up Showing Some of the Separated W/Co Particles
4.2.1 Pilot-Scale Results

Samples from the pilot-scale test were split (using a riffle splitter) into analytical subsamples at PNNL. For each major sample (e.g., 1st pass concentrate), three analytical samples were prepared and analyzed via the procedure described in Section 4.1. All analytical results are provided in Appendix B.

The test results from the pilot-scale test (and the laboratory-scale test) are summarized in Table 4.2. The material flow and material balance are shown in Figure 4.10. Because only a very small fraction of the tungsten present in the original simulant was removed during screening, the analysis of the pilot-scale run provided in Table 4.2 is based on the simulant feed composition after screening at 1180 µm.

### Table 4.2. Summary of Knelson Centrifugal Concentrator Test Results

<table>
<thead>
<tr>
<th>Test</th>
<th>Concentrate (enriched stream)</th>
<th>Tails (depleted stream)</th>
<th>Percent Tungsten (W) Recovered to Concentrate (based on analyses)</th>
<th>Overall Analytical Mass Balance Check(b)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Wt% of Feed [W] Wt%</td>
<td>Wt% of Feed [W] Wt%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Laboratory-scale concentrator test conducted with simulant feed containing ~2.78 wt% tungsten</td>
<td>20.2(c) 17.8</td>
<td>79.8 0.16</td>
<td>96.5</td>
<td>124</td>
</tr>
<tr>
<td>Pilot-scale concentrator test conducted with simulant feed containing ~3.79 wt% tungsten(d)</td>
<td>17.9(e) 18.7</td>
<td>82.1 0.07</td>
<td>98.4</td>
<td>90</td>
</tr>
</tbody>
</table>

(a) Feed stream was segregated into tungsten-enriched stream (concentrate) and tungsten-depleted stream (tails). Stream split (mass percent) and tungsten concentration (by assay) in each stream are provided. Check on measured (assayed) W in product streams vs. quantity used to prepare test material. For the pilot-scale test, values are based on the feed after sieving; see footnote (d).

(b) Value = 100% × [mass of W in concentrate (by assay) + mass of W in tails (by assay)] divided by mass of W in feed (mass was measured).

(c) Combined concentrate from four sequential passes through concentrator (e.g., tails from 1st pass used as feed for 2nd pass).

(d) Original feed exhibited a particle size distribution that ranged from submicron to ~4000 µm and contained ~2.78 wt% tungsten. To address particle size constraint associated with the pilot-scale test system, the feed was sieved to remove all particles greater than 1180 µm, resulting in a feed that contained ~3.79 wt% tungsten.

(e) Combined concentrate from two sequential passes through concentrator (e.g., tails from 1st pass used as feed for 2nd pass).

(f) Concentrate collected from 1st pass.

(g) Concentrate collected from 2nd pass.
Mass 219 Kg  
W* 6.1 Kg  
W* 2.78 %  

W* = determine from feed make up  
W** = determined by chemical assay  

Mass 160.5 Kg  
W* 6.08 Kg  
W* 3.79 %  

Mass 142.0 Kg  
W** 1.16 Kg  
W** 0.8 %  

Balance  
W 0.62 Kg [W in 1st Pass Feed - (W in Concentrate + W in Tails)]

**Figure 4.10.** Flowsheet for Pilot-Scale Testing of Knelson Continuous Centrifugal Concentrator

The tungsten concentration and mass values shown in Figure 4.10 for the Feed Tank, Oversize Material, and 1st Pass Feed to the CVD-6 were calculated based on the simulant make-up and characterization. However, for the 1st and 2nd Pass Concentrate and CVD-6 Tails, the tungsten concentration and mass values shown in Figure 4.10 are measured values (from chemical analysis of samples). About 90% of the calculated tungsten mass in the feed stream was found (measured) in the product streams (concentrate and tails) (i.e., 90% material balance closure).

The 1st pass concentrate contained 90.2% of the tungsten recovered. The high recovery in the 1st pass demonstrates that the uranium metal surrogate (W/Co) is readily recovered by the Knelson concentrator. A further increase in recovery of 8.2% was realized in the 2nd pass. Overall, when combining the concentrates from the 1st and 2nd passes, a total of 98.4% of the tungsten was recovered into 17.9% of the feed mass (10.5% of sample volume).

The final tailing grade was found to be low at 0.07% W on the one sample that was within detection limits.
4.2.2 Overall Assessment of Amenability of K Basin Sludge to Knelson Gravity Concentration Equipment

It was apparent from the test observations and analytical results that the Knelson gravity concentrator technology can achieve a high W/Co removal efficiency and produce a concentrate stream with a high W/Co content. The installation and operation of the concentrator for K Basin sludge processing may be challenged in:

- coarse particle processing
- underwater operation.

Coarse Particle Processing. It is anticipated that the K Basin sludge will contain coarse uranium metal particles, as well as other coarse debris. In order for the Knelson concentrator to operate efficiently, this material may need to be removed, since the large particles can block the concentrate release valves (pinch valves) shown in Figure 4.11(top).

