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STATE OF WASHINGTON  
DEPARTMENT OF ECOLOGY

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March 16, 1994

Mr. Eric Goller  
U.S. Department of Energy  
Richland Operations  
P.O. Box 550  
Richland, WA 99352

Dear Mr. Goller:

Re: Review of the 100 Area Soil Washing Bench-Scale Tests

The Washington State Department Of Ecology and the U. S. Environmental Protection Agency have completed their review of the *100 Area Soil Washing Bench-Scale Tests Document DOE/RL-93-107 Draft A*. Enclosed please find the comments. 33856

Ecology would like to resolve comments within a 30-day time period so that the pilot study schedule is not jeopardized. The report comparing XRF with standard SW-846 methods is still outstanding. Please submit it as soon as possible so that key decisions regarding the level of data QA/QC can be made in a timely manner.

If you have any questions, please contact me at (509) 736-3012.

Sincerely,

Ted Wooley, Unit Manager  
Nuclear Waste Program

TW:sl  
Enclosure

cc w/enclosure:  
Dennis Faulk, EPA  
Jim Field, WHC  
Administrative Record (100 Area Treatability Study)

cc w/o enclosure:  
Steve Wisness, USDOE



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## 100 AREA SOIL WASHING BENCH-SCALE TESTS

### DOE/RL-93-107 DRAFT A

#### GENERAL COMMENTS:

- 1) Overall this document provided valuable information about whether soil washing can effectively reduce radionuclide contamination in some 100 Area soil.
- 2) Based on the bench scale tests, several key parameters including particle size and radionuclide activity distribution, presence of aluminosilicate and iron oxide coatings on soil fractions and the quantity of micaceous minerals in the soil matrix have been identified to predict soil washing effectiveness. However, more data is needed to develop a reliable, predictive model.

The 116-C-1 soil was not effectively treated using the autogenous grinding methods with electrolyte solutions or chemical extractants employed in this study. It may be possible to achieve cleanup levels through more intense surface grindings, leaching with hot mineral acids, or conducting autogenous grindings in a hot chemical extractant.

- 3) More data is needed before or in parallel with the pilot-scale soil washing. The data requirements include:
  - A) Develop a predictive model based on soil characteristics conducive to washing
  - B) Determine if extractant mobilizes trace metals and clean backfill fails TCLP
  - C) Determine recyclability of electrolyte and extractant in waste stream
  - E) Determine variability of contaminant activities and particle size distribution of soil
- 4) The Executive Summary indicated that less attention would be given to Batch I soils because of the notable absence of radioactivity. To that end, it would be useful to define more clearly how data generated from analysis of the Batch I soils (in regards to Batch II and Batch III soils) should be used, and whether it is prudent to perform further analysis of Batch I soils in the future.
- 5) The discussion in Section 8 on percent activity removed, based on the use of proprietary extractants will not be considered useful, until the chemical composition of these extractants are revealed. This information will have to be available well in advance of regulatory approval of their use.

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- 6) There needs to be a more comprehensive discussion regarding the conformational sampling that will occur for soil particles greater than 2 mm in diameter. It is not a given that use of the analytical method XRF will be acceptable as replacement for SW-846 methods. Therefore, the validity of the report will be contingent on an acceptable solution to the analytical limitations that are plaguing this bench test.
- 7) Cleanup levels or target performance levels are based on the 1988 Westinghouse document. It is important that regulatory cleanup levels are set to determine the actual success of soil washing technology.

**SPECIFIC COMMENTS:**

- 1) Deficiency: Section 2, Page 2-3, Paragraphs 1 and 3

Wet-screening is referred to as both wet-screening and wet-sieving.

Recommendation: It may be confusing to use both terms. Use one or the other or if there is a difference, clarify what it is.

- 2) Deficiency: Section 3, Page 3-6, Soil Sample Collection

There is no discussion on the minimum acceptable cpm levels for soil samples. It is obvious that the highest levels are the most desirable for running tests on however, there should be discussion regarding the lowest cpm that samples were collected at, and why.

Recommendation: Add a sentence or two describing the rational for setting the lower limit.

- 3) Deficiency: Section 4, Page 4-6, Table 4-1

Y-axis title reads Cumulative % Finer by Weight.

Recommendation: Change Finer to Fines.

- 4) Deficiency: Section 4, Page 4-7, Table 4-3

This table reports TOC content for all batches. There is no indication as to the particle size that was analyzed.

Recommendation: Provide a legend that stipulates a particle size of 2 mm or less.

- 5) **Comment: Section 4, Page 4-8, Table 4-5**

It is noted in Table 4-1 that 97.2% of the batch II soils are greater than 4.75 mm. Table 4-5 reports CEC for soils 2 mm or less. What percentage of soil does the CEC number reported for batch II soils actually represent?

- 6) **Recommendation: Clarify the percentage of batch II soil that is actually 2 mm or less.**

- 7) **Comment: Section 4, Page 4-11, Table 4-8**

This table reports Accessible Soils Activity Limits. The reference indicates that these numbers came from the same document as the Test Performance Levels; however, it is not clear whether these two sets of numbers are the same or different.

