Formerly Utilized Sites Remedial Action Program (FUSRAP)

# ADMINISTRATIVE RECORD

for the Maywood Site, New Jersey



US Army Corps of Engineers.

N-763

### Characterization and Treatability Studies of Subsurface Soil Samples from the Maywood FUSRAP Site

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#### EXECUTIVE SUMMARY

The study describe herein was performed by Sanford Cohen and Associates under subcontract to Science Applications International Corporation. This report presents the results of the treatability study for subsurface soils at the Maywood FUSRAP Site. Particle-size and radionuclide distributions were determined for seven whole-soil samples from the Maywood Site. Further characterization and treatability studies were performed on two samples, Composites 1015 and 1016, which were formed by selectively combining the seven individual samples. The criteria and methodology used for compositing the samples are given. The primary radioactive contaminants in the soils at the site are those appearing in the U-238 and Th-232 radioactive decay series. The radionuclides of particular interest, because they are most closely related to site cleanup criteria, are Ra-226, Th-232, and U-238.

This treatability study was conducted in two parts, an initial characterization study and an extended study with process conceptualization. The initial characterization was conducted by Sanford Cohen and Associates (SC&A) at its Southeastern Environmental Laboratory in Montgomery, Alabama. The second portion of the study was performed by the Center for Coals and Mineral Processing of Virginia Tech University in Blacksburg, Virginia, under subcontract to SC&A. This report is presented in two parts to reflect the division of performance.

The primary objectives of the initial characterization of the samples were to: (1) determine if particle-size separation techniques can be effective in reducing the volume of contaminated soil, (2) identify any radiominerals present, (3) determine the presence and magnitude of contamination associated with particles of high density and ferromagnetism, and (4) determine if chemical scout extraction tests are effective in reducing the volume of contaminated soil in these samples.

#### Initial Characterization Study

The results of this initial characterization study led to the following conclusions and recommendations:

Particle-Size Separations

Particle-size separation alone, on Composite 1015, is not capable of achieving a Total Activity

Ratio (TAR) of one, while achieving significant volume reduction. The TAR is defined as:

$$TAR = \left(\frac{Ra - 226 + Th - 232}{5}\right) + \left(\frac{U - 238}{50}\right)$$

where Ra-226, Th-232, and U-238 are the concentrations of the respective radionuclides in pCi/g. Other target goals of less than 5 and 15 pCi/g for Ra-226 and Th-232 are achievable with volume reductions of greater than 60% for a 200-mesh-size cut. Particle-size separation alone will not be effective in meeting any target goals for Composite 1016.

#### Petrographic Analysis

Composite 1015 is comprised of a variety of natural and anthropogenic materials. Natural rock and minerals constitute more than half of the particles (54 percent). Radioactive materials and materials suspected to contain radioactivity are believed to be present in about one-third of the sample. Known natural radioactive minerals are monazite and zircon that occur only in the fine sand and coarse silt-size fractions. The monazite averages about 0.028 percent of the soil sample and zircon about 0.07 percent of the soil sample. A substance that potentially could be a higher contributor to the radioactivity in the soil is a white, clay-like, anthropogenic thorium substance that comprises three percent of the soil sample. This material is believed to be a thorium orthophosphate. Coal-fired, clinker slag comprising about 12 percent of the sample contributes residue ash of uranium and/or thorium that produces radioactivity above background levels.

Radioactive materials are believed to be contained in white glassy slag comprising four percent of the soil. Solid magnetic and nonmagnetic slag comprising, respectively, 7 and 13 percent of the soil sample, also might contain radioactivity above background levels. This material is suspected to be associated with the industrial manufacture of thorium and may possibly contain radioactive residual products.

Composite 1016 from Maywood is comprised of a variety of natural and anthropogenic materials. The natural rock and minerals comprise more than 45 percent of the soil and anthropogenic slags.

Radioactive monazite and zircon are natural minerals that are present as significant sources of radioactivity. Both these minerals are found only in the fine sand and coarse silt-size fractions; however, the monazite comprises 3.33 percent and zircon 0.82 percent of the soil sample. The

concentration level of these radioactive minerals suggests their presence is derived form ore minerals used to obtain thorium.

A white anthropogenic thorium substance comprises 8 percent of the soil sample tested. This material is probably an orthophosphate derived from the industrial process to obtain thorium metal. It is likely one of the main contributors of radioactivity in the soil sample.

Radioactivity is believed to be present in some of the slag materials that may be related to the industrial process used to manufacture thorium metal. Individual fractions of white glassy slag comprising 5 percent of the soil; and solid magnetic and nonmagnetic slag, comprising 21 percent of the soil sample, should be examined to determine if this material is an important source of the radioactivity in the soil.

• Dense and Magnetic Components

The results of the density and magnetics scoping study show that significant amounts of the radioactivity in Composites 1015 and 1016 are associated with dense (>2.89 g/cc) minerals. These results indicated that density separation in particular shows promise for the removal of beneficial amounts of thorium. These studies showed the need for further characterization of the radioactive mineral associations using SEM/XRF and the need for additional laboratory studies on the composite samples to evaluate the potential for additional partitioning by specific gravity and magnetic susceptibility. The results of these studies are presented in Part II of this report.

• Attrition

The attrition study on Composite 1015 did not indicate significant benefit from attrition to the -4/+100 mesh material. The results of the attrition test at 70 percent solids for Composite 1016 indicated discernable removal of radium and thorium. The use of a vigorous washing step before sieving the samples prior to attrition may have been sufficient to liberate the fine material from the coarse thus obscuring the benefit of such liberation. Any proposed process design would most certainly include some form of particle liberation as a precursor to any separation process.

Chemical Extraction

For Composite 1015 the most effective extractant for both thorium and radium (Ra-228) was 3M HCl followed by 0.22M sodium hexametaphosphate, with removals of 80 and 63 percent,

respectively. The resulting extracted soil concentrations of Ra-226, Ra-228, and Th-232, respectively, were 1.27, 4.48, and 1.48 pCi/g. Only 6 percent of the soil was digested during the extraction.

While not as effective as 3M HCl/sodium hexametaphosphate, 0.1M EDTA/0.5M Na<sub>2</sub>CO<sub>3</sub> removed 62 and 24 percent of the thorium and radium, respectively. The resulting Ra-226, Ra-228, and Th-232 concentrations of the extracted soil were 1.63, 7.31, and 2.84 pCi/g, respectively, which would result in a TAR of less than one. Less than two percent of the soil was solubilized in this extraction.

For Composite 1016 the most effective extractant for thorium, uranium, and radium (Ra-228) was  $3M \text{ HNO}_3$ , with removals of 77, 73, and 78 percent, respectively. The resulting extracted soil concentrations of Ra-226, U-238, and Th-232, respectively, were 8.5, 3.4, and 25.7 pCi/g (TAR = 6.9). Approximately 6 percent of the soil was digested during the extraction.

While not as effective as  $HNO_3$ ; 3M HCl followed by 0.22M sodium hexametaphosphate removed 62, 67, and 71 percent of the thorium, uranium, and radium, respectively. The resulting Ra-226, U-238, and Th-232 concentrations of the extracted soil were 9.8, 4.2, and 43.4 pCi/g (TAR = 10.7), respectively.

Chemical extration was not pursued beyond the scout testing and the remainder of the study concentrated on physical separation. The scout studies indicate that chemical separation, following an extensive optimization study, could offer remediation potential for Maywood soils.

#### Extended Characterization and Process Design Study

The results of this extended characterization and process design study (Part II) led to the following additional conclusions and recommendations:

• Extended Petrographic Analyses

Detailed petrographic analyses were conducted on Composite 1015 and Composite 1016 and on a sample collected from the Wayne FUSRAP site. In order to achieve more statistically valid results, some of the data from the analysis of that sample are included in this report. Scanning Electron Microscopy coupled with Automated Image Analysis indicated that the only radioactive component observable via x-ray analysis under SEM was thorium. Thorium was found to occur

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primarily as a monazite-type particle in the heavy mineral sands. The amount of soil uncontaminated by the presence of heavy mineral sands was generally found to increase as particle size decreased. Also, the percentage of free heavy minerals contained in each sample was generally found to increase with decreasing particle size. In all cases, the 100x200 mesh size fraction seemed to stand out as the worst in terms of heavy mineral contamination.

In an attempt to relate the radionuclide analysis to the characterization data, the TAR was compared to the heavy mineral content and the thorium mineral content of each size fraction. The TAR was found to correlate well with the thorium mineral content. A plot of TAR versus thorium mineral content indicated that these soils must be cleaned to a thorium mineral content of less than 0.06 percent in order to achieve a TAR of 1 or less. Based on the theoretical cut-off limit on thorium mineral content, Composite 1015 was found to exhibit characteristics of a soil that would be amenable to physical separation. Composite 1016, on the other hand, appeared to contain far too much locked material to ever meet target criteria.

• Laboratory Characterization Tests

Float-sink analyses were performed on the various size fractions of the Composite 1015 and Composite 1016 composite soil samples. The test data confirmed the findings of the detailed petrographic results which indicated that remediation of Composite 1016 could not be achieved, although the total activity of the soil could be significantly reduced. For Composite 1015, the float-sink tests indicate that the largest weight percentage of the intermediate-sized soil particles are present in the 2.6 x 2.7 density fraction. These particles have Th-232 and Ra-226 activities below 5 pCi/g.

Froth flotation tests conducted on the finer size fractions of the Composite 1015 soil indicate that flotation is capable of concentrating radionuclides in the froth product. For the minus 50 mesh flotation test, the Th-232 activity of the clean soil was reduced by 23 percent, while the concentrate Th-232 activity increased 63 percent. These values were changed to 20 percent and 320 percent, respectively, for the 50x200 mesh flotation test. The concentrate obtained for tests conducted on -200 mesh soil showed increases of up to 170 percent in Th-232 activity. Additional optimization tests are recommended based on these promising results.

Conceptual Flowsheet Development

Test data obtained from the petrographic and laboratory characterization studies were used to

develop a conceptual flowsheet for the Maywood FUSRAP site. The flowsheet was developed using spreadsheet-based mass balancing and process simulation programs that were developed specifically for this project. For the Composite 1015 soil sample, the simulation results indicate that primary screening followed by hydroclassification (Circuit I) is capable of meeting a Th-232 activity of 5 pCi/g at a soil recovery of 52 percent by weight. Adding a low-cost density separator (Circuit II) to reprocess the coarse underflow product from the hydroclassifier, results in achieving a Th-232 activity of 5 pCi/g and a soil recovery of 52 percent. However, an attempt to decrease the allowable Th-232 activity below 5 pCi/g showed a significant decrease in soil recovery for the two circuits. For example, at a 4.5 pCi/g of Th-232, the soil recoveries are 25 and 50 percent for Circuit I and Circuit II, respectively. These data indicate that operating Circuit I at a 5 pCi/g of Th-232 for the final product will have little margin for error. The decline in soil recovery is more subtle with Circuit II. Therefore, the Circuit II configuration will provide a "safety-net" around the cleanup level of 5 pCi/g and is the circuit of choice. Because of the importance of Th-232 removal in meeting the clean-up criteria, an additional series of optimization runs were performed in which the soil recovery was maximized at different levels of Th-232 activity. These simulation data show that only about 52 percent of the soil can be recovered at a Th-232 activity of 5 pCi/g. However, the mass recovery of clean soil can be increased to about 63 percent by increasing the allowable Th-232 activity to 13.5 pCi/g. A further increase in allowable Th-232 activity is not technically feasible with the Circuit I or Circuit II configurations. The best operating point for reducing the Th-232 activity while maximizing soil recovery would be to produce a clean soil with a Th-232 activity of approximately 5 to 13 pCi/g. These operating points would result in mass recoveries of approximately 55 and 63 percent and could be achieved using either Circuit I or II.

A preliminary cost analysis was conducted to examine the economic feasibility of the proposed circuitry. The cost analysis included an estimation of both capital and operating costs for the installation of processing facilities capable of treating as little as 62,100 tons and as much as 550,845 tons of contaminated soil. For the smaller volume, the estimated cost for remediation varied from \$198.42 to \$162.48 per ton of dry feed soil depending on whether the cleanup target was set to 5 pCi/g or 15 pCi/g, respectively. These values represent a cost savings of more than \$8.4 million as compared to complete excavation and disposal of soil from the Maywood site. For the greater tonnage of 550,845 tons, the estimated remediation cost was found to vary from \$179.69 to \$143.73 per ton of dry feed soil for the cleanup targets of 5 pCi/g or 15 pCi/g, respectively. In this case, a total cost savings of more than \$85 million may be realized by combining soil treatment and disposal at the Maywood site.

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to the extraction of thorium from monazite sands to be used as lantern mantles.

Process wastes migrated and were transported off the site such that the properties now know collectively as the Maywood Site include a portion of the original property (now owned by the Stephan Company); the Maywood Interim Storage Site (MISS), on a portion of the original property; residential vicinity; and commercial/governmental vicinity properties. The samples for this study are from the vicinity commercial properties; namely, subsurface soils from the Stephan and MISS properties.

Field Designation	Laboratory Number	SPCS East	SPCS North	Site East	Site North
Borehole 3	1004	2164081.97	752414.06	9600.33	9301.11
Borehole 4	1005	2164090.80	752976.07	9270.33	9756.12
Alt Borehole 5	1008	2164250.70	752671.07	9581.20	9607.97
Borehole 6	1010	216451.94	752524.29	10150.34	9851.12
Alt Borehole 7	1006	2164326.80	752083.42	9994.55	9183.38
Alt Borehole 8	1007	2165061.53	752502.07	10331.37	9959.04
Alt Borehole 10	1009	2165230.93	752035.29	10746.88	9687.13

Table 1-1: Maywood Sample Location and Identification

Following particle-size and radionuclide analyses, the seven (individual) samples were combined into two composite samples in a manner (Section 2.6) to best represent the Maywood soils during studies to assess their potential for treatment.

# PART I:

## **INITIAL CHARACTERIZATION**

#### 2.0 EXPERIMENTAL

#### 2.1 Analyses

#### 2.1.1 Gamma Spectrometry

Whole soil samples, particle-size fractions, and wash waters were analyzed for gammaemitting radionuclides using a high-purity germanium detector (EPA80). The wash water samples were counted for 1000 min, and the soil samples were counted for 100 to 1000 min, according to the levels of radioactivity. Due to the presence of uranium in the samples, erroneous Ra-226 values were first obtained from the gamma spectrometry analysis program. This is because the primary Ra-226 and the U-235 gamma-ray emissions are only approximately two kev apart and are not easily distinguished by gamma spectrometry analysis programs. Values now reported for Ra-226 are based on the Pb-214 values reported by the program and more accurately represent the actual Ra-226 concentrations. To assure that Ra-226 progeny, including Pb-214, were in radioactive equilibrium with Ra-226, each of the individual whole soil samples was sealed, counted, stored for approximately a month for Rn-222 ingrowth, and recounted. Within experimental error there were no differences in Pb-214 concentrations reported between the two counts, thus radioactive equilibrium is assumed between Ra-226 and Pb-214 and all Ra-226 data throughout this report are the Pb-214 values as reported by the gamma spectroscopy program.

#### 2.1.2 Alpha Spectrometry

Analyses for uranium and thorium radionuclides were performed by alpha spectrometry (EPA84). Uranium was extracted, coprecipitated with lanthanum fluoride carrier, and analyzed by alpha spectrometry. Thorium was separated by ion-exchange chromatography, coprecipitated with lanthanum fluoride carrier, and analyzed by alpha spectrometry.

2.2 Particle-Size Analysis

#### 2.2.1 Sample Preparation

The seven whole-soil samples for this study were received at the SEL where they were initially screened for gross beta/gamma activity using a Geiger-Mueller portable survey instrument. After initial screening, each sample was thoroughly mixed and split into 400-mL aliquots using a riffler which was set to allow the passage of material smaller than three-

fourths inch. The aliquots were weighed, dried at 60°C for 48 hr, and reweighed. Two aliquots from each whole soil were analyzed by gamma spectrometry. After the aliquots were ashed at 565°C for 72 hr (all weights reported are dry), they were analyzed for uranium and thorium by alpha spectrometry. The material removed by the riffler was thoroughly washed with water by hand to remove attached soil particles, dried, weighed, and gamma counted. The weight and radioactivity associated with this material were accounted for in the whole soil values.

#### 2.2.2 Vigorous Wash

After sample preparation, sufficient aliquots were combined to obtain a minimum of two kg of whole soil that was then vigorously washed in water for 30 min at a rotational velocity of 350 rpm with a liquid-to-solid ratio of 4 mL/1g (SCA-502). The vigorous washing process liberates small particles from large particles without generating excessive fines.

#### 2.2.3 Wet Sieving

After vigorous washing, an aliquot of each soil sample was fractionated by size using wet sieving with American Standard Testing of Materials (ASTM) standard sieves (SCA-503). The aliquots were separated into particle-size fractions at 4 mesh (4.75 mm), 8 mesh (2.38 mm), 16 mesh (1.19 mm), 30 mesh (0.590 mm), 50 mesh (0.297 mm), 100 mesh (0.149 mm), 200 mesh (0.074 mm), and 400 mesh (0.037 mm). The resulting fractions were dried at 60°C for 48 hr, weighed, and analyzed for radionuclide content by gamma spectrometry. Alpha spectrometry for uranium and thorium was performed on all fractions from all of the individual samples and the -200/+400 and -400 fractions of the two composite samples (see Section 2.6). Thorium analyses were performed on the remaining fractions of Composite 1015.

#### 2.2.4 Wash Water

Water from the vigorous wash and separation procedures for each individual and composite sample was collected and a Percol 788N flocculant added to settle suspended material. The water was then filtered through a 0.025-mm pore filter paper to separate suspended solids from the wash water. The material retained on the filter was analyzed by gamma spectrometry and, if called for, by alpha spectrometry and reported as the -400 activity, since the 400-mesh sieve is the last in the wet sieving stack. An aliquot of filtered wash water from each of the eight whole soil samples was analyzed by gamma spectrometry.

#### 2.3 Formation of Composites 1015 and 1016

The seven individual soil samples from Maywood were collected from subsurface locations on the Stephan and MISS site. Following particle-size and radionuclide analysis, the seven samples were combined into two samples of approximately 8 kg each. The composites were generated from the individual samples in such a manner as to best represent the material to be treated at the site, as forecast by these samples. The samples were composited based on particle-size distribution, radionuclide distribution, and quantity of site material estimated to be represented by each sample.

Figures 2-1, 2-2, and 2-3 show the Ra-226, Th-232, and U-238 concentration contours, respectively, for the Maywood site. These contours were generated from the radiological borehole data in DOE92. The locations of areas of the highest radionuclide concentrations at the Maywood site are easily identified from these plots. The area with the highest concentrations of all radionuclides is at approximately East 10000 to 10500 and North 96000 to 10100.

The concentrations as a function of depth cannot be accurately predicted because of the inconsistent sampling depths of the boreholes; therefore, the concentration contours are plotted on a single plane. The planer area represented by the higher concentrated locations is predicted to be approximately 25 percent of the site.

The seven samples received at SC&A were taken at locations whose coordinates and depths shown in Table 2-1. Borehole 6 and Alt Borehole 10 are located in two of the areas where high concentrations of radionuclides are expected. This agrees with their significantly higher whole-soil activities. Alt Borehole 8 is located on the edge of the area of highest concentration. Borehole 3, Borehole 4, Alt Borehole 5, and Alt Borehole 7 are located outside the areas with the highest radionuclide concentrations. The depths of the boreholes vary from 2.5 to 11.0 ft. No correlation between depth and activity can be drawn due to the variability in the sample depths.



🔄 Sorehole 3 🖬 Borehole 4 🕅 Alt Borehole 5 💭 Borehole 5 🛄 Alt Borehole 7 🙀 Alt Borehole 8 🖬 Alt Borehole 10

σ







Figure 2-3: Uranium Concentration Contour

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Semula ID	Coord	Depth	
Sample ID	East	North	(ft)
Borehole 3	9600	9300	9.0
Borehole 4	9500	9400	11.0
Alt Borehole 5	9581	9608	7.0
Borehole 6	10150	9850	6.0
Alt Borehole 7	9995	9183	2.5
Alt Borehole 8	10331	9959	4.0
Alt Borehole 10	10747	9687	9.0

Table 2-1: Maywood Borehole Sample Locations and Depths

No samples were excluded from the composite based only on their particle-size distributions. Borehole 3 has the only distinctly different particle-size distribution and was not weighted heavily in its composite.

From the whole-soil activity data, there are two distinct groupings. Borehole 6 and Alt Borehole 10 have significantly higher activities than the other five samples. Therefore, these two samples were not composited with the other five.

The location of the samples had a significant influence on the weighting of the composite. From the concentration contour data and the location of the samples, there are two groupings. Four samples, Borehole 3, Borehole 4, Alt Borehole 5, and Borehole 6 are from the MISS property and three samples, Alt Borehole 7, Alt Borehole 8, and Alt Borehole 10 are from the Stephan property. However, property identity was not a consideration in compositing. Alt

Borehole 8 is located on the border of the highest concentration area, therefore its activity may misrepresent the activity of the surrounding area. Based on its location, Alt Borehole 8 was weighted less in the composite. Alt Borehole 5 is located on the border of the storage pile, therefore the amount of area represented by its activity may be small. However, the concentration contour data shows that the area to the south and east has approximately the same activity. Thus, Alt Borehole 5 was assigned more weight in the composite than Alt Borehole 8, but not as great as other samples that represent a more definitive area. Borehole 3 and Borehole 4 are near the New Jersey State Highway 17 and, thus, do not represent a large area of soil on the site. Borehole 4 does represent a larger area than Borehole 3 and its particle-size distribution is more typical for the site, therefore it was weighted greater. Alt

Borehole 7 represents the largest area for the samples that are not in an area of highest concentration. Therefore, based on the concentration contour data and location, this sample has the greatest weight in the composite.