The concentrate release valves are pneumatically actuated rubber pinch valves. Figure 4.11(bottom) shows the rubber valve sleeves. The valve orifice is approximately 8 mm (8000 µm) in diameter. Assuming that three particles could bridge the opening, a particle diameter of around 2.7 mm (2700 µm) could be expected to cause operational problems. However, when Knelson tried screening the feed simulant at 12 mesh (1680 µm), the operation of the concentrator was unreliable. The screening was repeated at 16 mesh (1180 µm), and the concentrator operated efficiently. The fact that the concentrator feed needed to be screened to a finer product than was anticipated is possibly due to the particles being thrown against the closed valve at a high centrifugal force, allowing them to compact and bridge more readily. As a consequence, a large amount (26.7 wt% of the original feed) of the K Basin sludge simulant could not be fed to the concentrator.

Prescreening of the feed material, which presumably may also have to be conducted in an underwater environment, creates an additional, potentially challenging, processing step.

The option to backflush the valves with water or air was briefly considered and discussed with Knelson, but this would disrupt the concentrator process and would be complex mechanically.

Underwater Operations. The issue of operating the Knelson concentrator underwater was considered. The mechanical and electrical issues can be resolved. It is believed that underwater operation will not have a significant impact on the gravity concentration action. The higher backpressure from the flooded bowl can be compensated by increasing the back-pressure of the fluidization water.
Figure 4.11. Rubber Concentrate Release (Pinch) Valves Used in the Concentrator
5.0 Deployment Considerations

Results from vendor testing established the technical feasibility of using commercial gravity concentration equipment for the removal and concentration of the uranium metal in K Basin sludge. During the course of this investigation, some technical challenges were identified, as discussed below.

**Determination/specification of the uranium metal removal requirement.** The removal requirement for a specific stream depends on a number of factors:

- uranium metal concentration and particle size distribution in the stream
- hydrogen gas generation rate limit in the final package
- sludge loading in the final package (e.g., $^{239}$Pu fissile gram equivalents)
- reaction rate mitigation achieved by the solidification matrix
- pyrophoricity limit (1 wt%)
- aging of the package (to diminish uranium metal concentration and particle size).

Preliminary calculations show that if 96.6% of the initial uranium metal is removed from the KE floor and KE canister sludge to 15% of the original sludge volume, and the resulting sludge tails stream is dried or solidified in a matrix that reduces the rate of the uranium metal-water reaction by a factor of 4, then waste loading for packaging the sludge for disposition will be constrained by the WIPP FGE limit rather than by the hydrogen gas generation rate. Uranium surrogate removal efficiencies of this order were observed for both vendors’ tests. However, further development and refinement of the hydrogen generation rate limit and the uranium metal removal requirements are recommended in order to reflect the actual range of sludge streams to be encountered in processing; to develop practical means to assess uranium metal concentrations, particularly reactive surface areas; and to provide reliable bounds to guarantee that WIPP limits are not exceeded.

**Measurement of uranium metal concentration.** To verify the effectiveness of the segregation process, the uranium metal concentration in the concentrate and tails streams may need to be measured. Robust and rapid measurement techniques for determining the uranium metal content in the sludge matrix have not been identified. A process test may be necessary to qualify the segregation process, with intermittent verification testing (e.g., gas generation or fission product release tests) to confirm the process performance. Process gas generation or fission product gas release testing in a large reaction vessel, in which the sludge would be held at an elevated temperature (e.g., 60 to 95°C) for a period of time, could provide an estimate of the uranium metal reactive surface area remaining after segregation processing.

**Location/shielding.** The commercial gravity concentration equipment is not typically deployed in radiological or underwater environments. However, the equipment is designed for rugged, low-maintenance applications, and underwater deployment appears technically feasible. The equipment design likely would be refined by the vendor for the types of operations required for uranium metal segregation.

**Additional unit operations.** For the Knelson continuous centrifugal concentrator, a grinding or prescreening operation likely would be needed to reduce the maximum particle size from 6350 to 2500 µm. On average, only about 5 wt% of the KE floor and canister sludge is composed of particles greater than 2500 µm. It is likely that many of these larger particles are aluminum corrosion product
nodules, and a relatively simple, low-energy grinding process (e.g., ball mill) could be used to achieve the required size reduction. However, irradiated uranium metal fragments have been found to be remarkably tough in crushing and sieving tests (Schmidt et al. 2003). A size reduction or prescreening step will not be necessary with the Gekko equipment.

**Handling/storage of uranium metal concentrate stream.** The uranium metal particles collected in the concentrate stream will require vessels and systems designed to accommodate the potential high gas and heat generation rates. Components and design features of the concentrate storage and handling systems may therefore be designated as safety significant. It is worthwhile to note that, based on the vendor segregation testing, the concentrate streams arising without recycle likely will contain less than 30 wt% uranium metal. In comparison, the Knockout Pots have been analyzed and designed to contain sludge consisting of essentially 100% uranium metal as 500-µm particles. Consequently, it is expected that thermal stability and criticality analyses of handling and containment systems for the uranium segregation concentrate stream can build on existing KOP design and safety analyses.