**Recommendation: If there is no difference in the two sets of numbers, then use either TPL or ASAL, not both.**

- 8) **Comment: Section 8.3, Page 8-2, 3rd and 4th Paragraphs**

Conclusions provided in paragraph three indicate that no single standard extractant is capable of reducing activities of all contaminants of concern. Paragraph four discusses the effectiveness of the proprietary extractants I and II however, there is not even a minimum amount of information on the chemical characteristics of these solvents. It would be useful if characteristics such as pH, and solubility for a given molarity of extractant I and extractant II are provided so that a quick comparison could be made between the standard extractants and the proprietary ones. It is very unlikely that this type of information would allow patent embezzlement to occur.

**Recommendation: Provide this information.**

**EPA GENERAL COMMENTS:**

- 1) The report clearly describes the bench-scale tests completed by Pacific Northwest Laboratories (PNL) as part of the remedy screening phase (Phase I) of an overall three-phase treatability study. The treatability approach is well grounded in the geochemistry of the contaminants to be removed from soils (cobalt-60, cesium-137, and europium-152). However, the report does not integrate the approach presented in the *100 Area Soil Washing Treatability Test Plan* (DOE 1992), or the testing procedures described in the *100 Area Soil Washing Bench-Scale Test Procedures* (Freeman et al. 1992). The bench-scale tests report should present test

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results in the context of the approach required by the test plan and testing procedures.

- 2) Significant changes have been made in the target performance levels (TPL) identified in the test plan (Table 1-1, DOE 1992) and those identified in this study (Table 3-1). These changes have increased the TPLs by factors of 5 to 200 (in the case of strontium-90). Both the test plan (DOE 1992) and the bench-scale tests document refer to the environmental compliance manual (WHC 1988) as the source of significantly different data for TPLs. The TPLs presented in the test plan (DOE 1992) are significantly more protective than those presented in the bench-scale tests report. The choice of TPLs has enormous importance in determining the success or failure of the soil treatments evaluated in this report. The bench-scale tests report should clearly document any changes in TPLs agreed to by the three parties subsequent to finalization of the test plan (DOE 1992). Otherwise, the report should discuss the rationale for changing the TPLs to significantly higher values in greater detail.
- 3) Linear density gradient fractionation tests are not discussed in the bench-scale tests report. Although these tests are not specifically mentioned in the treatability test plan (DOE 1992), they are described in the bench-scale test procedures (Freeman et al. 1992). If these tests were completed, the results should be presented. If the tests were not performed, the text should explain why these tests were not conducted.
- 4) Heap leaching tests, discussed in Section 6.0 of the bench-scale test procedures (Freeman et al. 1992), do not appear to have been conducted during the bench-scale tests. However, static leaching tests are briefly described in Section 8.3 of the document, with results presented in Table 8-4. If the static leaching tests are the equivalent of the heap leaching tests, the report should clearly state so. If they are not, the differences between the two tests should be explained and a rationale for not conducting the heap leaching tests should be provided.
- 5) In the report, it is hypothesized that the majority of radioactive cesium contamination is bound to "wedge" sites on the edges of mica minerals. Techniques for separating mica from the bulk soil (such as density differences) should be investigated to determine if cesium can be efficiently concentrated and removed from contaminated soil by these methods. This task could be added to the supplementary data requirements described in Section 11.0 of the report.
- 6) Finally, the quality of the data obtained from the bench-scale tests should be discussed. The discussion should include analyses of the quality control samples, data validation procedures, and corrective actions taken to process unacceptable data. Completeness, measured in terms of valid data obtained from measurement system compared to the amount expected under normal conditions, should be

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identified. This information should be presented so that the data quality can be evaluated.

**SPECIFIC COMMENTS:**

- 1) Section 4.2.6, page 4-3. The text lists seven regulated metals that the samples were analyzed for by toxicity characteristic leaching procedure (TCLP) tests. However, Table 4-10 lists eight metals (the seven regulated metals plus silver) that were analyzed for using those tests. The text should include silver as a regulated metal for which these samples were also analyzed.
- 2) Section 4.4.2, page 4-9, first paragraph. This section identifies cobalt-60, cesium-137, and europium-152 as the contaminants that exceed the TPLs in batch III soils. Table 4-8, which lists the radionuclide data for the 100 area soils, indicates that europium-154 also exceeded the TPL in both batch II and III soils. This radionuclide should also be identified as exceeding the TPL.
- 3) Section 4.4.2, page 4-9, second paragraph. This section states that the >2mm fractions of the batch II and III soils were analyzed for cobalt-60, cesium-137, and europium-152 radionuclides. These results should be provided in this section.
- 4) Table 4-7, page 4-10. This table lists the trace element concentrations, including vanadium, in the 100 area soil samples using x-ray fluorescence spectrometry. Section 4.2.4 identifies targets used in the total element analyses. The target used for vanadium analysis should be identified in Section 4.2.4. In addition, Section 4.2.4 lists cobalt as one of the analyzed elements; thus, the concentration of cobalt should also be included in Table 4-7.
- 5) Table 4-9, page 4-11. This table lists the activities and concentrations of radionuclides. The reported concentrations, which are based on the specific activity of the radionuclides, are 1,000 times lower than the reviewer's calculation of these values as shown below for cobalt-60 and cesium-137.