Based on the above reasoning and using the particle-size, radioactivity, and concentration contour data developed for the individual samples, the composites were formed as shown in Table 2-2. Composite 1015 consists of a weighted composite of Borehole 3, Borehole 4, Alt Borehole 5, Alt Borehole 7, and Alt Borehole 8. The distribution of the weight percents are 10, 25, 20, 35, and 10, respectively. Composite 1016 consists of a weighted composite of Borehole 6 and Alt Borehole 10. The weighting percents are 80 and 20, respectively.

Borehole 3, Borehole 4, Alt Borehole 5, Alt Borehole 7, and Alt Borehole 8 are chosen to be composited based on their similarity in the whole-soil activities. Each sample is weighted based on its particle-size distribution and amount of area believed to be represented by it. Borehole 4, Alt Borehole 5, and Alt Borehole 7 are weighted the heaviest due to the large areas represented by these samples. Borehole 3 is weighted less due to its atypical particle-size distribution. Alt Borehole 8 is weighted less due to its location on the edge of the area of highest concentration: therefore, its activity may misrepresent the activity of the surrounding area.

Borehole 6 is weighted heavier than Alt Borehole 10 in the Composite 2 sample based on the larger area represented by its activity. This composite sample represents a less promising soil that may have some unique property that can be exploited to remove the radionuclide contaminants.

Composite Sample ID	Borehole ID	Percentage
	Borehole 3	10
	Borehole 4	25
Composite 1015	Alt Borehole 5	20
(More Promising)	Alt Borehole 7	35
-	Alt Borehole 8	10
Composite 1016	Borehole 6	80
(Less Promising)	Alt Borehole 10	20

Table 2-2: Percentages of Individual Samples Included in the Maywood Composite Samples

#### 2.4 Petrographic Study

Petrographic examination was made of the two composites for the purpose of determining the minerals or materials containing the radioactive particles and to ascertain the physical properties and composition of the host materials. The petrographic tests were conducted in accordance with procedure SCA-510.

#### 2.4.1 Procedure

Representative portions of the composite samples were wet sieved, following vigorous washing, and collected on the 4 (4.76 mm), 8 (2.38 mm), 16 (1.19 mm), 30 (0.590 mm), 50 (0.297 mm), 100 (0.149 mm), 200 (0.074 mm), and 400-mesh (0.037 mm) sieves (RIC89). The weight percent of soil fractions was obtained after drying the fractions at 60°C. Representative fractions were then prepared for radiochemical and petrographic analysis. The samples for petrographic analysis included the soil fractions on all the sieve sizes. The -400 sieve size (less than 0.038 mm) material was saved for SEM/EDX analysis to be performed at Virginia Tech (VPI).

Each of the sieve size fractions had its magnetic component removed by a hand magnet (SCA-507) and its radioactive concentrations measured. In addition, representative portions of the fine sand and coarse silt fractions were separated into a light and heavy fraction by the sink-float method. This density separation was made using sodium polytungstate (specific gravity = 2.89) as the heavy liquid in the sink-float method (SCA-506). Certain heavy materials are known to contain elevated concentrations of naturally occurring radioisotopes and, since the high density materials are a much volume than the light materials, this separation allows for a more accurate determination of their abundance in the sample.

The petrographic examination generally began with the coarse fractions by a visual classification of materials into categories. All material greater than gravel size (+4 sieve size) was examined visually and particles grouped into their respective categories. The various categories of materials were then weighed, and the compositions of the various fractions were determined as a weight percent. Physical properties, such as color, particle shape, structural strength, degree of hardness, particle density, degree of weathering, and porosity were noted as part of the examination.

The materials retained on the 8, 16, 30, and 50-mesh sieves were placed in a petri dish and examined under a binocular microscope. At least 100 particles in each sieve size were

examined and classified. Minerals and materials in the fractions retained on the 100, 200, and 400-mesh sieves were examined under the polarizing petrographic microscope. The materials were placed in index oils on a petrographic glass slide and more than 100 grains classified into their respective categories. The heavy minerals and materials, obtained by the sink- float method, were also examined under the petrographic microscope in a similar manner. The heavy mineral and materials were assigned to their various categories by point count. Transparent heavy minerals that are unique to this size range (fine sand and coarse silt-size) are an especially important category. The heavy minerals are generally present in minor quantities relative to the total size fractions, and their separation into mineral species warrants a separate tabulation with radioactive minerals highlighted.

Photomicrographs were made of minerals or other materials identified as radioactive, as were other categories of materials which might enhance reporting and characterization of the soil fractions.

#### 2.5 Attrition Study

Attrition removes surface material from soil particles by scrubbing while minimizing the fracturing or grinding of particles and the production of excessive fines. Attrition may be necessary when vigorous washing does not fully liberate surface coatings from particles.

The attrition studies were performed on the -4 to +100-mesh material for the two composite samples using procedure SCA-509. This procedure employs an industrial-size kitchen mixer. The use of a blender allows for the attrition testing using smaller samples sizes than those required for bench-scale attrition mills. The blender employs one impeller blade as opposed to two with opposing pitch usually found in an attrition mill unit. However, the scrubbing action obtained by the blender was sufficient to determine if attrition was likely to be an attribute in a treatment scheme.

Attrition was performed at 60 and 70 percent solids (60 percent only for Composite 1015) (soil weight/soil+water weight). These ratios were chosen because they are in the range of those encountered in attrition unit processes, while being able to maintain sufficient movement in the blender without overloading it.

Approximately 200 to 250 g of whole soil was wet sieved to obtain the -4/+100 material which was then diluted with tap water to 60 or 70 percent solids. The diluted material was placed in the industrial mixer, set at the lowest speed possible, and attritioned for 30 minutes.

The attrited material was then wet sieved at 4, 8, 16, 30, 50, 100, 200, and 400 mesh. The resulting fractions were dried at 60°C overnight, counted for gamma activity, and, after ashing, analyzed for thorium (Composite 1015 only).

2.6 Scout Chemical Extractions

Scout chemical extraction tests are conducted on the composite samples to assess the potential for remediation of whole or soil fractions to meet site criteria when physical separation techniques may fail to achieve the necessary degree of cleanup. Scout chemical extraction tests were performed on Composites 1015 and 1016 following procedure SCA-508.

The extraction solutions selected were  $0.1M \text{ EDTA}/0.5M \text{ Na}_2\text{CO}_3$  @pH=9.0; 3M HCl followed by 0.22M sodium hexametaphosphate; and 0.22M sodium hexametaphosphate. Composite 1016 was also extracted with 3M HNO<sub>3</sub>. The result of previous studies designed to extract thorium from contaminated soils led to the selection of these extractants.

#### 2.6.1 Sample Preparation

To conserve process costs and to maximize the radionuclide removal, it was desirable to perform chemical extraction only on portions of the sample requiring treatment beyond what physical processes can achieve and on those fractions where it is likely to be effective. Scout chemical extractions were performed with the three extracting solutions on the -4/+100 mesh material following attrition. The soils used for attrition were the -4/+100 material of Composites 1015 and 1016. The head sample for attrition on Composite 1015 was formed by reconstitution of particle-size fractions previously separated by wet sieving (-4 through +100 mesh). The head sample for attrition for Composite 1016 was whole-soil material that was wet sieved to exclude the +4-mesh and the -100-mesh material. Following attrition both samples were wet sieved to exclude the -100-mesh material generated by attrition. This material formed the head samples for chemical extraction.

#### 2.6.2 Chemical Extraction

For chemical extraction, a cylindrical reaction kettle was closed with a 4-neck cover and fitted with a reflux condenser, centigrade thermometer, stirrer with paddle, and thermocouple assembly. The stirrer was turned by a high-torque electric motor and its speed monitored every five min by an optical tachometer. The thermocouple assembly consisted of a ironconstantan thermocouple sealed in a Teflon tube to protect it from acid corrosion. Heat was

provided by a heating mantle whose temperature was monitored and controlled by a Glas-Col Digitrol II that was connected to the thermocouple and the variable autotransformer. Visual monitoring of temperature was also performed by observation of the centigrade thermometer.

For each test the extractant (10mL/g of soil) was heated to 90°C while being stirred at a nominal 350 rpm. Approximately 50 g of the sample was introduced to the reaction flask with a spatula over a five-min period to assure complete contact between the reagent and the sample. The sample was extracted while stirring for 60 min. The extracted sample was collected with the aid of a hot extractant-solution wash and separated from the extractant first by one centrifugation step and then by vacuum filtration. The filtercake was washed with three portions of hot extracting solution and three portions of hot water, thoroughly mixing the solid each time and collecting it by centrifugation. The extracted soil was dried at 60°C and analyzed by gamma spectroscopy and for thorium by alpha spectrometry.

#### 3.0 RESULTS AND DISCUSSION

#### 3.1 Particle-Size Distributions

#### 3.1.1 Individual Samples

The cumulative particle-size distributions for the individual samples are shown in Figure 3-1. The distributions fall into three groupings. The most obvious feature of the graph is that the sample from Borehole 3 has a distinctly different distribution from the other six. Over 60% of its weight is contained in particles with less than 400-mesh (0.038 mm) size and proportionally less material in the sand and gravel sizes. As stated in Section 2.3, Borehole 3 is located near the New Jersey State Highway 17 and on the border of the site property, and thus, may have materials incorporated which are not characteristic of the remainder of the site.

Samples from Borehole 4, Borehole 6, and Alt Borehole 10 have fairly similar distributions in the -50-mesh size material; however, Borehole 4 has less gravel and coarse-sand size material than the other two. All three samples have greater than 55% of the material with particle sizes larger than 200 mesh.

Samples from Alt Boreholes 5, 7, and 8 have similar distributions in the -100-mesh sizes with Alt Borehole 7 having noticeably more sand and gravel-size material. For each sample, more than 60% of the material is larger than 200 mesh.

All samples, with the exception of that of Borehole 3, have greater than 55% of their material with particle sizes greater than 200 mesh and, while the individual distributions are dissimilar in the respects described above, from the prospective of particle-size separation this data, considered alone, indicates potential success.

As described above, Section 2.3, the individual particle-size distributions were considered in the make-up of the composite treatability samples.

#### 3.1.2 Composite 1015

The cumulative and fractional particle-size distributions for Composite 1015 are shown in Figure 3-2. This figure also shows the cumulative particle-size distribution that was predicted from the distributions of the individual samples forming this composite. The particle-size distribution predicted from the weighted compositing was achieved to within a few percent at



FIGURE 5-2: CUMULA TIVE AND FRACTIONAL PARTICLE-SIZE DISTRIBUTION COMPOSITE 1015



🐨 Luta Will (Predicted) 🖞 Cura, Will (Actual) 📕 bract, Will (Actual)

all size fractions, as shown by the compliance of the predicted and actual cumulative distributions in Figure 3-2.

Approximately 64 percent of the material in Composite 1015 has particle sizes greater than 200 mesh (0.075 mm) and 52 percent greater than 100 mesh (0.150 mm). This amount of material available for particle-size separation in the +100-mesh particles does not preclude consideration of the use of soil washing as a treatment technology. The degree of success of this alternative is, of course, dependent upon the radionuclide concentrations relative to cleanup goals in the various size fractions, which are discussed in the following section.

#### 3.1.3 Composite 1016

The cumulative and fractional particle-size distributions for Composite 1016 are shown in Figure 3-3. The particle-size distribution that was predicted from the distributions of the individual samples which comprise this composite is compared to that determined from wet sieving the composited sample. The particle-size distribution predicted was achieved to within a few percent at all size fractions, as shown by the compliance of the predicted and actual distributions in Figure 3-3.

Approximately 60 percent of the material in Composite 1015 has particle sizes greater than 200 mesh (0.075 mm) and 47 percent greater than 100 mesh (0.038 mm).

The cumulative and fractional particle-size distributions for Composite 1016 are shown and compared to those of Composite 1015 in Figure 3-4. Compared to Composite 1016, Composite 1015 has discernible less material in the +4- and -400-mesh sizes, somewhat more in the +8- to +100-mesh sizes, and approximately the same amount in the 200 and 400-size fractions. Composite 1016, because it has less material in the +100-mesh sizes than Composite 1015, is less promising from the standpoint of remediation by particle-size separation. However, with 47% in the +100-mesh sizes, it could still be economically feasible to apply such technology. This is; however, without regard to the radionuclide concentrations which follow.

#### 3.2 Radionuclide Distributions

The concentrations of Ra-226, Ra-228, Th-230, Th-232, and U-238 in the individual and composite whole soils are summarized in Table 3-1.

#### FEGURE 3.5: COMULATIVE AND FRACTIONAL PARTICLE-SIZE DISTRIBUTION COMPOSISE 1016



FIGURE 3-4: COMPARISON OF PARTICLE-SIZE DISTRIBUTIONS OF MAYWOOD COMPOSITES 1015 AND 1016



- 🕶 Cum, W.S. (1018), (horms, Wr. (1016) 🗮 (ract. Wr. (1015)) 📕 Bract. Mr. (1016)

Location	Whole-Soil Radionuclide Concentration (pCi/g)				
	Ra-226	Ra-228	Th-230	Th-232	U-238
Borehole 3	3.5	24.7	4.4	22.6	5.9
Borehole 4	2.2	19.1	3.5	19.6	3.5
Alt Borehole 5	9.7	60.0	10.6	63.6	2.7
Alt Borehole 7	6.5	40.4	4.1	22.1	3.6
Alt Borehole 8	5.2	47.4	7.4	46.6	2.7
Composite 1015	5.2	33.6	4.7	.28.5	3.5
Borehole 6	237	1820	276	1262	89.0
Alt Borehole 10	44.2	228	49.8	338	77.5
Composite 1016	204	1440	159	1088	72.0

#### Table 3-1: Radionuclide Concentrations in Whole Soils

#### 3.2.1 Individual Samples

Figure 3-5 contains the Ra-226 (Pb-214), U-238, and Th-232 data obtained on the whole soils of Boreholes 3, 4, Alt 5, Alt 7, and Alt 8. Figure 3-6 gives this information for Boreholes 6 and Alt 10. Also shown in Figures 3-5 and 3-6 is the Total Activity Ratio (TAR), which is defined as:

$$TAR = \left(\frac{Ra - 226 + Th - 232}{5}\right) + \left(\frac{U - 238}{50}\right)$$

where Ra-226, Th-232, and U-238 are the concentrations of the respective radionuclides in pCi/g.

The radioanalytical data for the individual samples for all tests are given in Tables A-1 through A-7 of Appendix A. Th-232 is the most abundant radionuclide of the three considered in the Maywood cleanup criteria. This is true for all the samples considered in this study and it accounts for an average of 85 percent of the TAR calculated for the seven individual samples.

Samples from Borehole 6 and Alt Borehole 10 contain distinctly higher concentrations of all radionuclides than the remaining five. Boreholes 3 and 4 contain approximately equal concentration of both Ra-226 (Pb-214) and Th-232 (Figure 3-5). Samples from Alt Boreholes 5, 7, and 8 have relatively equal concentrations of Ra-226. However, Alt Borehole 5 has a



FUULESSE MAN 2000 2000 LESCEL CONCENTRATIONS BOREHOLDS 3, 4, ALT 5, AUL 7, and ALT 8

题 Ra 226 (Pb-244) 董 (h 232 圓1( 238 麗Tossel Act. Ra: 6

FIGURE 3-6: MAYWOOD WHOLE SOLL CONCENTRATIONS BOREHOLES 6 and ALL ID



Th-232 concentration almost twice that of Alt Borehole 7 and one and one-half times that of Alt 8.

Figure 3-7 shows the TAR for the oversized material as a function of particle size for Boreholes 3, 4, Alt 5, Alt 7, and Alt 8. The total activity of Alt Borehole 5 is the greatest and increases rather rapidly as the particle size decreases from the +4 to the +8 and +16 sizes. The TAR then remains relatively constant from the +16 through the +400 sizes. Borehole 4 has the lowest TAR and remains constant for decreasing particle sizes through 400 mesh. Samples from Alt Boreholes 7 and 8 have slightly increasing TARs through 100 mesh where the TAR increases more dramatically with decreasing size. Borehole 3 radioactivity increases slightly through 100 mesh and more rapidly through -400 mesh. The TAR is at or below one only for Borehole 4 through 400 mesh and Alt 7 to 50 mesh.

Figure 3-8 gives the TAR values for the oversize material for samples from Boreholes 6 and Alt 10. For both samples the total activity increases steadily with decreasing particle size. The Th-232 concentrations for Borehole 6 are roughly a factor of 10 greater than those of Alt 10 for all particle sizes. The TAR is less than one only for Alt Borehole 10 for particle sizes of +8 mesh.

#### 3.2.2 Composite 1015

The radioanalytical data for all tests performed on Composite 1015 are listed in Table A-8. Composite 1015 whole soil has concentrations (mean of two determinations) of 5.2, 28.5, and 3.5 pCi/g for Ra-226, Th-232, and U-238, respectively. The resulting TAR is 6.8. Figure 3-9 shows the concentrations of the significant radionuclides and the equilibrium of the U-238 and Th-232 decay series. The values indicate that the U-238 and Th-232 concentrations are depressed in their respective series.

Figure 3-10 shows the Ra-226, Th-232, and U-238 in the sieve sizes of Composite 1015. Th-232 has the greatest concentration in all size fractions. The U-238 values are predicted based on the uranium concentrations in the individual whole-soil samples and the amount of each sample forming the composite. Uranium analyses were not performed on all of the size fractions for this composite because U-238 concentrations predicted as above indicated that no size fraction would have a concentration greater than about 20% of the 50 pCi/g target value.

In Figure 3-11 the oversize concentrations (the concentration of a radionuclide in all material with particle sizes equal to and greater than a given size) are shown for Ra-226 and Th-232 and for the sum of the Ra-226 and Th-232. Because the uranium concentration is low, it



♦ BOREHOLE 2 → BOREHOLE 4 + BOREHOLE ALL 5 米 BOREHOLE ALL 7 ■ BOREHOLE ALL 8

FIGURE 3-8: RADIOACTINITY OF OVERSIZE MATERIAL BY PARTICLE SIZE BOREBOLES 6 AND ALL 10



22


[版10-238 #Th-230 #Ra-226 (Pb-244) 题Th-232 题Ra-228 测Th-228

FIGURE 3-10. RADIONUCLIDE CONCENTRATION IN PARTICLE SIZES COMPOSITE 1015





LOD PREDICTS - EROM (SEPTEMORAL SAMPLE)

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contributes little to the TAR. Thus, the sum of the Ra-226 and Th-232 concentrations equaling five is practically equivalent to a TAR of one.

The oversize Ra-226 concentration does not exceed 5 pCi/g until the -400 mesh fraction. The Th-232 oversize concentration exceeds 5 pCi/g for material over 30 mesh. The Ra-226 concentration in the oversize material never reaches 15 and that of the Th-232 exceeds 15 between 100 and 200 mesh. The sum of the two oversize concentrations is 5 pCi/g at the +8-mesh size; however, it is 7.24 at 100 mesh (TAR = 1.45).

Thus, particle-size separation alone will not achieve cleanup to a TAR of one for any significant quantity of material in Composite 1015. However, these results suggest that particle-size separation in conjunction with other physical separation techniques could be effective in achieving site cleanup goals. With only slight additional removal support from processes complementary to particle-size separation, there is potential to recover over 50% of the soil at levels below site cleanup goals.

## 3.2.3 Composite 1016

The radioanalytical data for all tests performed on Composite 1016 are listed in Table A-9. Composite 1016 whole soil has concentrations (mean of two determinations) of 203, 1247, and 82.5 pCi/g for Ra-226, Th-232, and U-238, respectively. The resulting TAR is 292. Figure 3-12 shows the concentrations of the significant radionuclides and the equilibrium of the U-238 and Th-232 decay series. Th-232 and U-238 are significantly deficient in their respective series.

In Figure 3-13 the oversize concentrations are shown for Ra-226 and Th-232 (predicted) and for the sum of the Ra-226 and Th-232. The Th-232 concentrations were not determined for all size fractions of Composite 1016. The values used are those predicted from the analyses of the individual samples making up this composite and the amount of each included in the composite. The oversize Ra-226 concentration is 27.8 pCi/g in the +4 material and increases to 56.4 in the +400 sizes. The Th-232 oversize concentrations are predicted at 25 pCi/g in the +4, 137 pCi/g in the +200, and is over 200 pCi/g in the +400- mesh material. The sum of the two oversize concentrations is 53 pCi/g at the +4-mesh size, increasing to 273 pCi/g in the +400 material.

Particle-size separation alone will not be effective in achieving any of the site cleanup goals, including 15 pCi/g, for either Ra-226 or Th-232 for Composite 1016.



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→ CUMULATIVE WEIGHT (%) 非Ra 226 (Pb 214) 参 th-232 \* Ra 226 + Th-232

FIGURE 3-12. FOUR BRIUM OF RADIONUCTIDES IN COMPOSITE 1046.





#### FIGURE 3: 5: CUMULATIVE WEIGHT AND OVERSIZE ACTIVITY COMPOSED: 1036

## 3.3 Petrographic Study

#### 3.3.1 <u>Composite 1015</u>

Composite 1015 was characterized by petrographic analysis to collect preliminary information on the material and mineral composition of the soil particles. Comparing and contrasting the physical properties of the particles could provide an indication of the potential for remediation of this radioactive soil by mineral processing technology. This information is very useful in directing the remainder of the characterization study.

The mineral and material compositions of the soil fractions are listed by categories in Tables 3-2 and 3-3. The groups categorized include rock particles, homogeneous minerals, slag materials, industrial thorium substance, woody organic particles, and miscellaneous building materials and other minor constituents. Each of these groups is described in the following paragraphs in terms of their abundance in the size fractions, physical properties, and potential contribution to the radioactivity of the soil.

## 3.3.1.1 Rock Particles

The rock particles occur in the gravel and coarse sand-size fractions and consist of sandstone/siltstone, igneous rocks, and black bituminous coal (Table 3-2).

The sandstone and siltstone are reddish brown in color, fine to medium grained, subrounded, rough to smooth surface, structurally strong to friable, and comprised predominantly of quartz clasts and minor clay and hematite matrix. These particles comprise 25 percent of the gravel and 2 - 12 percent of the coarse sand-size fractions. They are derived from the local New Jersey bedrock with radioactivity at normal background levels.