**Treatment/Final Disposition of the KOP and Concentrate Streams.** The segregation of sludge will create a uranium metal-depleted tails stream that can be processed and loaded for disposal in 55-gal containers, potentially to the 200-gram $^{239}$Pu FGE WIPP disposal limit. However, the remaining uranium metal-rich concentrate, as well as the KOP stream, will contain uranium metal concentrations ranging from 20 wt% to, potentially, 100 wt% on a dry basis. Options for the concentrate and KOP sludge streams include further upgrading and subsequent processing through cold vacuum drying or conditioning (e.g., oxidation of the uranium metal) to meet WIPP waste acceptance criteria.

**Water Balance.** Gravity concentration processing will require input water and may generate a dilute tails stream that will need to be thickened/settled. Options that facilitate the recycle of water within the circuit should be considered.

**Other Segregation Options.** Other options for uranium metal segregation include hydrocyclones and elutriation columns. For those options, several units, operating in series, may be required. These units would likely generate a significant overflow stream, requiring thickening/settling. Because of their relative simplicity, further evaluation of hydrocyclones and elutriation columns may be warranted.
6.0 References


6.1


Appendix A
Gekko Systems Test Report
Pacific Northwest National Laboratories

GRAVITY CONCENTRATION AND UPGRADING OF URANIUM SURROGATE SAMPLE

Authors:
Jennifer Abols
Damon Buchanan

DATE:
July 16, 2004

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Phone: 604-681-2288
Fax: 604-681-2220
Cell: 778-772-4052

Email: JenniferA@gekkos.ca
www.gekkos.com
SUMMARY

AIMS

- Investigate the amenability of the uranium surrogate sample to gravity recovery using the InLine Pressure Jig.
- Investigate the amenability of the recovered concentrate to upgrading using an InLine Spinner.

TEST PROGRAM

- The uranium surrogate sample was received, split and assayed. Approximately 20 kg of sample was tabled to produce concentrate and tailings samples over a range of mass pulls and to determine the potential for single pass recovery (Tungsten/Cobalt). The recovered table fractions were sieved at 500 µm and assayed for Tungsten, Cobalt, Iron, Nickel & Chrome to produce a recovery-yield curve for each element.
- Using the results from the first test a second sample was tabled to pull the required mass to concentrate to achieve 85-95% recovery of uranium surrogate (Tungsten/Cobalt). The table fractions were sieved at 500 µm and assayed for Tungsten, Cobalt, Iron, Nickel & Chrome to produce a recovery-yield curve for each element.
- The concentrate sample from the second test was fed to an InLine Spinner to determine whether the concentrate could be upgraded effectively. The recovered concentrate and tails fractions were sieved at 500 µm and assayed for Tungsten, Cobalt, Iron, Nickel & Chrome to produce a recovery-yield curve for each element.
- The tabling and spinner testwork protocols were repeated using a second sample to verify the results of the first set of tests.
- In total, three tabling tests and two spinner tests were performed.

OUTCOMES

- Primary gravity concentration was effective in producing a concentrate containing up to 99% of the tungsten and 96% of the cobalt into 32.3% of the mass.
- The Spinner was able to produce an upgraded concentrate containing 91% of the Tungsten and 81% of the cobalt (based on the amount of these metals in the primary concentrate) into 33.1% of the mass. This translates into an overall process recovery of 90% of the tungsten and 78.4% of the cobalt into 10% of the overall mass.
- When the mass pull on the Spinner was increased to 52.8% an upgraded concentrate containing 99% of the tungsten and 98% of the cobalt was produced. This translates into an overall process recovery of 97% tungsten and 94% cobalt into 17% of the overall mass.
IMPLICATIONS

- The uranium surrogate sample tested is highly amenable to gravity recovery using the InLine Pressure Jig.
- The primary gravity concentrate recovered is highly amenable to upgrading using an InLine Spinner.

RECOMMENDATIONS

- A two stage recovery system, consisting of a primary InLine Pressure Jig and InLine Spinner is recommended for the treatment of Pacific Northwest National Laboratories uranium sludge.

RESULTS

IPJ AMENABILITY

Gekko Systems received approximately 60kg of Uranium Surrogate sample. The sample was riffle split and assayed for Tungsten, Cobalt, Iron, Nickel & Chrome.

A tabling procedure was developed to enable a single pass grade recovery curve to be produced that would give a wide range of yield/recovery combinations.

The results of the tabling testwork are provided on the following pages. The resulting yield recovery and yield grade curves are also provided.

In the first tabling test recovery of tungsten and cobalt into the concentrate was 97.9% and 94.4%, respectively into 30.6% of the mass at a grade of 8.5% tungsten and 7107.4 ppm cobalt. Recoveries of chromium, iron and nickel into the concentrate were 19.6%, 25.2% and 24.3% respectively.