Cobalt-60:  $7 \text{ pCi/g} \times 1000 \text{ g/kg} \times 1 \text{ pg}/1133 \text{ pCi} \times 1 \text{ mg}/10^6 \text{ pg} = 6.17 \times 10^{-6} \text{ mg/kg}$

Cesium-137:  $0.74 \text{ pCi/g} \times 1000 \text{ g/kg} \times 1 \text{ pg}/87 \text{ pCi} \times 1 \text{ mg}/10^6 \text{ pg} = 8.5 \times 10^{-6} \text{ mg/kg}$   
(from Gorbitt 1989)

The calculations in the report should be checked and the table should be corrected accordingly.

- 6) Section 4.4.2, page 4-12, second paragraph. The last sentence of this section compares europium-152 recovery of batch I and III soils by sequential extraction. Radionuclide recovery data for batch I should be listed in Table 4-11.

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- 7) Section 5.2, page 5-2. This section states that at the end of the sieving cycle, soil fractions were rinsed with fresh deionized water. It should be explained how this rinse water was processed i.e., was it added to the recycled water or treated differently.
- 8) Section 5.3, page 5-6, first paragraph. This paragraph discusses Figure 5-4, which was not, but should be, included in the document.
- 9) Section 6.2, page 6-3, second paragraph. The last sentence of this section states that the wash water from the single stage attrition scrubbing was counted for radionuclide activity. These results should be provided in this report.
- 10) Section 7.1, page 7-1. The last sentence of this section states that since washing batch II gravels with water did not significantly reduce the activity of radionuclides, additional physical treatment such as autogenous grinding was tested. The results of water washing of batch II gravels should be provided in this report.
- 11) Table 7-1, page 7-3. This table provides the autogenous grinding data for gravels from batch II soil. Two of the tested treatment processes included grinding with sand. Percent fines for these processes are defined in the footnote as the fraction of fines generated from rocks or groundup sand. Procedures used to identify these fractions should be discussed.

Section 8.3, page 8-2, first paragraph. This section states that a minimum removal efficiency of 50 percent for cesium-137 is required to meet the TPL. The initial activities of cesium-137 in the samples analyzed from the 2- to 25-mm sized fraction range from 90 to 94 pCi/g (Table 8-1). With the TPL of 30 pCi/g for cesium-137, this removal efficiency should be about 67 percent. The source of this 50 percent removal requirement should be identified.

- 12) Table 8-2, page 8-3. The footnote to this table provides the solid-to-"solution" ratio for extraction II-3. This footnote should be corrected to show extraction II-C. In addition, Section 8.2 provides information on the weight of solids and extractant combination, which is the "solution." This information results in solid-to-"solvent" ratios of 1 to 2 and 1 to 4. The footnote should correctly identify this as a solid-to-"solvent" ratio.
- 13) Section 8.3, page 8-4, second paragraph. This section discusses the results of static chemical leaching of gravel fractions of batch II soils with extraction II. The concentration of this extractant used with the solid-to-solvent ratio should be identified.
- 14) Section 9.2, page 9-1, second paragraph. This section describes the combination

tests; the solution temperatures at which these tests were conducted should be specified.

Additionally, one of the experiments in the combination tests consisted of surface grinding the gravel-sized fraction with extractant II at 50 percent solids by weight. This concentration of solids is higher than the concentrations identified in Section 8 of this report (20 to 33 percent solids by weight). The rationale for selection of this concentration should be explained.

- 15) Section 9.3, page 9-2, first paragraph. This section provides the results of the combination tests, which include two-stage scrubbing in deionized water or in an electrolyte. The duration of this scrubbing should be identified for comparison of test results from each test.
- 16) Section 11.3, page 11-3, second paragraph. This section lists additional tests that may achieve the required cesium-137 removal from the contaminated soils. These tests include: more intense surface grinding, leaching with hot mineral acids, and autogenous grinding in hot chemical extractant. Two-stage autogenous grinding with extractant II should also be considered as a potential method to remove cesium-137 to below the TPL.

Table 11-1, page 11-5. The columns listing the average contaminant levels should identify the measurement units (i.e., pCi/g).

Appendix A, page A-1. This table shows that the vendor quotes were multiplied by 2.5 for Hanford for the "purchase and mobilize" items. The rationale for this increase should be explained.

## **REFERENCES**

DOE 1992. 100 Area Soil Washing Treatability Test Plan. DOE/RL-92-51. U.S. Department of Energy. November.

Freeman, H. D., M. A. Gerber, S. V. Mattigod, and R.J. Serne 1992. 100 Area Soil Washing Bench-scale Test Procedures. PNL-8520. Pacific Northwest Laboratory, Richland, Washington. December.

Gorbitt, Robert A. 1989. Standard Handbook of Environmental Engineering. McGraw Hill Publishing Company. New York.

WHC 1988. Environmental Compliance Manual. WHC-CM-7-5. Westinghouse Hanford Company, Richland, Washington.

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