The igneous rocks are granite and basalt particles that comprise one percent of the gravel-size materials and but trace amounts of coarse sand-size fractions. These particles are subangular, tough, dense, and durable. The radioactivity in these materials is essentially that of background levels.

Black bituminous coal particles comprises 3 percent of the gravel materials, 4 - 10 percent of coarse and median sand-size fractions and trace to 2 percent of fine sand and coarse silt-size fractions. This material is light weight, subangular shape, smooth surfaced, and durable. The bituminous coal in the United States averages 3 ppm uranium and 3.12 ppm thorium (ROG64). The coal therefore contributes relatively minor amounts of radioactivity, only

slightly higher perhaps, than average background radioactivity.

# 3.3.1.2 Minerals

Natural minerals in the soil fractions consist of quartz, feldspar, and detrital heavy minerals. Quartz and minor quartzite particles, included with the quartz, is the dominant material in the soil fractions (Table 3-2). The quartz comprises 2 percent of the gravel, 14 - 26 percent of the coarse sand, 50 - 54 percent of the median sand, 57 - 60 percent of fine sand, and 55 percent of the coarse silt-size fractions. The quartz particles are hard, dense, and durable and free of radioactivity or at background levels for the region.

Table 3-2:	Material	Composition	and	Weight Perce	nt of S	Soil	Fractions	from	Composite	1015
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	Gravel			Sa	nd			Silt	
Sieve Size	+4 8	8	16	30	50	100	200	400	Average
Weigh % <sup>1</sup>	17	8	7	8	14	19	16	11	Percent
			Perc	ent Composi	tion		•••••		• • • • •
			ROC	CK PARTICL	ES				
Sandstone/Siltstone	25	12	5	2	Т				6
Igneous Rocks	1	Ţ	T	Т					Т
Coal	3	10	10	5	4	2	2	Т	4
			<u>L</u>	MINERALS		•			
Quartz	2	14	26	54	50	57	60	55	45
Feldspar			1	1 .	2	5	4	8	3
Transp. H.M. <sup>2</sup>				Т	Т	2	3	2	1
			N	MATERIALS					
White Th Substance <sup>3</sup>		1	2	3	3	3	4	4	3
White Glassy Slag		1	6	5	5	6	5	6	4 -
Magnetic Slag	21	31	25	16	7	2	2	2	7
Dense Slag	20	12	10	6	10	8	15	25	13
Clinker Slag	17	11	15	12	18	15	5	2	12
Woody Materials	1	2	Ť	Т	1	T	T	Т	T
Miscellaneous <sup>4</sup>	10	7	Т	Т	1	Т	T		2

T = Trace amounts, less than 0.05 percent.

1. Weight percent for materials greater than 400 sieve size (0.038 mm).

2. Transparent heavy minerals greater than 3.0 grain density. Species listed in Table 3-3.

3. White, fine grained, friable, "clay-like", anthropogenic thorium substance.

4. Miscellaneous red brick, tan concrete, ceramic materials, glass, metal, and minor other.

# Table 3-3: Transparent Heavy Minerals in Fine Sand and Coarse Silt Fractions of Composite 1015

Sieve Size	-50/+100	-100/+200	-200/+400
Weight %'	2	3	2
	Percent C	omposition	<u>م</u>
Amphibole Group	56	27	34
Garnet Group	27	13	22
Epidote Group	10	5	3
Zircon	2	8	15
Monazite	5	46	24
Rutile		Т	1
Other <sup>2</sup>	Т	1	1

T = Trace Amounts (less than 0.05%).

1. Weight percent of transparent heavy minerals greater than 3.0 grain density in the sieve size fractions. Other heavy materials included in Table 3-2 listing, e.g., opaque minerals with magnetic materials, white Th substance, and other slag materials.

2. Other includes trace amounts of kyanite, hypersthene, tourmaline, and chlorite.

Feldspar comprises from 1 - 4 percent of the medium to fine sand and 8 percent of the coarse silt-size fractions. The feldspar is grey to pink in color, subangular, hard, dense, and durable. The radioactivity associated with this type of material is essentially at background levels.

Transparent heavy minerals occur in the fine sand and coarse silt-size soil fractions, ranging from 2 to 3 percent in these fractions. The transparent heavy minerals average approximately 1 percent of the soil material tested (Table 3-2). Opaque heavy minerals are relatively minor and included with other categories of materials. The transparent heavy minerals are listed in Table 3-3. The amphibole group, comprised in the main by hornblende, comprises 27 to 57 percent of these fractions and the garnet group comprises 13 - 27 percent of the transparent heavy minerals (see Figure B-1). The remainder of these materials consist of the epidote group (3 - 10 percent), zircon (2 - 15 percent), monazite (5 - 46 percent), rutile (up to 1 percent) and minor kyanite, hypersthene, tourmaline, and chlorite (trace - 1 percent). All these minerals are hard, dense, and durable and have persisted in nature because of their high resistance to weathering and abrasion. Of these minerals, monazite and zircon contain naturally occurring radioactive materials (see Figure B-1).

Monazite is the common ore mineral of thorium, which on the average contains from 3 to 10 percent thorium oxide and from 0.2 to 0.6 percent uranium (ADA59). Monazite in the -50/+100, -100/+200, and -200/+400 sieve-size fractions, contains respectively, 5, 46, and 24 percent of the transparent mineral fractions (Table 3-3). When these fractions of monazite are

calculated to representation in the total sample, they are respectively 0.01 percent, 0.24 percent, and 0.03 percent. This abundance of monazite in the soil could account for a major portion of the thorium contamination in this composite.

Zircon is the other natural mineral that contributes to the enhanced radioactivity in this soil sample. Zircon can contain up to 4 percent uranium or thorium (ADA59). Uranium-238 is the more common radionuclide in zircon. The zircon in the -50/+100, -100/+200, and -200/+400 sieve fractions is, respectively, 2, 8, and 15 percent of the transparent heavy mineral fraction. In respect to total sample, this is calculated as 0.005, 0.04, and 0.02 percent for zircon in the total sample.

## 3.3.1.3 Anthropogenic Materials

A white, fine grained, "clay-like" friable, thorium substance comprises 3 percent of the soil material tested (Table 3-2). This is believed to be a radioactive, anthropogenic material, produced in the extraction of thorium from ore. This material is absent in gravel as discrete particles but may occur in voids or pore space available in porous slag materials. It comprises from 1 to 4 percent of the soil fractions from coarse sand to coarse silt-size with increasing abundance in the smaller soil fractions (see Figure B-2). The particles are well represented in the heavy fraction, greater than 3.0 grain density and is believed to be a thorium orthophosphate material and appears to correlate with previous investigations (FIN88). The radioactivity in these particles are believed to be a major part of the radioactivity in the soil sample.

White, light weight, porous, angular to subrounded and nodular shaped, generally structural sound, glassy slag comprises 4 percent of the soil sample (Table 3-2). This slag is distinctive from the other categories of slag in this soil in its' creamy white to light tan color, glassy, and high luster appearance. Although absent in discrete particles in the gravel-sized materials, it may occur in voids or attached to other slag particles. Because of its' unique appearance, it is a suspicious candidate for containing minor amounts of thorium, although its low density suggests otherwise.

Magnetic materials comprise 7 percent of the soil sample (Table 3-2). The magnetic particles are most abundant in the gravel and coarse sand-size fractions (16 - 31 percent) with less representation in the smaller sieve sizes (2 - 7 percent). The magnetic particles are predominantly black to reddish colored, metallic lustered, flat and angular shaped, slag materials and minor amounts of black, subrounded, dense, magnetite and grey to black, dense, metallic, spherical balls (see Figure B-3A).

Solid industrial slag, black to reddish colored, flat to angular shaped, dull to metallic lustered, comprises 13 percent of the soil sample, and tan clinkers, highly porous, subrounded to nodular shaped, coal-fired, comprise about 12 percent of the soil sample (Table 3-2). The solid slag may be related to the thorium industrial process and contain residual radionuclides. The porous, clinker slag is believed to be residual ash from coal-fired material and probably has concentrations of uranium oxide from coal as part of the clinker. Although higher than background radiation levels, the clinker slag is probably but a minor contribution to the radioactivity of the soil sample. Slag materials in this sample are depicted in the photomicrograph of Figure B-3B.

Miscellaneous and woody, organic materials comprise, respectively two percent and trace amounts of the soil sample (Table 3-2). The miscellaneous materials occur predominantly in the gravel-size (10 percent) and coarse sand-size (7 percent) fractions and consist of construction materials including, in order of abundance, red brick fragments, grey concrete fragments, and minor ceramic material, glass, and metal fragments. Wood and plant material comprise 1 - 2 percent of the gravel and coarse sand-size fractions and from trace amounts to one percent of the finer sieve sizes. All this material is free of radioactivity beyond background levels.

# 3.3.1.4 Summary

Composite 1015 is comprised of a variety of natural and anthropogenic materials. Natural rock and minerals with radioactivity at background levels constitute more than half of the particles (54 percent). Coal, woody materials, and miscellaneous construction materials average about 7 percent, and their radionuclide concentration is at or very slightly above the normal background levels.

Radioactive materials known and suspected to contain radioactivity are believed to be present in about one-third of the sample. Known natural radioactive minerals are monazite and zircon that occur only in the fine sand and coarse silt-size fractions. The monazite averages about 0.028 percent and zircon about 0.07 percent of the soil sample. A component that potentially could be a higher contributor to the radioactivity in the soil is a white, clay-like, anthropogenic thorium substance that comprises 3 percent of the soil sample. This material is believed to be a thorium orthophosphate and has a specific gravity generally greater than 3.0. Coal-fired, clinker slag comprising about 12 percent of the sample, contributes residue ash of uranium and/or thorium that produces radioactivity above background levels. Suspect radioactive materials are believed to be contained in white glassy slag comprising 4 percent of the soil. All these minerals and materials which are believed to be the major contributors to

the elevated radionuclide levels in this sample have specific gravities greater than 3.0 and are candidates for removal by density separation processes. Further definition of the amount and radionuclide concentration of the dense (heavy) materials is given in Section 3.3.3. A more detailed study specifically related to treatment process removal is given in Chapter 5.

Solid magnetic and nonmagnetic slag comprising, respectively, 7 and 13 percent of the soil sample, also might contain radioactivity above background levels. This material is suspected to be associated with the industrial manufacture of thorium and may possibly contain radioactive residues. Further definition of the amount and radionuclide concentration of the magnetic and non-magnetic materials is given in Section 3.3.3.

# 3.3.2 Composite 1016

Composite 1016 was characterized by petrographic analysis to collect preliminary information on the material and mineral composition of the soil particles. Comparing and contrasting the physical properties of the particles would provide the first indication of the potential for remediation potential of this radioactive soil by mineral processing technology. This information is most useful in providing direction for the remainder of the characterization study.

The mineral and material composition of the soil fractions is listed by categories in Tables 3-4 and 3-4. The minerals and materials groups categorized include rock particles, homogeneous minerals, slag materials, industrial thorium substance, woody organic particles, and miscellaneous construction materials and other minor substances. Each of these categories of materials is described in the following paragraphs in terms of their abundance in the size fractions, their physical properties, and potential contribution to the radioactivity of the soil.

## 3.3.2.1 Rock Particles

The rock particles consist of sandstone and siltstone, igneous rocks, and coal (Table 3-4). The sandstone and siltstone, comprising 4 percent of the sample, are grey to reddish brown in color, fine to medium grained, subrounded, rough to smooth surfaced, and friable to structurally strong. The coarser sandstone is more abundant than the finer, smoother surfaced, siltstone. Mineral composition is predominantly quartz, and minor clay and hematite cement. These rock particles comprise 14 percent of the gravel and 2 - 6 percent of the coarse sandsize fractions. The sandstone and siltstone are from local bedrock, and the radioactivity of this material is at background levels for the region.

The igneous rocks are predominantly basalt with minor amounts of granite and hornblende gneiss. These grey to black colored, angular to rounded, hard, dense, durable particles comprise 1 percent of the soil fractions tested. The radioactivity in these particles is essentially at background levels.

Black bituminous coal particles comprise 3 percent of the soil materials tested. The coal particles are black in color with a high virtuous luster, light weight, subangular in shape, smooth surfaced, and durable. The bituminous coal in the United States averages 3 ppm uranium and 3.12 ppm thorium (ROG64). The coal constitutes a minor source of radioactivity above that of background.

# 3.3.2.2 Minerals

Natural minerals in the soil fractions consist of quartz, feldspar, and detrital heavy minerals.

	Gravel			Sa	nd	_ · · ·		Silt	
Sieve Size	+4	8	16	30	50	100	200	400	Average
Weigh % <sup>1</sup>	23	6	6	7	13	16	18	11	Percent
	_1		Perc	ent Composit	ion				
			ROC	K PARTICL	ES				
Sandstone/Siltstone	14	6	6	2					4
Igneous Rocks	4	2	1	Т			++	***	1
Coal	4	5	3	3	2	5	1	Т	3
			1	MINERALS					
Quartz	1	11	25	41	54	40	30	45	28
Feldspar		Т	Т	T	2	5	7	6	3
Transp. H.M. <sup>2</sup>				T	Т	6	21	7	6
	,,,,,,, _		Ņ	MATERIALS					
White Th Substance <sup>3</sup>	5	9	5	7	9	7	8	12	8
White Glassy Slag		10	7	6	6	7	6	3	5
Magnetic Slag	4	14	22	13	7	4	4	3	7
Dense Slag	12	17	14	14	11	17	13	19	14
Clinker Slag	12	21	7	14	9	9	10	5	10
Woody Materials	3	4	3	Т	Т	Ť	T	I	1
Miscellancous <sup>4</sup>	41	11	7	Т	Т	Т	T		10

Table 3-4: Material Comp	osition and Weight Perce	nt of Soil Fractions	from Composite 1016
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T = Trace amounts, less than 0.05 percent.

1. Weight percent for materials greater than 400 sieve size (0.038 mm).

2. Transparent heavy minerals greater than 3.0 grain density. Species listed in Table 3-5.

3. White, fine grained, friable, "clay-like", anthropogenic thorium substance.

4. Miscellaneous grey concrete, red brick, black asphaltic road metal, glass, metal fragments, ceramic material, and minor other.

Sieve Size	-50/+100	-100/+200	-200/+400
Weight % <sup>1</sup>	6	21	7
	Percent C	omposition	4
Amphibole Group	18	2	6
Garnet Group	4	4	1
Epidote Group	7	6	5
Zircon	18	16	12
Monazite	50	72	31
Rutile	1	Т	1
Other <sup>2</sup>	2	Т	1

# Table 3-5:Transparent Heavy Minerals in Fine Sand and Coarse Silt Fractions of<br/>Composite 1016

T = Trace Amounts (less than 0.05%).

1. Weight percent of transparent heavy minerals greater than 3.0 grain density in the sieve size fractions indicated.

2. Other transparent heavy minerals include kyanite, hypersthene, tourmaline, and chlorite.

Quartz and minor quartzite particles are the dominant materials in the soil sample (Table 3-4). The quartz comprises 1 percent of the gravel, 11 - 54 percent of the sand-size fractions, and 45 percent of the coarse silt fraction. The quartz particles are hard, dense, and durable and free of radioactivity above background levels.

Feldspar comprises approximately 3 percent of the soil fractions. The feldspar occurs in the fine sand and coarse silt fractions in the range from trace amounts to 5 percent. The feldspar is pink to grey in color, essentially rectangular to subrounded in shape, and generally hard, dense, and durable. The radioactivity in this material is generally in the background range.

Transparent heavy minerals (greater than 3.0 grain density) occur in the fine sand and coarse silt-size fractions. The percent of the transparent heavy minerals for the -50/+100, -100/+200, and -200/+400 sieve size is, respectively, 6, 21, and 7 percent (Table 3-4). The mineral species present in these size fractions are listed in Table 3-5. The monazite and zircon radioactive minerals are the dominant minerals with monazite 31 - 72 percent and zircon 12 - 18 percent. Other minerals include the amphibole group (2 - 18 percent), garnet group (1 - 4 percent), epidote group (5 - 7 percent), rutile (trace - 1 percent), and minor hypersthene, kyanite, chlorite, and tourmaline (trace - 2 percent). All these detrital, transparent heavy minerals are hard, dense, and very durable, having persisted in the environment for a very long time. It is apparent that the anomalous amounts of monazite and zircon are due to their presence as ore minerals for thorium extraction at an industrial site (see Figure B-4).

category of material (see Figure B-6B). The black, solid, metallic slag could contain some radioactivity if it is waste produced during the production of thorium from ore.

Solid slag, black to reddish colored, flat to angular shaped, dull to metallic lustered, comprises 14 percent of the soil materials tested. Tan colored slag, nodular to subrounded shape, highly porous, coal-fired clinker, constitutes 10 percent of the soil (Table 3-4). The slags are relatively uniformly distributed in all size fractions. The clinker slag contains residue ash of uranium from the burning of coal (ROG64). This material contributes to radiation levels above background, however, the clinker slag is not a strong contributor to the radiation levels of this soil sample.

Miscellaneous and woody, organic materials comprise respectively 10 and 1 percent of the soil sample, respectively (Table 3-4). The miscellaneous materials are predominantly construction materials comprised of concrete, brick, asphaltic road metal, glass, and minor metal particles. These materials occur in significant amounts only in the gravel and coarse sand-size fractions. Wood and plant particles are also abundant only in gravel and coarse sand-size fractions (Table 3-4). None of this material produces radioactivity above background levels.

#### 3.3.2.4 Summary

The composite soil sample, 1016 from Maywood is comprised of a variety of natural and anthropogenic materials. The natural rock and minerals comprise more than 45 percent of the soil and anthropogenic slags, thorium substances, and miscellaneous materials; woody materials constitute the remaining soil particles.

Radioactive monazite and zircon are natural minerals that are present as significant sources of radioactivity. Both these minerals are found only in the fine sand and coarse silt-size fractions; however, the monazite comprises 3.33 percent and zircon 0.82 percent of the soil sample. The concentration level of these radioactive minerals suggests their presence is derived form ore minerals used to obtain thorium.

A white anthropogenic thorium substance comprises 8 percent of the soil sample tested. This material is probably an orthophosphate derived from the industrial process to obtain thorium metal. It is probably one of the main contributors of radioactivity in the soil sample.

Radioactivity is believed to be present in some of the slag materials that may be associated with the industrial process used to manufacture thorium metal. Individual fractions of white

glassy slag comprising 5 percent of the soil and solid magnetic and nonmagnetic slag, comprising 21 percent of the soil sample, are possibly important sources of the radioactivity in the soil.

Both Composite 1015 and 1016 consist of natural and anthropogenic materials with natural rock and minerals comprising 54 and 45 percents, respectively. Both samples contain significant amounts of monazite and zircon in the fine sand and coarse silt-size fractions. Composite 1015 has 0.028 and 0.07 percent monazite and zircon, respectively, and Composite 1016 has 3.33 and 0.82 percent. A white anthropogenic thorium substance, believed to be orthophosphate from the industrial process, is present in both samples at 3 percent in 1015 and eight percent in 1016. Both composites contain slag materials which may be from the manufacturing process and possibly contain radioactivity above background levels.

## 3.3.3 Density and Magnetic Separation

To determine the potential for contaminant/host soil separation on the bases of ferromagnetism and density, scoping tests were performed as described in Section 2.3. The -50/+100, -100/+200, and the -200/+400 fractions of Composites 1015 and 1016 were subjected to a sink-float density separation at a specific gravity of 2.89; and the sink (heavy minerals) and the floats (light minerals) were collected, dried, and weighed. The heavy minerals were scanned with a hand-held magnet to remove ferromagnetic material and both fractions, magnetic and non-magnetic, were weighed. After combining the heavy minerals from each of the size fractions, a portion of each of the light, magnetic, and non-magnetic fractions was analyzed by alpha spectrometry for uranium and thorium. Insufficient material was available for gamma (Ra-228) analysis. The results of these analyses are presented in Table 3-6, below.

			Comp	osite 1015					
	HEAVY N	HEAVY MINERALS		LIGHTS		MAGNETIC		NON-MAGNETIC	
SIZE	% of Fraction	% of Whole Soil	% of Fraction	% of Whole Soil	% of Heavy Minerals	% of Whole Soil	% of Heavy Minerals	% of Whole Soil	
-50/+100	2.80	0.38	97.20	13.29	49	0.19	51	0.20	
-100/+200	4.29	0.49	95.71	10.97	25.1	0.04	79.9	0.12	
-200/+400	1.93	0.15	98.07	7.87	27.5	0.07	72.5	0.18	
U-238 (pCi/g)	121.7	± 15.3	1.49	± 0.55	4.75 ±	£ 1.05	175 ±	20.7	
Th-232 (pCi/g)	807.9	± 57.3	9.9	9 ± 2.9	37.1 =	± 11.0	1196	± 88	

Table 3-6:Heavy Minerals and Magnetic Materials in the -50/+400 Materials from<br/>Composites 1015 and 1016

			Compo	site 1016				
-50/+100-	13.45	1.87	86.55	12.00	15.8	0.29	84.2	1.57
-100/+200	37.77	4.33	62.23	7.13	5.8	0.25	84.2	4.08
-200/+400	12.7	1.02	87.3	7.00	10.6	0.11	89.4	0.91
U-238 (pCi/g)	117 :	± 17.3	17.6	± 2.52	53.3 :	± 6.53	135 ±	. 22.4
Th-232 (pCi/g)	918	± 77	198	± 29	561	± 54	1153	± 89

Table 3-6:Heavy Minerals and Magnetic Materials in the -50/+400 Materials from<br/>Composites 1015 and 1016 (continued)

Following the separation of the magnetic from the heavy mineral fraction, the magnetic material was separated from the more coarse fractions, +8 through 50-mesh, with a hand-held magnet. The amounts removed from these coarse fractions are presented in Table 3-7.