In the second table test the recovery of tungsten and cobalt into the concentrate was 97% and 94%, respectively into 31.5% of the mass. Recoveries of chromium, iron and nickel into the concentrate were much higher at 36%, 36% and 41% respectively. The higher values for chromium, iron and nickel have been attributed to the way the concentrate was split out from the sample. In the first pass an insufficient amount of concentrate was recovered and so the tails were re-tabled a second time.

In the third table test the recovery of tungsten and cobalt into the concentrate was 99% and 96%, respectively into 32.2% of the mass. Recoveries of chromium, iron and nickel into the concentrate were 27%, 31% and 34% respectively.

ISP AMENABILITY

The concentrate recovered from the second table test was fed to a Gekko InLine Spinner ISP02 set up with a feed rate of 1.86 kg/5 min and a dump cycle of 5 minutes. A total of 3.72 kg were fed to the spinner. The results from the spinner testwork are provided on the following pages as well as the resulting yield recovery and yield grade curves. The spinner recovered 99% of the tungsten and 98% of the cobalt in the primary concentrate into 52.8% of the mass.

The concentrate recovered from the third table test was fed to an ISP02 set up with a feed rate of 1.86 kg/6.5 min and a dump cycle of 6.5 minutes. A total of 3.71 kg were fed to the spinner. The results from this test are provided on the following pages as well as the resulting yield recovery and yield grade curves. The spinner recovered 91% of the tungsten and 81% of the cobalt in the primary concentrate into 33.1% of the mass.
IMPLICATIONS

The InLine Pressure Jig and InLine Spinner were very effective in concentrating and cleaning the Uranium Surrogate Sample. The results indicate that a combination of these devices should result in an overall recovery of no less than 90% of the Uranium in the Basin Sludge into 10-17% of the mass. If the jig and/or spinner tails are recycled the recoveries will likely be increased.

COMMENTS

• Size reduction of the feed material is not necessary.
• Prescreening of the feed material is not necessary.
• A recommended preliminary flowsheet is attached. The number of unit processes should be kept to a minimum to reduce the amount of maintenance required. The power consumption for the feed and storage tanks will be quite high as a great deal of agitation will be required to keep the uranium particles suspended. The flowsheet as shown can be batch fed. The circuit can be run with the tails from the jig and spinner in recirculation mode until no more product reports to final concentrate.
• The IPJ is best suited to direct pumped feed at medium to high density say >30% <80% solids by weight. The spinner can accept a fairly dilute feed, from 5% to 20% solids by weight. It would be a good idea to put in a small thickener at the front end of the circuit. This thickener, which could be a small conical settling cone with a rake in it, would produce a thickened feed suitable for feeding the IPJ and an overflow suitable for use as IPJ hutch water or spinner wash water. This would reduce the amount of fresh water needed in the circuit.
• Based on the testwork results, the IPJ/spinner combination has the potential to recover up to 98% of the uranium surrogate in the feed (based on the tungsten assays). The spinner mass pulls achieved in the uranium surrogate testwork are the highest we have ever seen. It is highly unlikely that higher single pass mass pulls would be achievable in the field. By recycling the jig and spinner tails, as shown in the flowsheet uranium metal recovery and mass pull to concentrate will be maximized. Replacing the spinner with a second jig would result in a higher overall mass pull but a much lower grade concentrate. Given the very high recoveries achieved in the testwork the jig/spinner combination is very well suited to and recommended for this application.
• The spinner is very well ventilated and the jig comes equipped with an air release valve so we do not expect any gas accumulation in either device.
• When compared with a hydrocyclone, the jig/spinner combination recommended for this project should result in a much cleaner tail and higher grade/smaller mass concentrate. This should aid in final disposal of the product streams. These devices separate particles based on density, while the hydrocyclone separates via particle size and density. Hydrocyclones require a large amount of water to achieve effective separation.
## Results

### Tabling Test #1

**Pacific Northwest Uranium**

19/04/2004  

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### Pacific Northwest Uranium

**Single Pass Tabling Combined Grades**

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**Nickel Distribution on Table**

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### Pacific Northwest Uranium Table 3

**05/07/2004**

**Single Pass Tabling on as received for 30% Concentrate**

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### Assay Feed

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Tabling Recovery Yield Curve

Tabling Grade Yield Curve
## Spinner Upgrade Test #1

### Pacific North West Uranium

29/04/2004

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### Spinner Upgrade Test #2

#### Pacific North West Uranium

**05/07/2004**

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<tr>
<td></td>
<td>g</td>
<td>%</td>
<td>cumulative ppm</td>
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<tr>
<td><strong>Spinner Concentrate</strong></td>
<td>1303.4</td>
<td>33.1%</td>
<td>33.1%</td>
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<tr>
<td><strong>Spinner Tails</strong></td>
<td>2635.9</td>
<td>66.9%</td>
<td>100.0%</td>
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<tr>
<td><strong>Calc’d Feed</strong></td>
<td>3939.3</td>
<td>100.0%</td>
<td></td>
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<tr>
<td><strong>Assay Feed</strong></td>
<td>3939.3</td>
<td>100.0%</td>
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<th>Chromium Distribution</th>
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<td>Assay ppm</td>
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</tr>
<tr>
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</tr>
<tr>
<td>30937</td>
</tr>
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<td>30460</td>
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<td>59337</td>
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<td>Assay ppm</td>
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<table>
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<tr>
<td>Assay ppm</td>
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</table>

A.12
Gekko Systems Pty Ltd - Disclaimer

Gekko has undertaken test work to characterize the response of your ore to certain separation techniques and/or to help your own experts make a decision as to whether you wish to purchase our product and, if so, the number and type.