Table 3-7:	Magnetic Material in the -4 to +50- Mesh Material from Composites 1015 and
	1016

		Composite 1015			
	MA	GNETIC	NON-MAGNETIC		
SIZE	PERCENT OF FRACTION	PERCENT OF WHOLE SOIL	PERCENT OF FRACTION	PERCENT OF WHOLE SOIL	
-4/+8	30.8	1.77	69.2	3.97	
-8/+16	25.4	1.31	74.6	3.83	
-16/+30	15.9	0.93	84.1	4.93	
-30/+50	7.0	0.67	93.0	8.92	
		Composite 1016			
	26.1	1.50	73.9	4.24	
-8/+16	22.2	1.14	77.8	4.00	
-16/+30	12.5	0.73	87.5	5.13	
-30/+50	7.10	0.68	92.9	8.91	

From Table 3-5 the selective concentration of U-238 and Th-232 in the heavy mineral fractions is obvious, with concentration ratios (pCi/g in heavy minerals/pCi/g in lights) for both radionuclides of approximately 80. While not as dramatic, concentration ratios for the non-magnetic to magnetic of 32 to 37 were obtained.

The results of this scoping study confirm the assumption made during the petrographic study that significant amounts of the radioactivity in Composites 1015 and 1016 are associated with dense (>2.89 g/cc) minerals. The identification of these materials was discussed above. The amounts of these minerals are small, one percent or less of the whole soil; however, the concentration ratios obtained, particularly for the heavy minerals, suggest that density

separation is worthy of further study. The results of the extended characterization of the radioactive mineral associations using SEM/XRF and the additional laboratory studies on the composite samples to evaluate the potential for additional partitioning by specific gravity and magnetic susceptibility are presented in Part II of this report.

3.4 Attrition Study

# 3.4.1 <u>Composite 1015</u>

Figure 3-14 gives the results of the attrition study of Composite 1015 at 60 percent solids. Some benefit was obtained since additional thorium was removed from the -4/+100-mesh material. The Th-232 concentration in the +100 mesh material was reduced from 6.4 to 4.6 pCi/g by attrition. The Ra-226 results indicate no benefication by attrition since the concentrations at all size fractions are the same for the head and the attrited samples.

Due to the nature of the attrition scout testing, the absolute thorium removal in a full scale process is not possible to fully assess. However, the benefit is obviously not large. The vigorous washing procedure (SCA-502) employed prior to the sieving process apparently sufficiently liberates fines such that attrition of the resultant material is not of major benefit. It is not clear if the small amount of fines (5 percent of the attrited material) generated in the attrition study, were the result of fines liberation, surface removal from coarse particles, or coarse particle fracturing.

## 3.4.2 Composite 1016

Figure 3-15 illustrates the attrition results at 70 percent solids for Composite 1016. It should be noted that the Th-232 concentrations for the head sample are those calculated from the concentrations in the two individual samples that comprise this composite and the relative amounts of material combined to form this composite. It is clear from a comparison of both Ra-226 and Th-232 for the oversize concentrations of the head and attrited soil that attrition was beneficial in reducing the radioactivity in the +4/-100 sizes. The reduction for Ra-226 in the +100-mesh oversize concentrations of both radionuclides remain well above target goals following attrition for this composite.

Less than one percent of the fines were generated by the attrition process. The results of the attrition of 1016 at 60 percent solids were comparable to those of the 70 percent solids.





\*\* HEAD SAMPLE COMPLATIVE -| ATTRITION SAMPLE COMPL. 米HEAD-Ro-226 OVERSIZE \* ATTRITION-Ro-226 OVERS ※HEAD-T6-232 OVERSIZE ◆ATTRITION-T6-232 OVERS

FIGURE 3-15: ATTENTION OF COMPOSITE 4946 70 PERCENT SOLDS





The results of attrition testing of both composites indicate that attrition, or some process for liberation of fines, is desirable and should be a component of a treatment scheme. Since the samples subjected to attrition testing had previously been through vigorous washing, it is not possible to independently evaluate the efficacy of attrition for fines liberation.

3.5 Chemical Extraction

# 3.5.1 Composite 1015

The gamma and alpha spectrometry analysis results for the head samples (-4/+100 mesh) and the extracted soils for Composite 1015 for the three extractants are presented in Tables 3-8, 3-9, and 3-10. The results are tabulated for Ra-226, Ra-228, and Th-232. Since the chemical extraction may have affected the radionuclide equilibrium between Ra-226 and its progeny, the use of Pb-214 to determine Ra-226 may be inappropriate. Ra-228 that is determined from its daughter Ac-228 (half life 6.13 hours) is likely a better determinant for the efficiency of chemical extraction. Sufficient time elapsed between extraction and the gamma counts to allow for greater than 96 percent secular equilibrium between Ra-228 and Ac-228.

# CHEMICAL EXTRACTION SCOUT TEST RESULTS COMPOSITE 1015

	Head Sample	Extracted Soil	Removal %	Recovered %
Sample #	1015-19WB	1015-19RB		
Wt. (g-dry)	48.70	47.93		98.4
Radionuclide	pCi/g-dry	pCi/g-dry		
Ra-226 (Pb-214)	1.65	1.63	l de la compañía de la	
Th-232	7.56	2.84	62	
Ra-228	· 9.67	7.31	24	

# Table 3-8: 0.1M EDTA/0.5M Na<sub>2</sub>CO<sub>3</sub> @pH 9.0 for Composite 1015

Table 3-9: 3M HCl followed by 0.22M Sodium Hexametaphosphate for Composite 1015

	Head Sample	Extracted Soil	Removal %	Recovered %
Sample #	1015-20WB	1015-20RB		
Wt. (g-dry)	48.30	45.45		94.1
Radionuclide	pCi/g-dry	pCi/g-dry		
Ra-226	1.94	1.27	35	
Th-232	7.56	1.48	80	
Ra-228	11.96	4.48	63	

Table 3-10: 0.22M Sodium Hexametaphosphate for Composite 1015

	Head Sample	Extracted Soil	Removal %	Recovered %
Sample #	1015-21WB	1015-21RB		
Wt. (g-dry)	48.60	47.92		98.6
Radionuclide	pCi/g-dry	pCi/g-dry		
Ra-226	1.62	1.63		
Th-232	7.56	4.44	41	
Ra-228	10.52	7.78	26	

The most effective extractant for both thorium and radium (Ra-228) was 3M HCl followed by 0.22M sodium hexametaphosphate, with removals of 80 and 63 percent, respectively. The resulting extracted soil concentrations of Ra-226, Ra-228, and Th-232, respectively, were 1.27, 4.48, and 1.48 pCi/g. Only 6 percent of the soil was digested during the extraction.

While not as effective as 3M HCl/sodium hexametaphosphate, 0.1M EDTA/0.5M Na<sub>2</sub>CO<sub>3</sub> removed 62 and 24 percent of the thorium and radium, respectively. The resulting Ra-226, Ra-228, and Th-232 concentrations of the extracted soil were 1.63, 7.31, and 2.84 pCi/g, respectively, which would result in a TAR of less than one. Less than two percent of the soil was solubilized in this extraction.

# 3.5.2 Composite 1016

The gamma and alpha spectrometry analysis results for the head (whole soil) samples and the extracted soils for Composite 1016 for the three extractants are presented in Tables 3-10, 3-11, and 3-12. In addition, an extraction with 3M HNO<sub>3</sub> was performed. The results of this extraction are presented in Table 3-14. The results are tabulated for Ra-226, Ra-228, U-238, and Th-232. The U-238 data for the head sample are taken from the attrition study for 60 percent solids.

# CHEMICAL EXTRACTION SCOUT TEST RESULTS COMPOSITE 1016

	Head Sample	Extracted Soil	Removal %	Recovered %
Sample #	1016-19WB	1016-19RB		
Wt. (g-dry)	47.60	46.18		97.0
Radionuclide	pCi/g-dry	pCi/g-dry		
Ra-226	27.6	16.5	40	
Ra-228	233.8	167.3	28	
Th-232	113.1	112.5		
U-238	12.6	7.1	44	·

# Table 3-11: 0.1M EDTA/0.5M Na<sub>2</sub>CO<sub>3</sub> @pH 9.0 for Composite 1016

Table 3-12: 3M HCl followed by 0.22M Sodium Hexametaphosphate for Composite 1016

	Head Sample	Extracted Soil	Removal %	Recovered %
Sample #	1016-20WB	1016-20RB		
Wt. (g-dry)	47.5	42.3		89.0
Radionuclide	pCi/g-dry	pCi/g-dry		
Ra-226	20.8	9.8	53	
Ra-228	236.7	67.9	71	
Th-232	113.1	43.4	62	
U-238	12.6	4.2	67	

Table 3-13: 0.22M Sodium Hexametaphosphate for Composite 1016

	Head Sample	Extracted Soil	Removal %	Recovered %
Sample #	1016-21WB	1016-21RB		
Wt. (g-dry)	47.5	46.3		97.5
Radionuclide	pCi/g-dry	pCi/g-dry		
Ra-226	19.3	16.6	14	
Ra-228	398.6	328.6	18	
Th-232	113.1	121.5		
U-238	12.6	9.8	23	

	Head Sample	Extracted Soil	Removal %	Recovered %
Sample #	1016-21WC	1016-21RC		
Wt. (g-dry)	47.2	44.42		94.1
Radionuclide	pCi/g-dry	pCi/g-dry		
Ra-226	21.8	8.5	61	
Ra-228	229.2	49.5	78	:
Th-232	113.1	25.7	77	· ·
U-238	12.6	3.4	73	

Table 3-14: 3M HNO<sub>3</sub> for Composite 1016

The most effective extractant for thorium, uranium, and radium (Ra-228) was 3M HNO<sub>3</sub>, with removals of 77, 73, and 78 percent, respectively. The resulting extracted soil concentrations of Ra-226, U-238, and Th-232, respectively, were 8.5, 3.4, and 25.7 pCi/g (TAR = 6.9) Approximately 6 percent of the soil was digested during the extraction.

While not as effective as  $HNO_3$ ; 3M HCl followed by 0.22M sodium hexametaphosphate removed 62, 67, and 71 percent of the thorium, uranium, and radium, respectively. The resulting Ra-226, U-238, and Th-232 concentrations of the extracted soil were 9.8, 4.2, and 43.4 pCi/g (TAR = 10.7), respectively. 0.1M EDTA/0.5M Na<sub>2</sub>CO<sub>3</sub> removed from 28 to 40 percent of the radium but was ineffective in its thorium removal.

The chemical extractions were performed to determine, on a broad scale basis, if previously used extractants might be effective in removal of radium, thorium, and uranium from the composite soils. No optimization of extraction conditions was attempted. Based on these limited results, both 3M HCl/sodium hexametaphosphate and 0.1M EDTA/0.5M Na<sub>2</sub>CO<sub>3</sub> are effective; with the latter extractant having less environmental and aesthetic considerations to overcome in its application to radionuclide removal. Due to resource and time considerations, no further consideration was given in this study to the use of extractants as the primary or as a supportive treatment process.

# PART II:

# EXTENDED CHARACTERIZATION AND PROCESS DESIGN

# 4.0 EXTENDED CHARACTERIZATION - SEM ANALYSES

## 4.1 Background

Physical separation processes are effective only if the particulate contaminants are adequately liberated from the host soil (i.e., present as discrete particles). Therefore, the first series of extended characterization tests were performed to evaluate the liberation behavior of the Maywood soil. These studies were performed using an advanced mineralogical identification system available at Virginia Tech. This system incorporates an automated image analyzer (AIA) together with a variety of spectroscopic techniques including optical petrographic microscopy, scanning electron microscopy (SEM) and energy dispersive x-ray (EDX). The system is totally dedicated to the evaluation of the separation potential of particulate materials and is used extensively to assist in solving separation problems throughout the minerals processing industry. This system allows the rapid determination of the types and relative amounts of each component present in the contaminated soil. The image processing system also allows the degree of association between the various components to be quantitatively established. This work provides a means of determining whether the contaminated components in the soil are sufficiently liberated to be rejected by physical cleaning processes. The findings of this subtask have a major bearing on the development efforts carried out under the remaining tasks since the selective removal of contaminants by physical separation techniques is possible only if the particles are adequately liberated.

The information reported under this subtask includes characterization data from both the Wayne and Maywood sites. This reporting approach was necessary in order to obtain petrographic correlations that were statistically meaningful. These correlations provide tremendous insight into the mode by which radionuclides occur at the FUSRAP sites. This knowledge is critical in the development of a successful remediation scheme for the FUSRAP soils.

# 4.2 Experimental Procedure

Characterization of three composite FUSRAP soil samples supplied by SC&A was performed by scanning electron microscopy/automated image analysis. One composite soil sample (Composite 1017) was obtained from the Wayne site, while two composite samples (Composite 1015 and Composite 1016) were obtained from the Maywood site. Mounts of individual size fractions were prepared by embedding particles in epoxy resin. Cross-sections were then cut from these mounts and remounted thus eliminating sample bias by particle settling. The final mounts were then polished and sputter-coated with gold before being placed in the SEM. The polished

samples were initially analyzed by electron backscatter imaging and energy dispersive X-ray spectra. This allowed for the identification of the various components of the soils and for the determination of gray-level intensities generated by the backscattered electron signal coming from these components.

It was determined that the samples could be divided into three types of material based on graylevel intensity. These three types included (i) a low intensity material consisting of predominantly quartz as well as other silicate minerals, (ii) an intermediate intensity material consisting of a porous slag, and (iii) a high intensity group made up of heavy minerals such as ilmenite, iron oxide, zircon, rutile, and monazite. Routine analyses of the samples were performed by transferring backscattered electron images to the image analyzer, which had been programmed to identify the three gray-level ranges. The image analyzer then measured the cross-sectional area of each particle and the area of each of the three-gray level ranges, if present in the particle. These data were then used to calculate the amount of each type of material in the sample and to classify the particles in terms of their composition.

Size fractions analyzed for Maywood Composite 1015 and Composite 1016 include 8x16, 16x30, 30x50, 50x100, 100x200, and 200x400 mesh. For the Wayne (1017) sample, the 16x30, 50x100, and 200x400 mesh size fractions were analyzed. Approximately 1500 to 2000 particles were counted for each size fraction.

The initial X-ray analysis of the soils indicated that one mineral phase in particular contained considerable amounts of thorium. Although the chemical composition of this phase is variable, it appears to be monazite-like in that it generally contains rare earth elements and phosphorous. Therefore, to determine the quantity of this phase in the soils, point counts were performed on the heavy mineral component by using X-ray spectra for phase identification. Given the percentage of "monazite" in the heavy minerals and the amount of heavy minerals in the total sample, the area percent "monazite" in the sample was calculated. In addition to the "monazite", minor amounts of a calcium-thorium-phosphorous phase, and thorium oxide were also found to be present. Therefore, the sum of these three phases provides an estimate of the total amount of radioactive material in the sample.

Although no radioactive components were observed in any minerals other than those that are normally classified as heavy mineral sands, several SEM photographs were taken to provide some examples of how the "monazite" occurs in association with other non-contaminated materials. Figure 4-1 is a photograph taken of the Composite 1016 sample for the 50x100-mesh

# Maywood Composite 1016 (50x100 Mesh)



200 microns

Figure 4-1: SEM photograph of 50 x 100 mesh Maywood Composite 1016 showing monazite rimming of quartz.

size fraction. This photograph shows two composite particles made up of "monazite" rimming quartz. Figures 4-2 and 4-3 show examples of "monazite" located in the pores of slag material. Figure 4-2 is a whole particle (not mounted in epoxy and polished) from the Composite 1016 50x100 mesh size fraction that floated during a density separation at a specific gravity of 2.49 g/cc. Figure 4-3 is a particle from the Composite 1016, 8x16-mesh size fraction showing a similar occurrence of "monazite". High magnification observations of "monazite" in these pores indicate that the "monazite" occurs as very fine particles resting in the pores and is not physically bound to the slag. Figure 4-4 is a photograph taken of the Composite 1016, 8x16 mesh size fraction which shows "monazite" disseminated throughout a slag particle as well as embedded in an iron oxide particle.

#### 4.3 Results and Discussion

Based on the preliminary SEM analyses, it appears that the radioactive components in these soils are generally contained in heavy mineral sands and are predominately in the form of monazitelike particles. These monazite-like particles can either be free, locked with other heavy mineral sands or locked with slag material. The exact distribution and occurrence of these particles can be quantified by examining the SEM-IA data.

Figures 4-5 to 4-7 show the size-by-size composition of Composite 1015, Composite 1016, and Wayne (1017) soils relative to heavy mineral content. These figures are probably best characterized by considering the amount of material containing no heavy mineral contamination. As shown, the amount of material containing zero percent heavy mineral increases with size until approximately 30 mesh, after which there is little change or only a slight increase. This is true in all cases with the exception of the 100x200-mesh size class for the two Maywood samples. In the case of the Maywood samples, there appears to be an abrupt increase in the heavy mineral contamination of the 100x200-mesh fraction. In general, it can be seen that the Wayne sample contains the largest amount of material that is uncontaminated by heavy minerals, followed by the Composite 1015 and the Composite 1016 soils.

If only the heavy mineral portion of Composite 1015, Composite 1016, and Wayne (1017) soils is considered, it is possible to characterize these samples on the basis of the heavy mineral occurrence. Figures 4-8 to 4-10 show the breakdown of the heavy minerals contained in each soil in terms of the amount that is free and that which is locked with other materials. In general, the amount of free (100 percent) heavy mineral increases as particle size decreases, with two exceptions to this trend. The 16x30-mesh fraction of Composite 1015 seems

# Maywood Composite 1016 (Float 2.49 SG, 50x100 Mesh)



200 microns



# Maywood Composite 1016 (8x16 Mesh)



200 microns

Figure 4-3: SEM photograph of 8 x 16 mesh Maywood Composite 1016 showing monazite contained in air pockets in a slag particle.

# Maywood Composite 1016 (8x16 Mesh) Slag Processed Monazite Iron Oxide Processed Monazite 1000 microns

Figure 4-4: SEM photograph of 8 x 16 mesh Maywood Composite 1016 showing monazite inclusions in iron oxide slag.



Figure 4-5: Volume distribution of Maywood Composite 1015 based on the volume percent heavy minerals in each particle.



Figure 4-6: Volume distribution of Maywood Composite 1016 based on the volume percent heavy minerals in each particle.



Figure 4-7: Volume distribution of Wayne composite based on the volume percent heavy minerals in each particle.



Figure 4-8: Volume distribution of the heavy minerals in Maywood Composite 1015 based on the volume percent heavy minerals in each particle.



Figure 4-9: Volume distribution of the heavy minerals in Maywood Composite 1016 based on the volume percent heavy minerals in each particle.



Figure 4-10: Volume distribution of the heavy minerals in Wayne composite based on the volume percent heavy minerals in each particle.
to show an increase in the amount of locked heavy mineral, while the 200x400-mesh fraction of Composite 1016 exhibits a similar condition. It is interesting to note that the sample containing the highest amount of radioactive contamination (Composite 1016) also seems to contain the largest amount of free heavy minerals. Since free particles are generally easier to remove than locked particles, this finding would seem to be encouraging from a remediation point-of-view.

All the results shown in Figures 4-5 to 4-10 are summarized in Figures 4-11 and 4-12. These plots show the combined analyses for all size classes, weighted according to the amount of material in each size class. The overall analysis of the three soil samples is shown in Figure 4-11. As shown, the Wayne (1017) soil contains the largest amount of material (85.6 percent) which is uncontaminated by heavy minerals. This is followed by the Composite 1015 sample with 76.7 percent and the Composite 1016 sample with 63.2 percent. Assuming that all radioactivity was caused by heavy minerals, these numbers would indicate the amount of material which could be returned as clean soil, provided a perfect separation could be achieved. If one considers the remaining material which is contaminated by heavy minerals (Figure 4-12), 66.7 percent of this material occurs as free heavy-mineral particles in the Composite 1016 sample, while approximately 46 percent is free in both the Wayne (1017) and the Composite 1015 soils. From the point-of-view of remediation, the percentage of free heavy minerals should represent the portion of the contamination that is easiest to remove. Thus, nearly two-thirds of the heavy minerals contained in the Composite 1016 sample, and half of those contained in the Wayne (1017) and Composite 1015 samples, are accessible for removal by a physical separation process.

The data collected from the SEM analysis were also used to determine the total amount of heavy minerals contained in each size class. These data are shown in Figure 4-13. As shown, there is generally little change or a slight decrease in heavy mineral content for all three samples as size is decreased from the 8x16-mesh class to the 50x100-mesh class. This is followed by an abrupt increase in the heavy mineral content at the 100x200-mesh class, and a decrease, once again, in the 200x400-mesh class. Thus, it would seem that the 100x200-mesh class contains the greatest contamination of heavy minerals. In fact, in the case of the Composite 1016 sample, over one-fourth of all the material in the 100x200-mesh class is composed of heavy minerals. In the case of the Composite 1015 sample, both the 100x200-mesh class and the 8x16-mesh class appear to be equally contaminated by heavy minerals (approximately 7 percent); while in the case of the Wayne (1017) sample, no class contains greater than 4 percent heavy minerals. The overall heavy mineral composition in the 8x400-mesh in Composite 1015, Composite 1016, and Wayne (1017) samples is shown in Figure 4-14. The Composite 1016 sample contains nearly



Figure 4-11: Weighted volume distribution of the 8 x 400 mesh Maywood #1 (Composite 1015), Maywood #2 (Composite 1016) and Wayne composite samples based on the volume percent heavy mineral in each particle.



Figure 4-12: Weighted volume distribution of the heavy minerals in the 8 x 400 mesh Maywood #1 (Composite 1015), Maywood #2 (Composite 1016) and Wayne samples based on the volume percent heavy mineral in each particle.



Figure 4-13: Volume percent heavy minerals in each size class for the Maywood #1 (Composite 1015), Maywood #2 (Composite 1016) and Wayne Composite samples.



Figure 4-14: Overall volume percent heavy minerals in the 8 x 400 Mesh Maywood #1 (Composite 1015), Maywood #2 (Composite 1016) and Wayne composite samples weighted according to the size distribution.

four times as much heavy mineral contamination as does the Composite 1015 sample, and nearly ten times as much as the Wayne (1017) sample. This finding correlates to the relative levels of radioactive contamination in each of the soils.