It is important that you understand that:

- Our testing is preliminary only.
- You should obtain, independent advice from all relevant specialists, including a metallurgist, before acquiring any equipment and before committing to and proceeding with your project.
- You must have your own experts examine the detailed analysis in our report to decide its applicability to your project.
- We analyse only the sample you provide. Any one of a number of factors may cause that sample inaccurately to reflect the ore body. You must determine the extent to which the sample represents the ore body. That includes the detection limits and confidence intervals relevant to our results.

At all times we endeavour to provide accurate testwork outcomes but you should not use our results as a basis for your broader business decisions about your project.

If we have not exercised due care with our tests, the limit of our liability, both at common law and under any statute, will be to provide a further set of test results to you free of charge.

You indemnify us with respect to all other loss and damage of every kind, including, without limitation:

- damage to or loss of property;
- injury to or death of any person; and
- economic and consequential loss

arising from the negligent act or omission of us or any one else in connection with our tests.
Appendix B

Knelson Concentrators Test Reports
(Laboratory Scale; Pilot Scale)
METALLURGICAL TEST REPORT

PROJECT: Battelle
PROJECT NO: KRTS 20080

Report Prepared For: Mr. Monte Elmore

Report Prepared By:

Ishwinder Grewal, M.A.Sc., P.Eng.
Manager, Knelson Research & Technology Centre

AJ Gunson, M.A.Sc.
Metallurgist

April 29, 2004
Mr. Monte Elmore, of Battelle - Pacific Northwest National Laboratory, contacted Knelson to discuss the potential of using gravity concentration technology to remove uranium particles from sludge material. The underlying concept for the test work was to simulate the sludge with an artificial composite using tungsten carbide (WC) as a substitute for uranium.

Based on the objectives described by Mr. Elmore, Knelson Concentrators recommended that a multi-stage gravity concentration test be performed using a laboratory-scale centrifugal concentrator (KC-MD3) to determine if gravity concentration would be effective. A positive result would provide confidence for proceeding with further test work at a pilot-scale using Knelson’s continuous centrifugal concentrator, the CVD (continuous variable discharge).

PROCEDURE

The general procedure for the test program is provided below. A flow chart of the test program is attached in appendix A.

i. The 3" Laboratory Knelson Concentrator (KC-MD3) operating parameters were set as follows; a fluidization water flow rate of ~3.5 lpm and a cone RPM corresponding to a force of 60 g’s.
ii. A ~4kg of composite sample was slowly processed through the KC-MD3.
iii. After the test, the tailings and concentrate samples were recovered and dried.
iv. A sub-sample of the tailings was taken for assay purposes.
v. The tailings were then reprocessed through KC-MD3 as per steps i-iv above.
vi. The above procedure was performed for a total of five stages.
vii. All samples were returned to the client for assaying.
The mass-balance of the results from the test work is presented below.

### Table 1  Mass Balance from MD-3 Pilot Test

<table>
<thead>
<tr>
<th>Product</th>
<th>Mass (g)</th>
<th>Assay (%)</th>
<th>Assay W (%)</th>
<th>Assay W (g)</th>
<th>Dist’n (%)</th>
<th>Cum Rec. (%)</th>
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</thead>
<tbody>
<tr>
<td>Stage 1 Conc.</td>
<td>220.0</td>
<td>5.47</td>
<td>36.46</td>
<td>80.2</td>
<td>59.3</td>
<td>59.3</td>
</tr>
<tr>
<td>Stage 1 MD3 Tails</td>
<td>98.1</td>
<td>2.44</td>
<td>7.08*</td>
<td>6.9</td>
<td>5.1</td>
<td></td>
</tr>
<tr>
<td>Stage 2 Conc..</td>
<td>182.0</td>
<td>4.52</td>
<td>13.14</td>
<td>23.9</td>
<td>17.7</td>
<td>76.9</td>
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<tr>
<td>Stage 2 MD3 Tails</td>
<td>109.7</td>
<td>2.73</td>
<td>0.88</td>
<td>1.0</td>
<td>0.7</td>
<td></td>
</tr>
<tr>
<td>Stage 3 Conc.</td>
<td>150.2</td>
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<td>8.97</td>
<td>13.5</td>
<td>10.0</td>
<td>86.9</td>
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<tr>
<td>Stage 3 MD3 Tails</td>
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<td>2.50</td>
<td>0.84</td>
<td>0.8</td>
<td>0.6</td>
<td></td>
</tr>
<tr>
<td>Stage 4 Conc.</td>
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<td>2.64</td>
<td>4.1</td>
<td>3.0</td>
<td>89.9</td>
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<td>Stage 4 MD3 Tails</td>
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<td>0.3</td>
<td>0.2</td>
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</tr>
<tr>
<td>Stage 5 Conc.</td>
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<td>0.2</td>
<td>0.1</td>
<td>90.1</td>
</tr>
<tr>
<td>Stage 5 Final Tails</td>
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<td>68.20</td>
<td>0.16</td>
<td>4.4</td>
<td>3.3</td>
<td></td>
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<tr>
<td>Totals (Head)</td>
<td>4024</td>
<td>100.0</td>
<td>3.36</td>
<td>135.4</td>
<td>100.0</td>
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<tr>
<td>Knelson Conc.</td>
<td>859.1</td>
<td>21.35</td>
<td>14.2</td>
<td>121.91</td>
<td>90.1</td>
<td></td>
</tr>
</tbody>
</table>

Note: All of the tails grades are assayed values. The Stage 5 Final Tails mass and the Totals (Head) mass are based on the initial sample weight.

*This is an anomalous assay, as the head grade is lower than the intermediate tail grade

First stage recovery at 59.3% indicates that the tungsten carbide is readily recovered in the Knelson Concentrator. Further increases in recovery are realized over the remaining 4 stages. About 96.5% of the gravity recoverable tungsten is removed within the first 3 stages, at a mass yield of 13.7%. The total tungsten recovery from the 5 stages was 90.1%, at a mass pull of 21%.

The overall mass of the final samples was 3926.7g, but the initial mass of 4024g was used for the mass balance calculations. This remaining 100g probably consists of a fine size fraction of the tailings that did not settle during the test work. Intermediate samples are decanted to insure the sample remains reasonable to handle during test work. The difference does not significantly affect the mass balance results.
DISCUSSION

The assay result of Stage 1 MD3 Tails is anomalous, as the grade at 7.08% W is significantly higher than the feed grade, at 3.36% W. This inconsistent grade considerably reduces the overall test recovery. A simple mass balance, based on the Stage 1 Concentrate grade and the Totals (Head) grade, would indicate a grade of 1.45% W, not 7.08% W. Adjusting this grade increases the overall recovery by a minimum of 4%, indicating that the overall recovery was probably closer to 95% than 90%. The Stage 5 Final Tails may also represent an anomalous result.

To further clarify the actually overall performance of the MD-3, the following calculation was undertaken.

Overall Recovery = \(100 - \frac{100 \times (Fm - Cm) \times t}{Fm \times f}\)

\(100 - \frac{100 \times (4024 - 859.1) \times 0.16}{4024 \times 3.36} = 96.3\%\)

Where:   
Fm  =  Feed mass  
Cm  =  Concentrate mass  
t  =  grade of final tails  
f  =  grade of feed material

The above variables are less susceptible to sampling or assay error than much of the data in Table 1.

The MD-3 upgrades dense particles on the surface of the bed in the rings of the cone. This bed is formed during the initial start-up of the MD-3, and consists of feed-grade material, thus much of the concentrate mass is not upgraded material. The continuous centrifugal concentrator, or CVD, does not require this initial feed-grade concentrating bed, and is thus likely to recover most of the tungsten carbide with a significantly lower mass yield.

The final tailing grade was found to be low at 0.16% W. As the Stage 5 concentrate was only 0.11% W, it is possible that there is some assay or sampling error with this result and the actual final concentrate grade could be even lower.

SUMMARY

The preliminary test work undertaken on a synthetic ore provided by Battelle with the MD-3 centrifugal concentrator indicates that sludge material containing uranium would be well suited to gravity recovery using a continuous centrifugal concentrator.

The simple mass balance results show an overall recovery of 90.1% of the tungsten with at 21% mass pull. However, an anomalous assay result significantly lowers the overall recover. Calculating the overall recovery using a method less susceptible to assay or sampling error results in an overall recovery of 96.3%. The final tailings grade was assayed to be 0.16% W and could in fact be lower. The CVD may be able to achieve similar or better recoveries at a lower mass pull.

Further pilot scale test work using the CVD is recommended to confirm and elucidate the mass recovery response of the tungsten carbide from the simulated sludge material.
APPENDICES

Appendix A
Test Program Flow Sheet Diagram

Appendix B
i) Metallurgical Results
ii) Masses and Grades of Products
iii) ICP Results
Appendix A
Test Program Flow Sheet Diagram

All Tail samples were dried, weighed and riffle split to produce a representative MD3 Tail Assay sample.