Since not all of the heavy minerals present in the sample contribute to the overall radioactivity, it was decided to break out those heavy minerals which contain thorium. These data are shown in figures 4-15 and 4-16. As shown in Figure 4-15, there is a clear trend in all three samples for the percentage of thorium-containing minerals to increase with decreasing particle size, with the greatest increase occurring in the 100x200-mesh size class. This is followed by a slight decrease in the thorium mineral content in the 200x400-mesh size class; although the thorium mineral content is still relatively high in this class. Thus, in terms of thorium-containing minerals, the 100x200 and 200x400-mesh size classes both have significant quantities. Furthermore, this finding seems to be true for all three samples; although the percentages are quite low for the Composite 1015 and Wayne (1017) samples. In terms of overall thorium-containing minerals content (Figure 4-16), over 7 percent of the Composite 1016 sample is composed of thorium-containing minerals. This accounts for about half of the heavy minerals in that sample. The Composite 1015 and Wayne (1017) samples, on the other hand, contain less than 0.5 percent thorium-bearing minerals.

In order to relate the characterization data to the radionuclide analysis, an attempt was made to correlate the TAR for each size class with the percentage of heavy minerals and the percentage of thorium minerals in each size class. These results, for all three soil samples, are shown in Figures 4-17 to 4-22. As shown, there appears to be a general correlation between TAR and both the percentage of heavy minerals and the percentage of thorium minerals; however, the correlation between TAR and percentage of thorium minerals appears to be better. For example, in the case of the Composite 1015 sample (Figures 4-17 and 4-18), the general trend in the thorium mineral composition with size and the TAR variation with size (Figure 4-18) is the same with the exception of the 8 x 16-mesh size class. This trend is not as clear if the total percentage of heavy minerals (Figure 4-17) is used. Similar findings can be seen for the other two soil samples. Based on these results, a plot of TAR versus thorium mineral content was generated. As shown in Figure 4-23, this plot results in a straight line on a log-log scale which seem to level out at a TAR of 1. The intercept of the line at a TAR of 1 is approximately 0.06 percent thorium minerals, which indicates that it is necessary to clean these soil samples to a thorium mineral content of less than 0.06 percent in order to achieve a TAR of 1 or less.



Figure 4-15: Volume percent thorium minerals in each size class for the Maywood #1 (Composite 1015), Maywood #2 (Composite 1016) and Wayne Composite samples.



Figure 4-16: Overall volume percent thorium minerals in the 8 x 400 Mesh Maywood #1 (Composite 1015), Maywood #2 (Composite 1016) and Wayne composite samples weighted according to the size distribution.



Figure 4-17: Relationship between volume percent heavy minerals and total activity ratio as a function of particle size for the Maywood #1 (Composite 1015) composite sample.



Figure 4-18: Relationship between volume percent thorium minerals and total activity ratio as a function of particle size for the Maywood #1 (Composite 1015) composite sample.



Figure 4-19: Relationship between volume percent heavy minerals and total activity ratio as a function of particle size for the Maywood #2 (Composite 1016) composite sample.



Figure 4-20: Relationship between volume percent thorium minerals and total activity ratio as a function of particle size for the Maywood #2 (Composite 1016) composite sample.



Figure 4-21: Relationship between volume percent heavy minerals and total activity ratio as a function of particle size for the Wayne composite sample.



Figure 4-22: Relationship between volume percent thorium minerals and total activity ratio as a function of particle size for the Wayne composite sample.



Figure 4-23: Relationship between total activity ratio and volume percent thorium minerals.



Figure 4-24: Volume percent thorium minerals as a function of particle size for the Maywood #1 (Composite 1015) composite showing the breakdown between free and locked particles.

Finally, the 0.06 percent limit on thorium mineral content was used as a means of evaluating the potential cleanability of each of the three soils. This was done by plotting the total thorium mineral content in each size class on a bar chart. Each bar was broken down to represent the portion of the thorium mineral present as free particles and the portion present as locked material. This information is shown in Figures 4-24 to 4-26. The line drawn on each chart represents the theoretical cut-off limit for thorium mineral content in order to achieve a TAR of 1. Any part of a bar which appears above the line represent material that must be removed to achieve the TAR of 1. Since locked particles are generally much more difficult to remove than free particles, any soil samples which contain locked material appearing above the line would probably be difficult to clean. As shown in Figure 4-24 for the Composite 1015 sample, only the 8 x 16 mesh and the 100x200 mesh size classes contains any locked material that would need to be removed, and in the case of the Wayne (1017) sample (Figure 4-26), only free material would need to be removed. Thus, these soil seems to show the most promise for remediation. In the case of the Composite 1016 sample, the scale is so large that the cut-off line is not even visible. A considerable amount of locked material would have to be removed along with the free material. Thus, the Composite 1016 soil is most likely not amenable to remediation by physical separation techniques alone.

## 4.4 Conclusions

Characterization of Composite 1015, Composite 1016, and Wayne (1017) composite soil samples was carried out using Scanning Electron Microscopy coupled with Automated Image Analysis. The major findings of this analysis are given as follows:

- The only radioactive component observable via x-ray analysis under SEM was thorium. Thorium was found to occur primarily as a monazite-type particle. These monazite-type particles were found to occur both as free and locked particles. In the coarser size fractions (8x16 mesh) monazite inclusions were often found in the pore structure of slag particles. At the finer sizes (minus 50 mesh), the monazite-type material was predominantly free; although some processed monazite was found to occur as a rim around quartz particles.
- The amount of soil uncontaminated by the presence of heavy mineral sands was generally found to increase as particle size decreased. In the case of the 8x400 mesh, Composite 1015 sample, 76.7 percent of the soil was uncontaminated, while the 8x400 mesh of Composite 1016 sample contained 63.2 percent uncontaminated soil. The best situation



Figure 4-25: Volume percent thorium minerals as a function of particle size for the Maywood #2 (Composite 1016) composite showing the breakdown between free and locked particles.



Figure 4-26: Volume percent thorium minerals as a function of particle size for the Wayne composite showing the breakdown between free and locked particles.

appeared to be for the Wayne (1017) sample which contained 85.6 percent uncontaminated material.

- The percentage of free heavy minerals contained in each sample was generally found to increase with decreasing particle size. For the Composite 1015 and the Wayne (1017) samples, the percentage of free heavy minerals amounted to approximately 46 percent. The Composite 1016 sample, on the other hand, contained 66.7 percent free heavy minerals. Thus, although the Composite 1016 sample was the most contaminated particles, it also contained the largest percentage of heavy minerals in a separable form.
- The volume percent of heavy minerals contained in each sample ranged from just over 14 percent for the Composite 1016, to approximately 3.5 percent for Composite 1015, and 1.5 percent for the Wayne (1017) sample. In all cases, the 100x200-mesh size fraction seemed to stand out as the worst in terms of heavy mineral contamination. For the Composite 1015 sample, the 8x16 mesh fraction was also significant, and for the Composite 1016 sample, the 200x400 mesh class was heavily contaminated as well.
- In terms of thorium mineral contamination, the Composite 1016 sample contained just over 7 percent thorium minerals, while the Composite 1015 and Wayne (1017) sample contained less than 0.5 percent. Once again, the 100x200 mesh size fraction stood out as the most contaminated in all three samples. In addition, the 200x400 mesh fraction was significant for the Composite 1016 and the Wayne (1017) samples.
- In an attempt to relate the radionuclide analysis to the characterization data, the TAR was compared to the heavy mineral content and the thorium mineral content of each size fraction. The TAR was found to correlate well with the thorium mineral content. A plot of TAR versus thorium mineral content indicated that these soils must be cleaned to a thorium mineral content of less than 0.06 percent in order to achieve a TAR of 1 or less.
- Based on the theoretical cut-off limit on thorium mineral content, the characterization
  data were used to determine the amenability of each soil for physical remediation. The
  Composite 1015 and Wayne (1017) samples were both found to exhibit characteristics of
  soils that would be amenable to physical separation. The Composite 1016 sample, on the
  other hand, appeared to contain far too much locked material to ever meet a TAR
  criterion of 1.

# 5.0 EXTENDED CHARACTERIZATION - LABORATORY TESTS

#### 5.1 Introduction to Laboratory Testing

In this subtask, two mineral processing techniques (i.e., density concentration and froth flotation) have been evaluated for the remediation of feed soil from the Maywood FUSRAP site. Density concentration exploits differences in the specific gravity of contaminated particles to remove them from the host soil. This technique is generally effective for treating sand-sized material. Froth flotation, which is a physicochemical process, exploits differences in the surface properties (i.e., wettability) of particles to achieve the desired separation. Froth flotation offers tremendous flexibility since particle wettability can be controlled through the use of appropriate surface modifying reagents. Another advantage of this technique is its ability to treat very fine silt-sized particles. The density concentration and froth flotation technologies are described in greater detail in the following sections.

### 5.1.1 Density Concentration

Float-sink testing is the simplest of all gravity processes and has long been a standard laboratory method for separating minerals of different specific gravities. In principle, given sufficient time, it is possible to make a perfect separation between two particles of differing density by placing them in a liquid whose density is intermediate between the two. This principle is shown in the following schematic.



The objective of float-sink testing is to separate the sample into a series of fractions according to specific gravity. These fractions are then weighed and analyzed to establish relationships between mass recovery of particles and the concentration of wanted or unwanted components. These relationships are used to construct the standard float-sink (washability) curves to estimate the response of a given sample to density concentration. These curves are developed by subjecting either the whole or various size fractions of the sample to float-sink testing. Float-sink tests are widely used in the laboratory (i) to evaluate the potential of density concentration

methods, (ii) to determine the economic separating density, and (iii) to evaluate the efficiency of an existing density concentration process.

Most float-sink tests are performed using organic liquids which are available in a wide range of specific gravities. Since most organic liquids are toxic and produce harmful vapors, the use of true heavy liquids has not been found practicable on a commercial scale. Most industrial density separations employ artificial heavy liquids (heavy media) consisting of finely ground solids suspended in water. These heavy media processes have the ability to make sharp separations at nearly any required density with a high degree of efficiency. The density of separation can be closely controlled and can be maintained under normal conditions for indefinite periods. The process is applicable to any material in which, after a suitable degree of liberation, there is a large enough difference in specific gravity between the particles to allow a separation. Heavy media processes are widely applied when the density difference occurs at a relatively coarse particle size (i.e., gravel-sized material). Separation in fine sizes can also be made through the use of centrifugal separators, although the separation efficiency decreases with decreasing size due to the slower rate of settling of finer particles.

Unfortunately, the small production rate and high costs associated with ancillary equipment are likely to prevent the use of heavy media processes for soil remediation. Fortunately, several water-based techniques are commercially available for density concentration. These units exploit differences in the settling velocities of light and heavy particles to achieve a selective separation. Examples of commercially available techniques include jigs, teeter-bed separators, water-only cyclones, and flowing-film (table and spiral) concentrators. A jig uses water pulsed from below to segregate a particle into high and low density fractions. This technique is generally not effective if too many near gravity particles are present. Teeter-bed separators are a special class of hydroclassifiers which create an artificial density by fluidizing particles from the host material in an upward current of fluid. The effectiveness of these units is generally limited to a very narrow size range. Water-only cyclones may also be used for density separations, although they also result in particle classification. Flowing-film concentrators, such as shaking tables and spirals, are the most popular water-based density concentrators. These devices take advantage of differences in the movement of particles of various densities as they move through a thin film of moving liquid. Flowing-film concentrators are effective over a relatively wide range of particle sizes and require little in terms of ancillary equipment.

### 5.1.2 Froth Flotation

Flotation is undoubtedly the most important and versatile mineral processing technique. In this technique, fine particles suspended in water are treated with chemical reagent which selectively makes some of the particles nonwettable (hydrophobic), while others remain wettable (hydrophilic). A separation is achieved when the hydrophobic particles selectively adhere to air bubbles which are introduced into the flotation cell. Particles adhering to the air bubbles are carried to the top of the flotation pulp and collected in the form of froth. Particles which do not attach to air bubbles remain suspended in the pulp and are eventually discharged from the bottom of the flotation cell.

Most minerals are not hydrophobic in their natural state so flotation reagents must be added to the pulp. The most important reagents are the collectors. Collectors selectively adsorb on mineral surfaces, render them hydrophobic and facilitate bubble attachment. In fact, the flexibility of flotation is largely due to the availability of a wide variety of collectors which can render different types of minerals hydrophobic. To prevent the drop-back of particles after the bubble reaches the surface of the flotation pulp, a frothing agent is normally added to promote the formation of a stable froth. Also, regulators are commonly used to control the flotation process. These reagents either activate or depress mineral attachment to air bubbles and are also used to control the pH of the system. A brief review of the role of these reagents and the physical variables influencing the flotation are presented below.

*Collectors:* Froth flotation is a viable process only if the wettability of particles can be controlled. To achieve this, surfactants known as collectors are added to the pulp. A period of time is normally allowed for collector adsorption during agitation in what is known as the conditioning period. Collectors are organic compounds which render selected minerals hydrophobic by adsorption of molecules or ions on to the mineral surface. The adsorption reduces the stability of the hydrated layer separating the mineral from the air bubble to such a level that particle-bubble attachment can be made on contact. Collector molecules may be ionizing compounds, which dissociate into ions in water, or non-ionizing compounds, which are practically insoluble and render the mineral hydrophobic by covering its surface with a thin, nonwettable film. Ionizing collectors have found wide application in flotation. They have complex molecules which are asymmetric and are heteropolar, i.e., the molecule contains a nonpolar hydrocarbon group and a polar group. The nonpolar hydrocarbon radical has pronounced water-repellent properties, whereas the polar group reacts with water. Ionizing collectors are classified in accordance with the type of ion, anion or cation, that produces the

water-repellent effect in water.

Because of the chemical, electrical, or physical attraction between the polar portions and mineral particles, collectors adsorb on the particles with their nonpolar ends orientated towards the bulk solution, thereby imparting hydrophobicity to the particles. Collectors are added in small amounts, usually just sufficient to form a monomolecular layer on the particle surfaces. In fact, an excessive concentration of a collector can have an adverse effect on the recovery of valuable minerals. Apart from the cost, an excessive collector addition will tend to float other minerals, thus reducing selectivity. However, it not uncommon to add more than one collector to a flotation system. A selective collector may be used at the head of the circuit, to float the highly hydrophobic minerals, after which a more powerful, but less selective, collector is added to promote the recovery of slower floating minerals.

*Frothers:* Frothers are water soluble organic reagents that adsorb at the air-water interface. They are heteropolar molecules, with a polar group to provide water solubility, and a nonpolar hydrocarbon group. A frother is required to provide a froth above the pulp that is stable enough to prevent undue froth breakage and subsequent return of particles to the pulp before the froth is removed. The frothing action is due to the ability of the frother to adsorb at the air-water interface and reduce the surface tension. It is important that the froth breakdown rapidly once the froth is removed from the top of the flotation cell. Frothers are in many respects chemically similar to ionic collectors, and, indeed many of the collectors are powerful frothers. In fact, they are too powerful to be used as efficient frothers, since the froths which they produce can be too stable to allow efficient transport to subsequent processing steps. A good frother should have negligible collecting power, and also produce a froth which is just stable enough to facilitate transfer of floated mineral from the cell surface to the froth launder.

*Regulators:* Regulators or modifiers, are used extensively in flotation to modify the action of the collector, either by intensifying or reducing its water repellent effect on the mineral surface. They make collector action more selective towards certain minerals. Regulators can be classed as activators, depressants, or pH modifiers. Activators are the reagents which alter the chemical nature of mineral surfaces so that they become hydrophobic due to the action of the collector. Activators are generally soluble salts which ionize in solution, the ions then react with the mineral surface.

Depressants are used to increase the selectivity of flotation by rendering certain minerals hydrophilic, thus preventing their flotation. The mechanisms in which these depressants achieve

their effects are well documented in the literature and various theories have been offered as to their mode of action. These reagents act by preferential adsorption onto the surface of the unwanted material and reduce its state of flotation or increase its hydrophilic properties, thus giving it less tendency to adhere to air bubbles. These depressants require close control since a slight excess may change the entire flotation process.

Regulators for pH are used to control the pulp alkalinity and play a very important role in flotation. In practice, selectivity in complex separations is dependent on a delicate balance between reagent concentrations and pH. Flotation, where possible, is carried out in an alkaline medium, as most collectors are stable under these conditions. Pulp alkalinity is controlled by the addition of lime, sodium carbonate or sodium hydroxide. These chemicals are often added to the slurry prior to flotation to precipitate heavy metal ions from solution. Since the heavy metal salts precipitated can dissociate, to a limited extent, and allow ions into the solution, other chemicals are often used with the alkali to complex them. By carefully controlling the pulp alkalinity, the critical pH can be determined, below which any given mineral will float, and above which it will not float. This critical pH value depends on the nature of the mineral, the particular collector, and its concentration.

The surface charge of the particles also plays a significant role in the adsorption of a collector on the mineral surface. When a particle is suspended in an aqueous solution, a surface charge is developed at the interface under equilibrium conditions. Generally, three interface potentials are of interest for the charged surfaces. These are (a) the surface potential, (b) the Stern potential (the potential of first layer of counter ions), and finally, (c) the zeta potential. The nature of the zeta potential can be seen in Figure 5-1, which shows a model of the electric double layer at the particle surface. The zeta potential is the potential at the shear plane, where slip takes place when the surface moves relative to the liquid. An important parameter for characterizing the charged surface is the point of zero charge (PZC). When the net surface charge is zero, the surface is considered to be at its PZC and have a zeta potential equal to zero, as shown in Figure 5-2. According to flotation chemistry theory, the surface hydrophobicity is maximum at the PZC.

In order to develop a flotation circuit, preliminary laboratory test must be undertaken in order to determine the choice of reagents for a given throughput as well as flow sheet data. The bulk of the laboratory test work is carried out in batch flotation cells, usually with 500 g, 1000 g and 2000 g of material. The cells are mechanically agitated, the speed of the rotation of the impellers being variable, and simulate the large-scale models commercially available. The air is



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Figure 5-1: The nature of zeta potential showing a model of the electric double layer at the particle surface.

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usually introduced through a hollow standpipe surrounding the impeller shaft. The action of the impeller draws air down the standpipe, the volume being controlled by a valve. The air stream is sheared into fine bubbles by the impeller, and rise to the surface, where any hydrophobic particles are removed as mineralized froth.

# 5.2 Experimental

# 5.2.1 Float-Sink Analysis

The section describes the procedure used to develop float-sink (washability) curves for Composite 1015. These experiments provide data that can be used to establish if gravity concentration is a viable approach for separating thorium and radium radionuclides from contaminated soils.

The float-sink analyses were conducted on Composites 1015 and 1016 for five different size fractions; namely,  $4 \ge 8$ ,  $8 \ge 16$ ,  $16 \ge 30$ ,  $30 \ge 50$ , and  $50 \ge 100$  mesh. Lithium Metatungstate (LMT), having a specific gravity of 3.0, was used in the experiments and solutions with lower specific gravities were prepared by simply adding distilled water to LMT and mixing well.

Solutions of different specific gravities ranging from 2.5 to 3.0 in incremental steps were prepared. The representative soil sample was weighed and introduced into the solution of lowest density. The tests were performed in a separatory funnel, and the sinks product was removed, washed and dried. After the sinks product was completely dried, it was placed in the liquid of the next higher density, whose sinks product is then transferred to the next higher density and so on. The floats products were recovered, washed and dried, and weighed together with the final sinks product, to give the density distribution of the sample by weight. The entire procedure was repeated for five size fractions for both composite samples.

All the density fractions were analyzed by gamma and alpha spectrometry as described in Sections 2.1.1 and 2.1.2.

## 5.2.2 Flotation Tests

The effective use of flotation technology requires the development of an appropriate reagent blend so that radioactive particles are dispersed and at the same time made hydrophobic. Only under these circumstances is it possible for froth flotation to be successful in cleaning the

contaminated soils. The following section summarizes the procedures used for all flotation tests conducted on Composite 1015.

The optimal particle size range for froth flotation is normally 40-300 microns (400 to 50 mesh), depending on the mineral type and surface chemistry. As was reported in Part I, most of the radioactive contaminants in the soils are concentrated in the fine-size fractions. Hence, the bench-scale flotation tests were conducted on 50x200 and -50 mesh size fractions for all soil samples.

A representative soil sample weighing 1600 g, mixed with 6 kg/t of sodium carbonate, was scrubbed at 40 percent solids for 10 minutes in the flotation cell. The slurry was then screened using 50 and 200-mesh sieves. The soil fractions consisting of -50 mesh, 50x200, and -200 mesh, were stored in plastic containers for the flotation tests. A Denver flotation machine was used for bench-scale tests in order to remove radionuclides from the soil samples. The flotation tests were carried out in a 4-liter cell at room temperature as described by the experimental procedure in Figure 5-3. Two groups of tests were performed, one with feed size of -50 mesh and other with 50 x 200-size fraction. An 800 g aliquot of the soil sample was taken into the cell with an appropriate tap water addition to make a 20 percent solids suspension by weight. The suspension was first conditioned for about 10 minutes with the addition of the desired modifier to regulate the suspension pH. Sodium Oleate (a fatty acid collector) was then added and the suspension was conditioned for another 10 minutes prior to flotation. Four drops of MIBC (methyl isobutyl carbinol) frother was added and the pulp was conditioned for additional five minutes. The air was applied and a single stage flotation was carried out at 1500 rpm for an appropriate time period (10-15 minutes) or until no mineralized bubbles were observed. The experimental operating conditions and the amounts of reagents added during the test are listed in Table 5-1. After flotation, both the concentrate and tails (the clean soil) were collected, filtered and dried for analysis. The concentrate, tail, and feed samples were analyzed by gamma and alpha spectrometry.



Figure 5-3: Procedure for bench-scale flotation tests of contaminated soil samples.