Feed
4.024 kg

Process thru KC-MD3 (1)

MD3 Ai Concentrate
MD3 Ai Tails Sample

Process thru KC-MD3 (2)

MD3 Aii Concentrate
MD3 Aii Tails Sample

Process thru KC-MD3 (3)

MD3 Aiii Concentrate
MD3 Aiii Tails Sample

Process thru KC-MD3 (4)

MD3 Aiv Concentrate
MD3 Aiv Tails Sample

Process thru KC-MD3 (5)

MD3 Av Concentrate
MD3 Av Tails Sample
MD3 Av Bulk Tails
## Appendix B

### i) Metallurgical Results

<table>
<thead>
<tr>
<th>Product</th>
<th>Mass (g)</th>
<th>Assay W (%)</th>
<th>W (g)</th>
<th>Dist’n (%)</th>
<th>Rec. (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stage 1 Conc.</td>
<td>220.0</td>
<td>5.47</td>
<td>36.46</td>
<td>80.2</td>
<td>59.3</td>
</tr>
<tr>
<td>Stage 1 MD3 Tails*</td>
<td>98.1</td>
<td>2.44</td>
<td>7.08</td>
<td>6.9</td>
<td>5.1</td>
</tr>
<tr>
<td>Stage 2 Conc.</td>
<td>182.0</td>
<td>4.52</td>
<td>13.14</td>
<td>23.9</td>
<td>17.7</td>
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<tr>
<td>Stage 2 MD3 Tails</td>
<td>109.7</td>
<td>2.73</td>
<td>0.88</td>
<td>1.0</td>
<td>0.7</td>
</tr>
<tr>
<td>Stage 3 Conc.</td>
<td>150.2</td>
<td>3.73</td>
<td>8.97</td>
<td>13.5</td>
<td>10.0</td>
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<tr>
<td>Stage 3 MD3 Tails</td>
<td>100.5</td>
<td>2.50</td>
<td>0.84</td>
<td>0.8</td>
<td>0.6</td>
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<tr>
<td>Stage 4 Conc.</td>
<td>156.0</td>
<td>3.88</td>
<td>2.64</td>
<td>4.1</td>
<td>3.0</td>
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<tr>
<td>Stage 4 MD3 Tails</td>
<td>112.1</td>
<td>2.79</td>
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<td>2744.5</td>
<td>68.20</td>
<td>0.16</td>
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<tr>
<td>Knelson Conc.</td>
<td>859.1</td>
<td>21.35</td>
<td>14.2</td>
<td>121.91</td>
<td>90.1</td>
</tr>
</tbody>
</table>

Note: All of the tails grades are assayed values. The Stage 5 Final Tails mass and the Totals (Head) mass are based on the initial sample weight.

*This is an anomalous assay as the head grade is lower than the intermediate tail grade

Overall Recovery (R):

\[
R = \frac{100 - 100*(Fm - Cm)*t}{Fm*f}
\]

\[
= \frac{100 - 100*(4024-859.1)*0.16}{4024*3.36}
\]

\[
= 96.3\%
\]

where

- \(Fm\) = Feed mass
- \(Cm\) = Concentrate mass
- \(t\) = grade of final tails
- \(f\) = grade of feed material
### Appendix B

ii) Masses and Grades of Products

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<th>Sample #</th>
<th>Name</th>
<th>Description</th>
<th>Mass (g)</th>
<th>Assay (% W)</th>
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<td>Fifth Pass Tails bulk final tails</td>
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### Appendix B

#### iii) ICP Results

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<th>Conc (wt%)</th>
<th>Conc (wt%)</th>
<th>Conc (wt%)</th>
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<th>Conc (wt%)</th>
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<th>Conc (wt%)</th>
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B.8
METALLURGICAL TEST REPORT
PROJECT: Battelle - Pacific Northwest National Laboratory
PROJECT NO: KRTS 20098

Report Prepared For: Mr. Monte Elmore

Report Prepared By:
Ishwinder Grewal, M.A.Sc., P. Eng.
Manager, Knelson Research & Technology Centre

Linda Duncan, AScT
Sr. Lab Technologist

June 14, 2004
1.0 Background

Preliminary test results using a lab scale centrifugal concentrator, the Knelson KC-MD3, provided promising results for the recovery and removal of tungsten carbide (WC) from a synthetic sludge sample supplied by Battelle - Pacific Northwest National Laboratory (PNNL). This previous test work indicated the potential of removing in excess of 90% of the target species. These findings were issued in a separate report, Knelson Project KRTS20080, submitted to PNNL in April 2004.

Following the promising results from the lab scale tests, a decision was made to perform test work using the pilot scale continuous centrifugal concentrator (the Knelson CVD-6). In actual operation, the CVD would provide the required mass yield flexibility for effective recovery of the target species.

The underlying concept for the test work was to simulate a potentially radioactive sludge with an artificial composite using WC as a substitute for uranium.

2.0 Procedure

The general procedure for the test program is provided below. A flow chart of the test program is attached in appendix A.