Sample Size	50 x 200 mesh
	- 50 mesh
Pulp Density	20 percent solids by weight
Collector	Sodium Oleate
	90 g/Ton
Frother	MIBC (4 drops)
Initial pH	9.6
Dispersant	Sodium Carbonate
	6.0 kg/Ton
Impeller Speed	1500 rpm
Aeration Rate	Air flow valve completely open

Table 5-1: Experimental Operating Conditions

Release analysis tests were performed to provide a measure of ultimate cleanability of a sample by froth flotation. Release analysis is based on the assumption that, provided recovery by entrainment is eliminated, changes in flotation operating conditions can be used to generate results along a single "ideal" separation curve. In this approach, the sample was initially separated into floatable and non-floatable components by utilizing recurrent stages of cleaning. The floatable material was then separated into components having various degrees of floatability by collecting froth products (concentrates) as a function of increasing aeration rate and impeller speed. This procedure results in four concentrates and an overall tailings from which the release curve was constructed. The release analysis tests were conducted on -200 mesh size fraction for both composite samples.

Release analysis tests were carried out in a 4-liter batch flotation cell using a Denver D-12 flotation apparatus. In this technique, approximately 200 g of the sample was taken into the cell with an appropriate tap water addition to make a 5 percent solids suspension by weight. The pulp was then conditioned with appropriate amounts of modifiers, collectors, and frothers prior to flotation at an impeller speed of 1700 rpm. The first stage of release analysis involved the separation of the non-floatable material from the floatable material. Flotation started with the impeller speed at 1700 rpm and air inlet valve completely open. A high pulp level was maintained throughout this stage, and the froth was gently removed to minimize entrainment. Extra frother was added as needed to maintain stable froth and was continued until the froth appeared to be barren. The tail material was then saved and the froth product was repulped for

additional cleaning. This procedure was repeated three times to minimize the presence of entrained material in the froth product. The resulting products from the first stage consisted of a concentrate containing all truly floatable material and a tail product containing all the non-floatable material.

The second stage of the release analysis procedure involved the separation of the floatable material into components of various degrees of floatability. In this stage, the concentrate product from the first stage was repulped and floataion was initiated with an impeller speed of 1100 rpm and an aeration rate of approximately 50 percent of full range. These weak floataion conditions allowed only the most floatable material to report to the froth product. After the froth became barren, the collection basin was changed, and the impeller speed was increased to 1300 rpm and aeration rate of 60 percent, until the second concentrate is formed. This concentrate was removed as the second froth product. The procedure of increasing impeller speed and aeration rate was repeated two additional times to produce a total of four concentrates. The tail material was combined with the first stage tail product to produce an overall tail product. All four concentrates and the combined refuse product were filtered, dried, and weighed. The dry weights were recorded and the concentrate, feed, and tail samples were analyzed by gamma and alpha spectrometry.

#### 5.3 Results and Discussion

#### 5.3.1 Float-Sink Analysis

The density separation and petrographic data from the initial characterization study indicate that a significant amount of the radioactivity in Composite 1015 is associated with dense (>2.89 g/cc) minerals. These results led to the recommendation to further characterize the Maywood samples by density partitioning. Float-sink analyses were performed on the 4 x 8, 8 x 16, 16 x 30,  $30 \times 50$ , and  $50 \times 100$ -mesh fractions to better define the radionuclide concentrations in these fractions based on density. The fractions were partitioned at Float 2.5, 2.5 x 2.6, 2.6 x 2.7,  $2.7 \times 2.8$ ,  $2.8 \times 2.9$ ,  $2.9 \times 3.0$ , and Sink 3.0 SG fractions. The resulting fractions were analyzed by gamma and alpha spectrometry as discussed in 2.1.1 and 2.1.2, respectively.

It should be noted that detailed petrographic results for Composite 1016 indicated that remediation by soil treatment will not be feasible due to the percent thorium removal that has to be achieved to reach the clean-up criteria target goals. However, these data are not conclusive and it was recommended to perform the float-sink analyses to better evaluate the potential for

remediation of this composite sample. The float-sink data are presented in Appendix C. The float-sink tests did not significantly reduce the Th-232 activity in the density fractions. Therefore, density separations does not reduce the radionuclide activities to reach the target clean-up criteria.

The data for Composite 1015 are summarized in Figures 5-4 through 5-8 and presented in Appendix C. These figures present the results of the density partitioning for Ra-226, Ra-228, and Th-232. The results indicate that the contamination is distributed throughout the density fractions. However, these results show that the contamination is mostly associated with the high density partitions. These results are expected based on the assumption that the radionuclide are associated heavy minerals. The float-sink data confirms the SEM/XRF results including that indicated the contaminants are associated with the heavy minerals.

Figures 5-4 through 5-8 also indicate that the density fractions with the highest weight percent are the Float 2.5 and 2.6 x 2.7 fraction. The Th-232 and Ra-226 activities are below 5 pCi/g in these density fractions. Therefore, these results are very promising and suggest that a density separation unit operation may be beneficial in reducing the contamination for Composite 1015 soil at the Maywood site.

#### 5.3.2 Flotation Tests

The detailed petrographic data indicate that as the particle size decreased, the percent liberation increases. It should be noted that the -400-mesh material was not analyzed and that the percent liberation became constant around 70 to 80 percent liberation. For Composite 1015 and Composite 1016, approximately 36 and 41 percent of the soil, respectively, is -200 mesh. Also, the -200-mesh material has high radionuclide concentrations. Therefore, these data indicate that for successful remediation with a large volume reduction, contaminated particles in the fine size classes must be removed. The preferred, cost effective method for fine-particle separation is flotation. Flotation tests were performed on the -50, 50 x 200, and -200-mesh material for Composite 1015 and Composite 1016. The resulting fractions are the feed, tail, and concentrate. These fractions were analyzed by gamma and alpha spectrometry as discussed in 2.1.1 and 2.1.2, respectively in this report.

It should be noted that detailed petrographic results for Composite 1016 indicated that remediation soil treatment will not be feasible due to the percent thorium removal that must be achieved to reach the target goals. However, these data are not conclusive and it was

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recommended that flotation tests be performed to better evaluate the potential for remediation of this composite sample. The flotation test data are presented in Appendix C. The flotation tests did not significantly reduce the activity for any radionuclide, thus confirming the results from the detailed petrographic work. Therefore, based on these data it does not appear that soil representative of Composite 1016 can be treated at the Maywood site using physical separation processes.

The data for Composite 1015 sample are summarized in Figures 5-9 through 5-11 and presented in Appendix C. These figures present the results of the flotation tests for Ra-226, Ra-228, and Th-232. The results indicate that the contamination is concentrated in the concentrate product. These results also indicate that Th-232 and Ra-228 are the major radionuclides of concern. This is consistent with the particle-size and density partitioning data.

Figure 5-9 presents the results for the -50-mesh flotation test. The activities decreased in the tail product and increased in the concentrate product, as expected. However, the tail product has a Th-232 activity of approximately 33 pCi/g, which is above the clean-up goals. The Th-232 feed activity is approximately 43 pCi/g and the concentrate has an activity of 70 pCi/g. Therefore, the -50 mesh flotation test produced a tail product with a 23 percent Th-232 reduction and a concentrate with a 63 percent increase in Th-232 activity. These percent changes are promising, but not sufficient to meet the clean-up criteria. The flotation chemistry is more difficult on the ultra-fine material (-200 mesh), which contains the highest concentrations. This means that the selective collector and the operating parameters of the flotation circuit must be optimally controlled. Limited resources did not allow these parameters to be optimized. However, there are supportive data, from both the detailed petrography and this flotation test to indicate that flotation may be successful in reducing the activities below the clean-up criteria. Further laboratory testing should be done to optimize the operating parameters, as well as the selective collector.

Figure 5-10 presents the results for the 50 x 200-mesh flotation test. The ultra-fine material was removed for this test due to the problems discussed in the above paragraph. Again, the activities decreased in the tail product, but not significantly, and increased in the concentrate product, as expected. In this test, the tail product has a Th-232 activity of approximately 32 pCi/g. The Th-232 feed activity is approximately 40 pCi/g and the concentrate has an activity of 168 pCi/g. Therefore, the 50 x 200 mesh flotation test produced a tail product with a 20 percent Th-232 reduction and a concentrate with a 320 percent increase in Th-232 activity. These percent changes are more promising than what is observed in the -50 mesh flotation test indicating that







the problems associated with the ultra-fine material are confirmed. Again, the clean-up goals are not met for the Th-232, however, with additional controlled laboratory testing, the selective collector and operating parameters can be optimized. Thus, the data for the 50 x 200 mesh floatation test indicate great promise and support further laboratory testing.

Figure 5-11 presents the results for the -200 mesh release analysis flotation test. The ultra-fine material was used in this test to evaluate the effectiveness of the collector and operating parameters used in the previous tests. Since it is known that the ultra-fine material is the most difficult to remediate, these data are useful in determining a starting point for finding the optimal conditions for an ultra-fine flotation circuit. From this starting point, conditions can be modified and the test repeated. This process is continued until the maximum recovery of the concentrate is achieved. In this test, the resulting fractions are the feed, tail, concentrate 1, concentrate 2, concentrate 3, and concentrate 4. As in the previous tests, the activities decreased in the tail product and increased in the concentrate products. The tail product has a Th-232 activity of approximately 37 pCi/g. The Th-232 feed activity is approximately 41 pCi/g. The Th-232 activities for concentrate 1, concentrate 2, concentrate 3, and concentrate 4 are 47, 76, 110, and 111, respectively. Therefore, the tail product has a 10 percent Th-232 reduction. Concentrate 1, concentrate 2, concentrate 3, and concentrate 4 have a 15, 85, 168, and 171 percent increase in Th-232, respectively. These data are not as promising as in the previous tests, however, as stated before, the flotation chemistry for the ultra-fine material will be more difficult and these data will be used as a starting parameters in optimizing the ultra-fine flotation circuit. Further release analysis flotation tests should be done to determine if the ultra-fine material can be remediated for the Maywood site.

## 5.4 Conclusions

## 5.4.1 Float-Sink Analysis

Float-sink analyses were performed on the 4 x 8, 8 x 16, 16 x 30, 30 x 50, and 50 x 100 mesh fractions to better define the radionuclide concentrations based on density for Composite 1015 and Composite 1016. The fractions were partitioned at Float 2.5, 2.5 x 2.6, 2.6 x 2.7, 2.7 x 2.8,  $2.8 \times 2.9$ ,  $2.9 \times 3.0$ , and Sink 3.0 SG fractions. Based on the results, the following conclusions are made.

• The contamination is distributed throughout the density fractions for Composite 1015 and Composite 1016; however, it is mostly associated with the high density partitions.

- The float-sink results for Composite 1016 confirmed the detailed petrographic results that indicated that remediation will not be feasible due to the percent thorium removal that must be achieved. The float-sink tests did not significantly reduce the Th-232 activity in the density fractions such that it would meet the clean-up criteria. Therefore, based on these data, the Maywood site is not treatable by the physical separation processes evaluated.
- For Composite 1015, the density fractions with the highest weight percent are in the Float 2.5 and 2.6 x 2.7 density fraction. Also, in these fractions the Th-232 and Pb-214 activity are both below 5 pCi/g.
- The float-sink results for Composite 1015 support the conclusion that for soils representative of this composite, a density separation unit operation may be beneficial for reducing the activity of radionuclides for at the Maywood site.

## 5.4.2 Flotation Tests

For the Composite 1015 and Composite 1016, approximately 36 and 41 percent of the soil, respectively, is -200 mesh and high activities are associated in this material. The detailed petrographic data indicated that as the particle size decreased, the percent liberation increases. These data indicate that for successful remediation with a large volume reduction, contaminated particles in the fine size classes must be removed. Therefore, flotation tests were performed on the -50, 50 x 200, and -200-mesh material for the Composite 1015 and Composite 1016. Based on the results, the following conclusions are made.

- The contamination is concentrated in the concentrate product for each flotation test for both composites.
- Th-232 and Ra-228 are the radionuclides with the highest concentrations.
- The flotation tests results for Composite 1016 confirmed the detailed petrographic results that indicated that remediation will not be feasible due to the percent thorium removal that has to be achieved. The flotation tests did not significantly reduce the Th-232 activity in the density fractions such that it would meet the TAR clean-up criteria.

# 6.0 FLOWSHEET DEVELOPMENT

# 6.1 Introduction

After successfully completing the characterization studies, a conceptual flowsheet for a production-scale soil treatment plant was developed for the Maywood site. The preferred process flowsheet was selected based on the ability of the circuitry (i) to meet the desired clean-up criteria, (ii) to provide the maximum volume reduction of the contaminated soil, (iii) to minimize the production of secondary wastes, and (iv) to achieve these objectives in a cost-effective manner. Equipment selection was limited to proven, field-tested mineral processing technologies that can be implemented in the field with little or no additional research and development effort.

Specific activities that were performed under the flowsheet development effort included:

- formulation of consistent mass balances which specify the expected solid mass rates and contamination levels based on particle size analyses, float-sink data, and other characterization information,
- development of a process simulation program capable of identifying the preferred circuit configuration for soil treatment,
- preparation of a list of required unit operations including equipment type, unit size, capacity, reagent/chemical requirements, power requirements, etc., and
- construction of process flow diagrams that clearly illustrate mass flow rates, particle size distributions, contamination levels, etc., for all primary process streams.

These efforts are described in the following sections of this report.

# 6.2 Mass Balances

## 6.2.1 Background

Mass balances are routinely performed to evaluate test data obtained from particulate separation processes. Mass balances are based on the law of mass conservation which dictates that the mass of any given component entering a process must equal the mass leaving the process when

operating under steady-state conditions. The concept provides the elementary foundation upon which all process engineering calculations and circuit design efforts are based. In the present work, mass balances are required (i) to obtain consistent data sets for use in the simulation programs and (ii) to provide independent checks on the reliability of the experimental test data.

The mass balance concept has been discussed extensively by Wills (WIL88). In this procedure, the mass flow of material through any series of processes is mathematically represented by a series of interconnected nodes and streams using a connection matrix. The connection matrix is of size  $m \ge n$  where m is the total number of streams and n is the total number of stream connection points (nodes). Two types of nodes are possible, i.e., separators and junctions. A separator is defined as a node having one stream entering and two exiting, while a junction has two streams entering and one exiting. Within the connection matrix, +1 indicates an inflow to the node, -1 indicates an outflow from the node, and 0 indicates the stream has no influence on the node. The sum of any row indicates the type of stream present (i.e., feed stream = +1, product stream = -1, internal stream = 0).

Using the connection matrix (C<sub>ii</sub>), the generalized mass balance equations for the circuit become:

$$\sum_{i=1}^{m} C_{ij} M_i = 0$$
 [6.1]

$$\sum_{i=1}^{m} C_{ij} A_i^k M_i = 0$$
 [6.2]

where  $M_i$  is the mass flow rate of each stream *i* and  $A_i^k$  is the assay for each component *k* in stream *i*. All of the results obtained from the laboratory characterization tests must satisfy these expressions.

Although conceptually very simple, consistent mass balances can be difficult to obtain when conflicting (over-defined) data sets are evaluated. This occurs when redundant streams are sampled or when multiple independent assays (activities) are available for the same sample. For example, activities for each particulate stream may result in different (but equally valid) estimates of mass distributions. These differences are due to experimental errors associated with process fluctuations, sampling techniques and laboratory procedures. These experimental errors are generally assumed to be randomly distributed.
One method of resolving the mass balance dilemma is to develop a self-consistent data set which satisfies the mass balance criteria given by Equations [6.1] and [6.2]. This procedure must be performed such that the minimum total adjustment is made to the measured data set. Mathematically, this can be achieved by minimizing the weighted sum-of-squares (WSSQ) given by:

$$WSSQ = \sum_{k=1}^{c} \sum_{i=1}^{m} \frac{(A_i^{k^*} - A_i^k)^2}{(S_i^k)^2} + \sum_{i=1}^{m} \frac{(M_i^* - M_i)^2}{(S_i)^2} = 0$$
[6.3]

where  $S_i^k$  and  $S_i$  are standard deviations of the measured assay values and measured mass flow rates, respectively. The superscript \* is used to distinguish estimated values from experimental values. In the present work,  $S_i^k$  and  $S_i$  are assumed to be proportional to two-sigma errors obtained from the radionuclide measurements.

Several mathematical techniques can be used to perform the mass balance minimization. These include the use of Lagrangian multipliers (WIE79) and direct search techniques (MUL79). Commercially available computer programs have been developed and marketed for this purpose. However, these programs are costly, incompatible with other software packages, require user training, and have capabilities which exceed the needs of this project. To overcome these shortcomings, a spreadsheet-based mass balance program has been developed in the present work for the analysis of the soil washing test data. The program was developed using Microsoft Excel<sup>TM</sup> for Windows 95<sup>TM</sup>. A disk containing the program has been included with the master copy of this report. The mass balance spreadsheet has been configured to handle up to 20 streams and up to 10 nodes (20x10). The spreadsheet provides a convenient, user-friendly interface for entering experimental data and displaying numerical output.

#### 6.2.2 Procedure

All of the characterization data from the sieve analysis and density partitioning were subjected to the mass balance procedure. The mass balances were performed as follows. First, the connection matrix for the given characterization test was entered in the first page of the spreadsheet. The names of each node and stream were manually entered and +1, 0 or -1 were entered to indicate the interconnections. The next page of the mass balance spreadsheet was used to enter the experimentally measured mass distributions and activities. Two separate sets of activities were entered for the actinide (i.e., Th-228, Th-230, and Th-232) and gamma (i.e., Pb-

214, Ra-228, Bi-214 and Pb-212) data. The actinide data for uranium (i.e., U-234, U-235, and U-238) were also included in the mass balances conducted for sieve analyses. In all cases, the experimental mass distributions were assumed to be reliable and were not adjusted. This approach was reasonable since good sieve recoveries were achieved in all cases. In addition, the head activities for the overall composite soil sample were assumed to be reliable and were not adjusted. A third spreadsheet page was used to enter the two-sigma errors for the remaining activity measurements. Very small errors of essentially zero (i.e., <0.00001) were entered in the first column and first row of each page to prevent adjustments to the sieve mass distributions and head activities.

In order to obtain the "best" estimates of the remaining activities, the total weighted sum-ofsquares (WSSQ) given by Equation [6.4] must be minimized. The value of WSSQ is given at the bottom of the fourth page of the mass balance spreadsheet page. The individual cells on this page represent the weighed sum-of-squares for each estimate. For reference, the relative change in the measured values (expressed as a percentage) are also provided on the right-hand side of this page. In the Excel<sup>TM</sup> spreadsheet, the WSSQ minimization is conducted using a built-in tool known as SOLVER. After SOLVER identifies a solution, the updated estimates of the mass flow distributions and activities are displayed on the right side of second spreadsheet page. To assist in the comparison of the measured and adjusted values, the percentage change is shown on the right side of the fourth spreadsheet page. These values are considered to be the best statistical estimates of the true activities of the soil samples. Data sets which required little adjustment were deemed to be reliable, while those requiring large adjustments were considered to be less reliable.

## 6.2.3 Results and Discussion

#### 6.2.3.1 Sieve Balances

All of the characterization data obtained from the sieving tests were analyzed using the spreadsheet-based mass balance program. The results of these analyses are summarized in Appendix D. In order to illustrate the degree of data adjustment required, the experimentally measured activities were plotted as a function of the estimated (i.e. mass balanced) activities for each size fraction. In these plots, a perfect mass balance would be represented by a straight line passing through the origin with a slope of one.

Figures 6-1 to 6-3 show that excellent balances were generally obtained for both the actinide and gamma data. Only small adjustments to the data sets were typically required to obtain reasonable balances. Problems associated with poor balances were overcome by correcting data entry errors or by repeating analytical measurements. The only poor balance which could not be corrected was obtained for U-235. This difficulty was attributed to the small magnitude of the U-235 activities (<0.3 pCi/g). The only other data points which required any adjustments were found to correspond to the minus 400 size fraction. Fortunately, the activity of the minus 400 mesh material had relatively little impact on the final design of the process flowsheet since the ultrafine particles were difficult to treat and would require off-site disposal. The generally good balances suggest that the activity measurements and test procedures used to generate the sieved samples were reliable.

#### 6.2.3.2 Float-Sink Balances

Mass balances were also conducted for each of the samples obtained from the float-sink (density partitioning) testing of the 4x8, 8x16, 16x30, 30x50 and 50x100-mesh size fractions. The results of these analyses are summarized in Appendix E. In order to maintain an internally consistent set of data, the head activities for each series of float-sink tests were obtained from the mass balances of the sieving test data. The experimentally measured activities were then plotted as a function of the estimated activities in order to evaluate the suitability of the mass balance. Separate plots were generated for actinide (Th-228, Th-230, and Th-232) and gamma (Pb-214, Ra-228, Bi-214, and Pb-212) data for each of the five size fractions subjected to float-sink testing. As before, a perfect mass balance would be represented by a straight line passing through the origin with a slope of one. Since the uranium concentrations were determined to be below the clean-up criteria for all but the finest size fractions, no analytical determinations were made for these actinides in the float-sink test samples.

Figures 6-4 to 6-8 show the mass balances for the float-sink actinide data. The data adjustments were larger than those required to balance the data from the sieve tests. Good balances were obtained for the 4x8 and 8x16 mesh size fractions, while average balances were obtained for the 16x30 and 50x100 mesh fractions. All of these balances were considered to be reasonable given the experimental difficulties associated with accurate measurements of activities. Unfortunately, a very poor balance was obtained for the 30x50 mesh size classes. Therefore, it was necessary to rely on the estimated values of activity for this particular sample. The difficulty in obtaining a consistent mass balance have been attributed to the large amount of sample handling and partitioning required to generate the float-sink test data.



Figure 6-3: Comparison of measured and estimated activities (gamma) obtained after the mass balance procedure for the sieve characterization test data.



Figure 6-4: Comparison of measured and estimated activities (actinide) obtained after the mass balance procedure for the 4x8 mesh float-sink test data.



Figure 6-5: Comparison of measured and estimated activities (actinide) obtained after the mass balance procedure for the 8x16 mesh float-sink test data.



Figure 6-6: Comparison of measured and estimated activities (actinide) obtained after the mass balance procedure for the 16x30 mesh float-sink test data.



Figure 6-8: Comparison of measured and estimated activities (actinide) obtained after the mass balance procedure for the 50x100 mesh float-sink test data.

The mass balances for the float-sink gamma data are shown in Figures 6-9 to 6-13. As shown, the balances for the gamma data were similar to those obtained for the actinide data. The 4x8, 8x16, and 30x50 mesh size fractions were found to mass balance relatively well. Some degree of scatter was noted for the Ra-228 values for the 4x8 mesh data. In contrast, the mass balance for the 16x30 mesh particles was found to be the most difficult to balance and, hence, the least reliable data. The remaining balances were generally acceptable, although a few outlier points were noted. To date, a suitable explanation for the poor balances could not be identified. However, it was noted that many of the density fractions had large two-sigma errors which made the back-calculation of the head activities difficult. Since errors tend to be cumulative, good mass balances may simply be difficult to obtain when a large number of weight fractions and activities having large two-sigma errors are mathematically recombined. This problem may be responsible for the larger adjustments that were generally required to provide consistent mass balances for the density separations.

#### 6.3 Process Simulations

# 6.3.1 Background

Two types of information are needed to identify the most appropriate processing strategy for treating contaminated soils, i.e., property data and efficiency data. In the present work, property data was provided by the characterization tests described in earlier sections of this report. These characterization tests defined the intrinsic properties of the feed soil in terms of the occurrence of radionuclides as functions of particle size, particle density and magnetic susceptibility. These data represent the separation performance that may be achieved using "ideal" separation processes which are perfectly efficient. In other words, the property characterization data provide a convenient measure of the "ultimate" cleanability that should be expected for one or more physical separation processes. Characterization data of this type have the advantage that they depend solely on the inherent properties of the given sample. As a result, the information obtained from property characterization tests are independent of the process eventually used to achieve the desired separation.

Because of inefficiencies associated with "real-world" separation processes, it is rarely possible to achieve the ideal separations predicted by property characterization data. Therefore, a second type of information which characterizes the efficiency of the separation process must be obtained. This information is typically provided in the form of an efficiency plot commonly referred to as a partition curve. A partition curve is simply a plot of the desired separation property (e.g., particle



Figure 6-9: Comparison of measured and estimated activities (gamma) obtained after the mass balance procedure for the 4x8 mesh float-sink test data.



Figure 6-10: Comparison of measured and estimated activities (gamma) obtained after the mass balance procedure for the 8x16 mesh float-sink test data.



Figure 6-11: Comparison of measured and estimated activities (gamma) obtained after the mass balance procedure for the 16x30 mesh float-sink test data.

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Figure 6-12: Comparison of measured and estimated activities (gamma) obtained after the mass balance procedure for the 30x50 mesh float-sink test data.

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Figure 6-13: Comparison of measured and estimated activities (gamma) obtained after the mass balance procedure for the 50x100 mesh float-sink test data.

size or specific gravity) as a function of the mass fraction of particles reporting to a given product stream. The partition curve for an ideal separator would be represented by a step function which runs parallel to the abscissa at the specific value of the separation property. Less efficient separators would result in the misplacement of particles and would be represented by an S-shaped curve which deviates from the ideal curve. Over normal operating ranges, partition curves are generally assumed to be independent of the inherent properties of the particles. Therefore, the shape of the partition curve is largely dictated by the nature of the separation process (i.e., each unit operation has its own unique family of partition curves).

The use of partition curves allows realistic predictions to be made for the mass recovery and quality of soil samples treated by different types of separators. Partition curves may be obtained directly from experimental test programs conducted with the unit operation under consideration. Although more accurate, this experimental approach is very costly and time consuming. Fortunately, the partition curve data can in many cases be obtained from existing data bases of empirical expressions developed from extensive laboratory and field tests. These expressions can be readily incorporated into computer-based simulation programs that are capable of predicting realistic levels of separation performance. These programs require only property characterization data and significantly reduce the amount of experimental work required to establish the most attractive process flowsheet.

## 6.3.2 Procedure

The property characterization data described in previous sections of this report indicate that the most promising methods for treating soil samples from the Maywood site are particle sizing/classification and density separation. As a result, a spreadsheet-based simulation program has been developed in the present work to evaluate the technical performance of these processes.

### 6.3.2.1 Size Separation Modeling

Property data obtained from the sieving tests have been used to characterize the association of various radionuclides within different size fractions of the feed soil. These ideal separation results, which have been discussed extensively within earlier sections of this report, cannot be achieved in actual practice due to process inefficiencies. The actual separation performance in terms of oversize mass recovery  $(M_0)$  and activity  $(A_0^k)$  for each radionuclide (k) can be calculated from:

$$M_{o} = \sum_{i=1}^{n} P_{i}^{s} M_{i}$$
 [6.4]

$$A_{o}^{k} = \frac{\sum_{i=1}^{m} P_{i}^{s} M_{i} A_{i}^{k}}{\sum_{i=1}^{m} P_{i}^{s} M_{i}}$$
[6.5]

where  $P_i^s$  is the sizing partition factor,  $M_i$  the mass fraction and  $A_i^k$  the activity of each radionuclide k present in size fraction i. The magnitude of  $P_i^s$  has been estimated in the present work from the generalized classification curve identified by Heiskanen (HIE93). This curve can be mathematically represented by:

$$P_{i}^{s} = (1 - R_{f}) \left[ 1 - \exp\left\{ 1 - 0.63 \left( \frac{D_{i}}{D_{50}} \right)^{\beta} \right\} \right] + R_{f}$$
[6.6]

where  $R_f$  is the by-pass correction factor,  $D_i$  the mean particle diameter of size fraction *i*,  $D_{50}$  the cut-point particle size, and  $\beta$  is the size separation sharpness index (HEI93). In this case,  $P_i^s$  is defined as the mass fraction of particles of size *i* that report to the coarse product of the sizer.

# 6.3.2.2 Density Separation Modeling

In some cases, particle size separations are incapable of producing acceptable volume reductions at the specified clean-up criteria. Property data obtained from the petrographic studies and float-sink tests suggest that further improvements in the separation performance can be obtained using density concentrators to extract radioactive contaminants present as heavy minerals from the various size fractions. The separation performance of density separators can be calculated from the property characterization data and efficiency curves available in the literature. In this case, the mass recovery  $(M_0)$  and activity  $(A_0^k)$  of the cleaned soil for each radionuclide (k) can be calculated by:

$$M_{o} = \sum_{i=1}^{n} P_{i}^{s} P_{ij}^{d} M_{ij}$$
[6.7]

$$A_{o}^{k} = \frac{\sum_{i=1}^{m} \sum_{j=1}^{n} P_{i}^{s} P_{ij}^{d} M_{ij} A_{ij}^{k}}{\sum_{i=1}^{m} \sum_{j=1}^{n} P_{i}^{s} P_{ij}^{d} M_{ij}}$$
[6.8]

where  $P_i^s$  is the sizing partition factor,  $P_{ij}^d$  the density partition factor,  $A_{ij}^k$  is the activity of each radionuclide k, and  $M_{ij}$  the mass fraction of particles present in each size fraction i and density fraction j. In this case,  $P_{ij}^d$  is defined as the mass fraction of particles of size i and density j that report to the clean soil product.

As before, the magnitude of  $P_i^s$  has been estimated in the present work using Equation [6.6]. Likewise,  $P_{ij}^d$  has been estimated from the Lynch-Rao (LYN77) equation given by:

$$P_{ij}^{d} = \frac{\exp\left\{\alpha \frac{SG_{j}}{SG_{50}}\right\} - 1}{\exp\left\{\alpha \frac{SG_{j}}{SG_{50}}\right\} + \exp\{\alpha\} - 2}$$
[6.9]

where  $SG_j$  is the mean specific gravity of particles in density class *j* and size fraction *i*,  $SG_{50}$  the cut-point specific gravity, and  $\alpha$  is the density separation sharpness index. Processes of higher efficiency are identified by larger  $\alpha$  values. Typical  $\alpha$  values can be estimated from:

$$\alpha = 1.089 \frac{SG_{50}}{E_p}$$
[6.10]

in which Ep is the Ecart probability (KAW95). Larger values of Ep are indicative of less efficient processes. In many cases, Ep values may be reported by equipment manufacturers using an imperfection index (1) defined by:

$$I = \frac{E_p}{SG_{50}} \tag{6.11}$$

Extensive studies conducted in Australia by Woods (WOO95) and others indicate that suitable estimates of *Ep* for density-based separators can be obtained from:

$$E_p = \frac{K}{D_i}$$
[6.12]

in which K is an efficiency constant for a given type of separation device. In the present work, a K factor or 0.08 has been used to represent typical water-based density separation processes. Equation [6.12] indicates that the partition factors  $(P_{ij}^{d})$  for density-based separation processes are a function of both particle size (i) and density (j). As should be expected, finer particles are separated less efficiently than coarser particles due to the smaller Ep values.

# 6.3.2.3 Simulator Development

Equations [6.4]-[6.12] have been used in the present work to develop a spreadsheet-based simulation program for evaluating the cleanability of soils containing radionuclides. The program was developed using Microsoft Excel<sup>TM</sup> for Windows 95<sup>TM</sup> (Version 7.0 or later). The spreadsheet format provides a convenient, user-friendly interface for entering experimental data, performing calculations and displaying numerical output. A disk containing a source-code for the simulation program is provided with the master copy of this report.

Simulations are carried out using the following procedure. First, the user enters the mass distribution of particle sizes and densities obtained from property characterization tests. The radionuclide content of each size and density fraction must also be entered. Data entry is achieved using the first workbook page of the simulation spreadsheet. Next, the user enter relevant design parameters or operating conditions for each separation process. This information is entered using the second workbook page of the simulation spreadsheet. At present, the spreadsheet has been configured to include a primary particle sizing unit, a secondary classification unit, and two stages of density concentrators. Some of the float-sink data indicate that radionuclide contaminants may be present within low-density particles containing gas or organic inclusions. Therefore, the density separators have been configured so that the first unit can reject the high-density particles (such as monazite), while the second unit can reject low-density particles (containing inclusions).

The user can select specific types of sizing devices or density separators by entering the appropriate values which define the partition curve for the particular unit. Values which must be input for the primary sizer include the sharpness index ( $\beta$ ), bypass factor ( $R_f$ ) and the particle size cut-point ( $D_{50}$ ). The same types of information must be entered for the secondary classifier. Values entered for the density separators include the efficiency constant (K) and the specific

gravity cut-point  $(SG_{50})$ . If desired, simulation runs can be conducted under ideal conditions by entering the appropriate responses. For convenience, a third workbook page has been incorporated into the spreadsheet to graphically display the partition curves for each operation.

After completing the data entry procedures, the simulation program automatically calculates detailed information related to the separation performance of each unit operation. These include the mass rate of each product and concentrations of various radionuclides. The data from the individual unit operations are then mathematically combined and used to calculate the amounts and qualities of the clean and contaminated products that would be generated by the overall soil treatment circuitry. For convenience, a summary page is provided at the top of the second spreadsheet page which summarizes the results obtained by the following circuit configurations:

(A) the primary classifier only,

- (B) the combined primary and secondary classifiers,
- (C) the combined primary and secondary classifiers with a single stage of density separation, and
- (D) the combined primary and secondary classifiers with a two stages of density separation.

In each case, the total amount of clean product soil recovered and the concentrations of all relevant radionuclides are reported on the summary page.

The simulation approach offers many potential advantages. First, the simulator allows the user to compare the performance of different unit operations and circuit configurations and to establish the impact of different operating modes on separation performance. The simulator can provide consistent results with a rapid turn-around, resulting in substantial savings in analysis time, manpower and cost. Second, the simulator allows the cleanability of other soil samples to be evaluated with a minimum of experimental testing. In essence, the simulator will provide a vehicle for extrapolating the findings of this study into other soil treatment projects.

#### 6.3.3 Results and Discussion

# 6.3.3.1 Equipment Selection

The first series of simulation runs were performed to identify the types of equipment that would be required to meet the clean-up objectives. In these simulations, it is assumed that the feed soil has been vigorously agitated to completely disperse all particles. Previous studies by SC&A indicate that the dispersion may be achieved using a rotary tumbler or trommel. A similar approach is recommended in the current flowsheet design.

The primary particle sizing unit was evaluated over a wide range of values for the sharpness index  $(\beta)$  and bypass factor  $(R_f)$ . The particle size cut-point  $(D_{50})$  of 4.75 mm was selected for the primary sizer and held constant in all simulations. Figure 6-14a and 6-14b show the impact of variations of  $\beta$  and  $R_f$  on the Th-232 and Pb-214 activities, respectively, for the Maywood soil. The simulations show that the  $\beta$  has a relatively smaller impact on the activities. A value of 10 or larger was found to have little impact on the separation performance. This value as also deemed to be obtainable using one of several commercially available sizing devices. On the other hand, the simulations show that even a small increase in the  $R_f$  value has a dramatic adverse impact on the resulting activities. This implies that the bypass of fines to the coarse product cannot be tolerated in the primary sizing unit. Therefore, to meet these requirements, a multi-deck vibrating screen was selected due to its high efficiency and low cost per unit capacity. The screen will need to have an adequate retention time and a well designed fresh water spray system to ensure that the all particles are completely rinsed of fines prior to discharge of the coarse product. Failure to do will result in an increase in  $R_f$  and an significant increase in the activities for the product from the screening operation.

Simulation runs for the secondary classifier are shown in Figures 6-15a, 6-15b, 6-16a, and 6-16b. In this case, simulations were performed to show variations in the Th-232 and Pb-214 activities as functions of particle size cut-point ( $D_{50}$ ) for different  $\beta$  and  $R_f$  values. As expected, a reduction in the cut-size ( $D_{50}$ ) resulted in a rapid increase in activities, particularly below 1 mm. An increase in the sharpness index ( $\beta$ ) above approximately 10 also increased the activities. More importantly, the simulations show that even small amounts of bypass (as indicated by nonzero values of  $R_f$ ) result in an unacceptable increase in the activities, especially Th-232. In fact, the Th-232 activity was increased from about 3.9 to 4.9 pCi/g by allowing just 1 percent of the material to bypass this sizing step. None of the commercially available screening devices were deemed to be suitable for the secondary sizer because of the tight tolerance on misplacement and



Figure 6-14a: Influence of sharpness index ( $\beta$ ) and bypass factor ( $R_f$ ) on Th-232 activity for primary particle sizing.



Figure 6-14b: Influence of sharpness index ( $\beta$ ) and bypass factor ( $R_f$ ) on Pb-214 activity for primary particle sizing.



Figure 6-15a: Effect of particle size cut-point ( $D_{50}$ ) and sharpness index ( $\beta$ ) on Th-232 activity for secondary classification.



Figure 6-15b: Effect of particle size cut-point ( $D_{50}$ ) and sharpness index ( $\beta$ ) on Pb-214 activity for secondary classification.



Figure 6-16a: Effect of particle size cut-point  $(D_{50})$  and bypass factor  $(R_f)$  on Th-232 activity for secondary classification.



Figure 6-16b: Effect of particle size cut-point  $(D_{50})$  and bypass factor  $(R_f)$  on Pb-214 activity for secondary classification.

the smaller cut-size required to achieve reasonable clean soil recoveries. Available alternatives include spiral classifiers, hydrocyclones (single and multi-stage) and hindered-bed hydroclassifiers.

The simulation data indicate that a single-stage hydrocyclone would not be capable of producing an acceptable separation due to the high bypass ( $R_f > 0.2$ ). In addition, a typical imperfection index (1) of 0.3-0.4 for a single-stage hydrocyclone is lower than would be desired for this separation. Multiple stages of serially-connected hydrocyclones are capable of significantly reducing the bypass ( $R_f < 0.05$ ) and improving the imperfection (0.15-0.20). However, nearly complete elimination of ultrafine particles is necessary to maximize contaminant removal and clean soil recovery. The must suitable unit for achieving this separation would be one of several hindered-bed hydroclassifiers such as the Floatex, Linatex or Stokes units. These devices use a counter-current flow of fresh wash water to remove fines which often short-circuit to the coarse particle stream. As a result, these devices are capable of providing a bypass factor  $(R_f)$ approaching zero. In addition, a well operated unit can achieve very sharp separations with an imperfection index (1) in the range of 0.10. This corresponds to a sharpness index ( $\beta$ ) of approximately 10. Hydroclassifiers offer relatively low capital and operating costs, although the clarified water consumption is high compared to other fine sizing devices. The high water consumption will require that the thickener be properly designed to offer adequate clarified water capacity.

A variety of devices are available to provide the desired density separation. These include pneumatic jigs, flowing film concentrators (mineral tables and spirals), cyclones (water-only and dense-media), and centrifugal gravity concentrators. Water-only cyclones are too inefficient and the ancillary units required to support dense-media cyclones are too costly. Centrifugal gravity concentrators such as the Multi-Gravity Separator, Falcon Concentrator and Knelson Concentrator are not suitable for treating the large particle topsize that occurs in the feed stream. Therefore, these units were not considered in the present flowsheet. A pnuematic jig would be an acceptable choice if the final clean-up level dictates that only the coarsest particle size fractions be recovered. However, a more attractive option would be to utilize flowing-film separators such as tables or spirals. These units are generally efficient over a relatively wide particle size range (4-0.1 mm), require little in terms of ancillary equipment, and are ideally suited to treat the high-solids feed slurry prepared by the hydroclassifier. In addition, small heavy particles which report with the coarser light particles in the present design, mineral spirals are recommended over tables since they offer a significantly lower cost per unit capacity. Figure 6-17 shows the conceptual process flowsheet for the proposed circuitry. The circuit includes a dispersion operation (rotary scrubber), a primary sizing unit (screen), a secondary classifier (hydroclassifier) and density separator (spiral module). The interconnections for all key ancillary equipment are also specified in the conceptual flowsheet.

# 6.3.3.2 Performance Optimization

After completing the baseline equipment selection, an additional series of simulation runs were conducted to optimize the circuit performance. Parameters examined in the optimization runs included the particle size cut-point for the secondary particle classifier (hydroclassifier) and the specific gravity cut-points for each of the density separators (spirals).

Two circuit configurations were found to provide adequate levels of radionuclide removal to meet the desired clean-up criteria. The first configuration (Circuit I) includes only particle sizing/classification, while the second (Circuit II) includes an additional step of density separation. A third configuration (Circuit III) which included two stages of density separation was incapable of further improving the separation performance. Therefore, the results of these simulations are not reported in the present work.

Figures 6-18 and 6-19 show the mass of clean soil recovered under optimum separation conditions as a function of the Th-232 activity for Circuit I and II (these plots differ only by the scale of the x-axis). These plots account for the site background Th-232 activity of 1 pCi/g. At Th-232 activities greater than 5 pCi/g, there were no difference in mass recovery between Circuit I and II. However, the data show that a significant decline in mass recovery from 50 percent to 25 percent occurs for sizing and classification alone at a Th-232 activity of approximately 4.5 pCi/g. This sharp decline was attributed to the rejection of intermediate sand-sized particles that were contaminated at levels just about the stated Th-232 activity. These particles responded well to density treatment and, as a result, mass recoveries of 45-50 percent could be maintained down to Th-232 activities of about 2.5-4.5 pCi/g by utilizing Circuit II (sizing and density separation). In fact, only small improvements in soil recovery were obtained using the density separator for Th-232 activities greater than 4.5 pCi/g. A mass recovery of 52 percent was achieved using Circuit I and II, respectively, when operating at a Th-232 activity of 5pCi/g.

Figure 6-20 shows the mass recovery as a function of Pb-214 (Ra-226) activity. The Pb-214 plot shows that a mass recovery of 100 percent was achieved operating at a Pb-214 activity of 4.16



Figure 6-17: Conceptual flowsheet for the proposed soil treatment facility incorporating primary sizing, secondary classification and density separation.

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Figure 6-18: Simulated relationship between mass recovery (%) and Th-232 activity for Circuit I (sizing and classification) and Circuit II (sizing, classification and density separation).



Figure 6-20: Simulated relationship between mass recovery (%) and Pb-214 activity for Circuit I (sizing and classification) and Circuit II (sizing, classification and density separation).

pCi/g, which is below the target 5 pCi/g. This was expected since the activity for the Pb-214 was already near or below 5 pCi/g in the feed soil (i.e., 5.16 pCi/g for Pb-214). In contrast, the Th-232 in the feed soil was significantly higher at 25.3 pCi/g. Thus, achieving the clean-up criteria for a treated soil from the Maywood site appears to be primarily controlled by the recovery and/or rejection of particles containing Th-232.

Because of the importance of Th-232 in meeting the clean-up criteria, an additional series of optimization runs were performed in which the soil recovery was maximized at different levels of Th-232 activity. The results of these simulations are also shown in Figures 6-18 and 6-19 (these plots differ only by the scale of the x-axis). The simulation data show that only about 52 percent of the soil can be recovered at a Th-232 activity of 5 pCi/g. However, the mass recovery of clean soil can be increased to about 68 percent by increasing the Th-232 activity to 13.5 pCi/g. A further increase in Th-232 activity is not technically feasible with the current circuit configurations. To recover additional mass, the secondary classifier cut-point (D<sub>50</sub>) must decrease. This is because 36 percent of the feed soil is below 200 mesh. However, there are no separation unit operations that will achieve a cut-point below 200 mesh, especially at the  $\beta$  and  $R_f$  values that are needed. The data suggest that the best operating point for reducing the Th-232 activity while maximizing soil recovery would be to produce a clean soil with a Th-232 activity of approximately 5 to 13 pCi/g. These values correspond to just above the "knee" in the recovery-activity plots. Figure 6-19 shows that these operating points would result in mass recoveries of approximately 55 and 66 percent. These values could be achieved using either Circuit I or II.

For reference, process flow diagrams showing the optimum configurations to achieve the desired Th-232 activity of 6 pCi/g (5 + 1 pCi/g background) are provided in Figure 6-21 for Circuit I and Figure 6-22 for Circuit II. The complete spreadsheet output from these two series of simulation runs are provided in Appendix F and G, respectively. For reference, plots of the partition curves for primary sizing, secondary classification and density separation have also been included.