Initially the sample was screened at 12 mesh (1.70mm) and sample processing was tried however, due to the specific gravity and grain size of some of the feed constituents, the pilot plant agitation and feed system was not able to effectively supply the feed to the CVD6. Specifically, it was not possible to effectively suspend the material as homogeneous slurry as the higher density and coarser particles would preferentially settle to the bottom of the feed tank. These settled material would then feed as a high-density product to the CVD-6 from the bottom of the feed tank and plug the feed line. Testing was discontinued and the feed sample was re-screened at 16-mesh (1.18mm) prior to reloading into the feed system. This screened feed was used for testing. Two complete passes through the CVD were accomplished using 73% of the original feed mass.

i. The Knelson CVD operating parameters were set as follows; a fluidization water flow rate of 8gpm, cone RPM corresponding to a force of 60 g’s, pinch valve settings were .04 sec open for both passes and 10 sec closed and 20 sec closed respectively for pass 1 and 2. Air pressure at the pinch valve was set to 34 psi and feed rate to the machine was ~.28 T/hr solids to the machine. Feed density for the test was 30% solids and slurry feed rate was .96 T/hr.

ii. The sample in its entirety was processed through the CVD with the entire concentrate generated during testing being drawn off.

iii. The recovered concentrate sample was dried, weighed and packaged.

iv. The tailings were then reprocessed through KC-CVD6 as per steps i-iii above.

v. A final tails sample was collected from the tanks, dried and packaged.

vi. All samples were returned to the client for assaying.
3.0 Summary of Results and Discussion

The mass-balance of the results from the test work is presented below.

<table>
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<tr>
<th>Product</th>
<th>Mass (g)</th>
<th>Mass (%)</th>
<th>Assay %</th>
<th>Dist (%)</th>
<th>Rec. (%)</th>
<th>Assay %</th>
<th>Dist (%)</th>
<th>Rec. (%)</th>
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<tbody>
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<td>Stage 1 Conc.</td>
<td>18472.0</td>
<td>11.51</td>
<td>26.66</td>
<td>90.2</td>
<td>90.2</td>
<td>2.18</td>
<td>89.0</td>
<td>89.0</td>
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<tr>
<td>Stage 2 Conc.</td>
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<td>6.42</td>
<td>4.33</td>
<td>8.2</td>
<td>98.4</td>
<td>0.37</td>
<td>8.4</td>
<td>97.4</td>
</tr>
<tr>
<td>Final Tails</td>
<td>131730.0</td>
<td>82.07</td>
<td>0.07*</td>
<td>1.6</td>
<td>0.01</td>
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<td>100.0</td>
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<td>Knelson Conc.</td>
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<td>17.93</td>
<td>18.7</td>
<td>98.4</td>
<td>1.53</td>
<td>97.4</td>
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</table>

Note: The Final Tails mass and the Totals (Head) mass are based on the sample weight after screening to 16 mesh (1.18mm)

*Two of the three assay results were below detection limits for W- the result is of the only assay obtained.

First stage recovery at 90.2% indicates that the tungsten carbide is readily recovered in the Knelson Concentrator. A further increase in recovery of 8.2% is realized in the second pass for a total of 98.4% of the tungsten being removed at a mass yield of 17.9%. Based on calculations done by PNNL, and included in the appendices, the mass yield by volume is 6.2% in the first stage and 4.3% in the second stage.

The total sample mass supplied by PNNL was 219 kg, but a mass of 160.5 kg was used for the mass balance calculations, as this was the actual mass used in the test. The remaining 58.5 kg constitutes the coarse size fraction that could not be processed due to the aforementioned feed system limitations.

Although the WC only makes up a total of 3.4% of the feed, the characteristics of the feed are such that a higher mass to the concentrate is required in order to recover sufficient of the target mineral.

As hypothesized in the previous report (Knelson Project KRTS20080), the CVD was able to recover more tungsten carbide in a lower mass yield than the batch machine (the KC-MD3).

The final tailing grade was found to be low at 0.07% W on the one sample that was within detection limits.

Complete ICP results of the products are included in the appendices along with mass balances of the various elements associated with the sample.
4.0 Summary

The preliminary test work undertaken on a synthetic mixture provided by PNNL with the continuous centrifugal concentrator (CVD6) indicates that sludge material containing uranium would be well suited to gravity recovery using a CVD.

The simple mass balance results show an overall recovery of 98.4% of the tungsten in a 17.9% mass yield. The final tailings grade was assayed to be 0.07% W. The actual tailings grade may be lower as some of the tailings samples analyzed were below the assayable detection limits.
APPENDICES

Appendix A
Test Program Flow Sheet Diagram

Appendix B
Metallurgical Results

Appendix C
ICP Results
Battelle Manipulated and Reduced Data
Appendix A

Test Program Flow Sheet Diagram

Feed 219.016 kg

Screened 12 mesh

Screened 16 mesh

Feed 160.500 kg

CVD 1st Pass

CVD 2nd Pass

CVD Tails

Oversize 58.516 kg

Concentrate Stage 1 18.472 kg

Concentrate Stage 2 10.298 kg

7.1.1.1 CVD-6 Operating Parameters

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<tr>
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Appendix B

Metallurgical Results

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<th>W (g)</th>
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Note: The Final Tails mass and the Totals (Head) mass are based on the sample weight after screening to 16 mesh (1.18mm)

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Appendix C

ICP Results

PNNL Manipulated and Reduced Data
ICP Results as received from PNNL

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### PNNL Manipulated and Reduced Data

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Data Analysis – 6/8/04

CVD Pilot Test

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Knelson Research and Technology Centre