# 6.4 Economic Feasibility

# 6.4.1 Background

The test results obtained under this project indicate that a significant portion of the contaminated soil from the Maywood site can be successfully cleaned and recovered using physical separation processes (i.e., particle size and density separators). In order to assess the economic feasibility of



## Maywood Composite #1 - Optimum Screen and Hydroclassifier

Figure 6-21: Process flow diagram showing the optimum configuration for Circuit I (sizing and classification) required to achieve Th-232 = 5 pCi/g (6 + 1 pCi/g background).



Maywood Composite #1 - Optimum Screen, Hydroclassifier and Density Separator

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Figure 6-22: Process flow diagram showing the optimum configuration for Circuit II (sizing, classification and density separation) required to achieve Th-232 = 5 pCi/g (6 + 1 pCi/g background).

this approach, a cost-benefit analysis of the proposed circuitry was conducted using the conceptual flowsheet design. Two series of economic analyses were performed. The first analysis was conducted to evaluate the cleanup of the contaminated soil to a total Th-232 activity of 5 pCi/g (6 pCi/g including background). The second analysis was performed assuming a relaxed cleanup target for Th-232 of 15 pCi/g (16 pCi/g including background). As discussed previously within this report, the activity of Th-232 was found to be the major limitation in meeting the soil cleanup criteria. The activities of the remaining radioisotopes of interest were found to fall below the established cleanup targets when the activity of Th-232 was reduced to the specified cleanup levels.

Items addressed in the preliminary economic evaluation included:

- summary of estimated capital costs for the complete installation of the full-scale treatment plant circuitry and any required ancillary operations including activities related to procurement, construction, and fabrication of the facility,
- summary of expected operation and maintenance costs including electrical power, makeup water, chemical reagents, replacement parts, soil handling/disposal, and other miscellaneous consumables, as well as labor costs, skilled and unskilled, to operate and maintain the soil treatment facility, and
- cost-benefit analysis that specifies the expected savings based on the soil treatment and remaining disposal costs.

## 6.4.2 Procedures

# 6.4.2.1 Baseline Assumptions

All of the cost analyses were performed for a production-scale circuit incorporating a primary sizing unit (screen), secondary classifier (hydroclassifier), density separator (spiral module) and water clarifier (thickener). Results obtained from the optimized simulation runs were used to provide the necessary data for the economic analyses. A summary of the circuit performance data and associated economic calculations are provided in Appendix H. The spiral module was included in all of the economic analyses despite the fact that the simulation results indicated that a density separation step would not be necessary to meet a relaxed cleanup target of 15 pCi/g for Th-232. Since the spiral module added little to the overall cost of the circuit, this backup system

was included to serve as a "safety net" to reject isolated pockets of contaminated heavy mineral sands which may enter the plant during the excavation of "hot spots" in the host soil.

The economic analyses were performed to address the costs associated with the partial excavation, treatment and disposal of a minimum of 62,100 tons (46,000 cubic yards) of contaminated soil from the Maywood site up to the complete excavation, treatment and disposal of all 550,845 tons (408,033 cubic yards) of contaminated soil estimated to be present at the Maywood site. Costs related to the excavation/loading (\$15.19/ton), transportation (\$119.72/ton) and disposal (\$156.00/ton) of the contaminated soil were provided by SAIC. Costs associated with the purchase and haulage of backfill material were estimated to be \$6.50/ton, with an additional fee of \$2.10/ton for backfill placement, landscaping and revegetation. For calculation purposes, the feed soil was assumed to enter the plant with a total moisture content of 10% by weight. The moisture contents of the total cleaned soil product and contaminated waste product were assumed to be 8% and 20% by weight, respectively. All tonnage values involved in the excavation, transportation, treatment and disposal activities have been properly corrected to account for added weight due to moisture content.

For the purpose of this study, the feed rate to the treatment facility was maintained below 75 tph (dry basis) since a larger plant size was not considered practical due to site limitations and constraints on equipment availability. The facility was assumed to operate two 8-hour shifts per day for 20 days per month, i.e., 320 hours/month. A 62% safety factor on productivity has been assumed. The cost analyses were conducted for a variable plant life of 6-36 months in 6 month increments. An annual inflation rate of 4 percent was assumed and, in order to be conservative in the estimation, the full debt load was carried forward for all months of operation. As is typical for small mineral processing plants, the salvage value of the plant was assumed to be equivalent to the costs associated with the decommissioning and disposal of the plant equipment and hardware. No tax payments or depreciation factors were considered in the economic analysis. Costs associated with analytical support and radiological/safety support were also not considered in the economic analyses. In practice, these cost elements are expected to be somewhat higher for site treatment and partial disposal than for complete disposal due to the greater number of process streams that must be sampled during treatment.

# 6. 4.2.2 Estimation of Capital Costs

In order to estimate the capital costs of the proposed circuits, preliminary scale-up projections were made for a mid-range 50 tph (dry basis) treatment facility. These calculations indicated one

6x4 ft multi-deck screen and one 4x4 ft square hydroclassifier would be required to handle the desired production rate. The cost of the screen, including as associated controls, was estimated to be \$21,500. Likewise, the cost of the hydroclassifier unit was estimated to be \$14,000. A module containing four twin-turn mineral spirals was also required for the proposed density separation. This module was estimated to cost \$9,500. In addition to these soil cleaning units, several key ancillary operations were also included in the listing of capital costs. These included a feed hopper-conveyor (\$36,000), rotary scrubber (\$28,000), thickener (\$52,000) and filter press (\$122,000). A variety of miscellaneous items such as sumps, pumps, bins, meters, etc., were also included in the capital cost estimation. A complete listing of these items is provided in Appendix H.

All equipment estimates were provided by verbal vendor quotes and are believed to be reliable. Capital cost estimates for equipment sizes other than 50 tph were calculated using the "seventenths rule" which states that the ratio of capital costs for different sized units is proportional to the ratio of equipment capacities raised to the 0.7 power. Installation cost was estimated by multiplying the total equipment cost by a factor of 1.1. These formula are routinely used by fabricators in the mineral and coal processing industries for conducting preliminary cost estimations of process circuitry. Other costs considered in the capital estimation included a 15 percent fee for engineering and an overhead rate of 20 percent.

# 6.4.2.3 Estimation of Operating Costs

Annual operating costs were estimated for the 50 tph (dry basis) plant. Elements included in the operating costs were power consumption, equipment maintenance, personnel and miscellaneous consumables (i.e., reagents, lubricants, etc.). Electrical power consumption was estimated for the feed conveyor (12 HP), rotary scrubber (25 HP), primary screen (20 HP), thickener (12 HP), filter press (40 HP), and transfer pumps (4 x 7.5 HP). These values were adjusted as deemed appropriate using the seven-tenths rule. For the primary unit operations, a power load factor of 80 percent was used to estimate actual power requirements, while a power factor of 15-20 percent was used for the instrumentation and small reagent pumps. Power costs were estimated at an industrial rate of 0.05/kW-hr.

The treatment circuit was assumed to require one operator (\$65,000/yr), one mechanic/electrician (\$45,000/yr) and one administrative support person (\$50,000/yr) for each working shift. In addition, one technician position (\$35,000 each) was added for each 25 tph (dry basis) of feed tonnage feed to the plant for each working shift. All positions were assumed to be full-time with

benefits. Fringe benefits were estimated as 50 percent of the base salary. Pay scales were estimated for a working year of 2,080 hours per employee.

The major consumable item included in the annual operating costs was the clarification reagents (i.e., flocculant @ \$0.90/lb). Lubrication costs were estimated to be \$0.01/ton of feed. Make-up water consumption was estimated by doubling the water consumption calculated from the differences in the moisture contents of the feed soil and treated products. Water and sewage rates have been assumed to be \$2.50 per 1000 gallons of water/sewage handled. Miscellaneous annual equipment maintenance costs were calculated as 10 percent of the total capital cost of the proposed circuitry. This guideline has been found to provide reasonable estimates of maintenance costs for small-to-medium scale processing plants.

#### 6.4.3 Results and Discussion

#### 6.4.3.1 Cost Sensitivity Analysis

The first series of economic analyses for the Maywood site were conducted to evaluate the impact of various cost elements on the total estimated cleanup cost. The first two factors examined in the sensitivity analyses were plant feed rate and operating duration. Figure 6-23 shows the total tonnage of soil treated as a function of the number of months of operation for 25, 50 and 75 tph processing plants. The corresponding data for soil treatment cost (reported as dollars per ton of feed soil processed) are shown in Figures 6-24 and 6-25. These figures were constructed assuming cleanup targets for Th-232 of 5 pCi/g and 15 pCi/g, respectively. Figure 6-23 indicates that the plant will require about 34 months of operation at the maximum capacity of 75 tph in order to cleanup the entire 550,845 tons of contaminated soil at the Maywood site. Likewise, approximately 12 months of operation at 25 tph would be required to treat only a portion of the site (i.e., 62,100 tons). The data given in Figures 6-24 and 6-25 show that the treatment costs would vary between \$143/ton to \$213/ton depending on the particular selection of operating duration, plant capacity and cleanup level. However, the plant should not be operated for a period of less than about 12 months (at any rated capacity) in order to avoid the rapid rise in treatment costs associated with difficulties in recovering the capital investment. This data given in these figures also show that significant savings can be realized by utilizing larger plants (due to the economy-of-scale). Unfortunately, the savings begin to diminish rapidly after a rated feed capacity of approximately 50 tph is exceeded.



Figure 6-23: Calculated tonnage of soil treated as a function of number of months of operation for 25, 50 and 75 tph plant capacities.

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Figure 6-24: Estimated treatment cost required to achieve <5 pCi/g of Th-232 activity as a function of number of months of operation for 25, 50 and 75 tph plant capacities.



Figure 6-25: Estimated treatment cost required to achieve <15 pCi/g of Th-232 activity as a function of number of months of operation for 25, 50 and 75 tph plant capacities.

Since the reliability of the economic analysis depends on the accuracy of the cost estimates, a sensitivity analysis was performed to determine the influence of increases in capital and operation/maintenance costs on overall treatment cost. This analyses were conducted assuming that a mid-range value of 300,000 tons of contaminated soil would be treated. The results of these analyses are shown in Figures 6-26 and 6-27 for Th-232 cleanup targets of 5 pCi/g and 15 pCi/g, respectively. The results from three sets of calculations are given: (1) escalation in capital costs only, (2) escalation in operation/maintenance costs only, and (3) escalation in capital and operation/maintenance costs combined. For the case of meeting the 5 pCi/g target, an escalation in operation/maintenance costs was found to result in a slightly higher rise in treatment cost than an escalation in treatment costs. In contrast, an escalation in capital costs was found substantially more important than an escalation in operation/maintenance costs when attempting to meet a relaxed cleanup criteria of <15 pCi/g of Th-232 activity. Fortunately, an escalation factor of 2 (i.e., doubling of the estimated costs) for both capital and operation/maintenance costs increased the treatment cost for the 5 pCi/g case from only \$182/ton to \$198/ton. Thus, a doubling of the estimated processing cost increased the treatment cost by only \$16/ton (<9%). Likewise, an escalation factor of 2 (i.e., doubling of the estimated costs) for both capital and operation/maintenance costs increased the treatment cost for the 15 pCi/g case from only \$148/ton to \$164/ton. In this case, a doubling of the estimated processing cost increased the treatment cost by only \$16/ton (<11%). These analyses indicate that the major cost drivers for the site cleanup are not strongly dependent on the soil processing cost.

# 6.4.3.2 Estimated Cleanup Costs

The first series of detailed cost analyses for the cleanup of the Maywood soil are summarized in Table 6-1. These calculations were performed assuming that approximately 62,100 tons of soil would require treatment. This required 12 months of plant operation and a feed rate capacity of about 25 tph (i.e., 23.5 tph). The economic analyses show that the estimated cost for complete disposal of 62,100 tons of contaminated soil is \$298.65/ton. Alternatively, site remediation to a total Th-232 activity of <5 pCi/g would reduce this cost to \$198.42/ton, a savings of \$100.23/ton. This cost reduction represents a total savings of approximately \$6.2 million compared to complete disposal without treatment. Under the relaxed cleanup criteria of <15 pCi/g, the cost of site remediation is further reduced to \$162.48/ton, a savings of \$136.17/ton. This cost reduction represents a total savings of approximately \$8.5 million for the treatment of 62,100 tons of soil. According to the values provided in Table 6-1, the major cost elements in the site remediation are the contaminated soil transportation and disposal costs. These items constitute 69-74% of the



Figure 6-26: Influence of an escalation in estimated capital and/or operation and maintenance costs on the treatment of 300,000 tons of contaminated soil for a Th-232 cleanup target of <5 pCi/g.



Figure 6-27: Influence of an escalation in estimated capital and/or operation and maintenance costs on the treatment of 300,000 tons of contaminated soil for a Th-232 cleanup target of <15 pCi/g.

total cost of the site remediation depending on the level of cleanup desired (i.e., 5 or 15 pCi/g). In contrast, processing of the soil by the proposed circuitry represents only 16-19% of the total site remediation cost.

Table 6-2 shows the cost analyses for the remediation of all contaminated soil (approximately 550,845 tons) at the Maywood site. This case required 34 months of operation at a feed rate capacity of about 75 tph (i.e., 73.5 tph). As before, the analysis indicates that complete disposal of the soil without treatment would cost \$298.65/ton. Remediation of the site to a total Th-232 activity of <5 pCi/g would reduce this cost to \$179.69, a savings of \$118.96/ton. Compared to complete disposal, this cost reduction represents a total savings of approximately \$65.5 million. Furthermore, an additional savings of \$154.92/ton can be realized under the relaxed cleanup criteria of <15 pCi/g. In this case, the cost of site remediation is reduced to \$143.73/ton. This cost reduction represents a total savings of approximately \$85.3 million for the treatment of 550,845 tons of soil. Again, the major cost elements in site remediation are the contaminated soil transportation and disposal costs. These items account for 78-82% of the total cost of the site remediation cost.

# 6.5 Conclusions

A conceptual flowsheet for the Maywood FUSRAP site was carried out in the present work using process engineering methods commonly employed in the coal preparation and minerals processing industries. The major conclusions of this effort are as follows:

- Test data obtained from the experimental characterization studies were evaluated using a spreadsheet-based mass balance program. In general, very good mass balances were achieved for the size-by-size (sieving) data. The good balances indicate that the activity measurements and sizing procedures were generally reproducible and reliable. As expected, the mass balances for the float-sink data were generally more variable, although reasonable balances were still achievable.
- The characterization test data for the Maywood site indicated that particle size and density separations could be used to reduce the volume of soil requiring disposal. In order to evaluate these remediation processes, a circuit simulator was developed using process models which are available in the technical literature. Process models were included for primary sizing (screening), secondary sizing (hydroclassification) and

Table 6-1: Economic analysis for partial treatment (62,100 tons) of Maywood site soil.

	Option 1 - No boli Treatment with Complete boli Disposal					
Site	Dry Solids	Moisture	Total Soil	Unit Cost	Total Cost	
Activity	(tons)	(tons)	(tons)	(\$/ton)	(\$)	
Soil Excavation	55,890	6,210	62,100	\$15.19	\$943,299	
Backfill Material Backfill Placement	55,890	0	55,890	\$6.50	\$363,285	
	55,890	0	55,890	\$2.10	\$117,369	
Soil Transportation	55,890	6,210	62,100	\$119.72	\$7,434,612	
Soil Disposal	55,890	6,210	62,100	\$156.00	\$9,687,600	
Totals				\$298.65	\$18,546,165	

Option 1 - No Soil Treatment with Complete Soil Disposal

Option 2 - Soil Treatment to <5 pCi/g Th-232 with Partial Soil Disposal

Site	Dry Solids	Moisture	Total Soil	Unit Cost	Total Cost
Activity	(tons)	(tons)	(tons)	(\$/ton)	(2)
Soil Excavation	55,890	6,210	62,100	\$15.19	\$943,299
Soil Processing	55,890	6,210	62,100	\$31.14	\$1,933,675
Soil Replacement	29,342	2,552	31,894	\$2.10	\$66,977
Backfill Material	26,548	0	26,548	\$6.50	\$172,560
<b>Backfill Placement</b>	26,548	0	26,548	\$2.10	\$55,750
Soil Transportation	26,548	6,637	33,185	\$119.72	\$3,972,871
Soil Disposal	26,548	6,637	33,185	\$156.00	<b>\$5,176,81</b> 1
Totals				\$198.42	\$12,321,944

Option 3 - Soil Treatment to <15 pCi/g Th-232 with Partial Soil Disposal

Site	Dry Solids	Moisture	Total Soil	Unit Cost	Total Cost
Activity	(tons)	(tons)	(tons)	(\$/ton)	(\$)
Soil Excavation	55,890	6,210	62,100	\$15.19	\$943,299
Soil Processing	55 <b>,89</b> 0	6,210	62,100	\$30.67	\$1,904,657
Soil Replacement	35,619	3,097	38,716	\$2.10	\$81,304
Backfill Material	20,271	0	20,271	\$6.50	\$131,763
Backfill Placement	20,271	0	20,271	\$2.10	\$42,570
Soil Transportation	20,271	5,068	25,339	\$119.72	\$3,033,600
Soil Disposal	20,271	5,068	25,339	\$156.00	\$3,952,904
Totals				\$162.48	\$10,090,097

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<b>Option 1 - No Soil Treatment with Complete Soil Disposal</b>					
Site	Dry Solids	Moisture	Total Soil	Unit Cost	Total Cost
Activity	(tons)	(tons)	(tons)	(\$/ton)	(\$)
Soil Excavation	495,761	55,085	550,845	\$15.19	\$8,367,366
Backfill Material	495,761	0	495,761	\$6.50	\$3,222,443
Backfill Placement	495,761	0	495,761	\$2.10	\$1,041,097
Soil Transportation	495,761	55,085	550,845	\$119.72	\$65,947,163
Soil Disposal	495,761	55,085	550,845	\$156.00	\$85,931,820
Totals	-			\$298.65	\$164,509,859

Table 6-2: Economic analysis for complete treatment (550,845 tons) of Maywood site soil.

Option 2 - Soil Treatment to <5 pCi/g Th-232 with Partial Soil Disposal

Site	Dry Solids	Moisture	Total Soil	Unit Cost	Total Cost
Activity	(tons)	(tons)	(tons)	(\$/ton)	(\$)
Soil Excavation	495,761	55,085	550,845	\$15.19	\$8,367,336
Soil Processing	495,761	55,085	550,845	\$12.41	\$6,834,620
Soil Replacement	260,274	22,633	282,907	\$2.10	\$594,104
Backfill Material	235,486	0	235,486	\$6.50	\$1,530,661
Backfill Placement	235,486	0	235,486	\$2.10	\$494,521
Soil Transportation	235,486	58,872	294,358	\$119.72	\$35,240,515
Soil Disposal	235,486	58,872	294,358	\$156.00	\$45,919,816
Totals				\$179.69	\$98,981,547

Option 3 -	Soil Treatm	ent to $< 15  \mathrm{nCi}/$	o Th-232 with	Partial Soil 1	Disposal
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Site	Dry Solids	Moisture	Total Soil	Unit Cost	Total Cost
Activity	(tons)	(tons)	(tons)	(\$/ton)	(\$)
Soil Excavation	495,761	55,085	550,845	\$15.19	\$8,367,336
Soil Processing	495,761	55,085	550,845	\$11.92	\$6,567,499
Soil Replacement	315,948	27,474	343,422	\$2.10	\$721,186
Backfill Material	179,812	0	179,812	\$6.50	\$1,168,780
Backfill Placement	179,812	0	179,812	\$2.10	\$377,606
Soil Transportation	179.812	44,953	224,765	\$119.72	\$26,908,916
Soil Disposal	179.812	44,953	224,765	\$156.00	\$35,063,405
Totals				\$143.73	\$79,172,728

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density separation (spiral separator). The simulation results indicate that primary screening and hydroclassification alone are capable of achieving soil recoveries of 52.5 percent and 63.7 percent at Th-232 values of 5 and 15 pCi/g, respectively. However, the density separation step (spiral module) was included in the circuit to act as a "safety net" to reject contaminated heavy mineral sands which may occur in "hot spots" at the site. The spiral module added little to the overall cost of the circuit. The activities of U-238 and Pb-214 remained below 5 pCi/g for all simulations.

• After completing the technical evaluation, a preliminary cost analysis was conducted to examine the economic feasibility of the proposed circuitry. The cost analysis included a summary of estimated capital and operating costs for the complete installation of both 25 tph and 75 tph treatment facilities to treat 62,100 tons and 550,845 tons of contaminated feed soil, respectively. For the smaller volume of 62,100 tons, the estimated cost for site remediation was found to vary from \$198.42 to \$162.48 per ton of dry feed soil depending on whether the cleanup target was set to 5 pCi/g or 15 pCi/g, respectively. These values imply that a total cost savings of more than \$8.4 million may be realized by combining soil treatment and disposal at the Maywood site. For the greater tonnage of 550,845 tons, the estimated remediation cost was found to vary from \$179.69 to \$143.73 per ton of dry feed soil for the cleanup targets of 5 pCi/g or 15 pCi/g, respectively. In this case, a total cost savings of more than \$85 million may be realized by combining soil treatment and disposal at the Maywood site.

# 7.0 CONCLUSIONS AND RECOMMENDATIONS

## 7.1 Particle-Size Separations

Particle-size separation alone, on Composite 1015, is not capable of achieving a TAR of one while achieving significant volume reduction. Other target goals of less than 5 and 15 pCi/g for Ra-226 and Th-232 are achievable with volume reductions of greater than 60% for a 200-mesh-size cut.

Particle-size separation alone will not be effective in meeting any target goals for Composite 1016.

### 7.2 Petrographic Analysis

# 7.2.1 Composite 1015

Composite 1015 is comprised of a variety of natural and anthropogenic materials. Natural rock and minerals constitute more than half of the particles (54 percent). Radioactive materials and materials suspected to contain radioactivity are believed to be present in about one-third of the sample. Known natural radioactive minerals are monazite and zircon that occur only in the fine sand and coarse silt-size fractions. The monazite averages about 0.028 percent of the soil sample and zircon about 0.07 percent of the soil sample. A substance that potentially could be a higher contributor to the radioactivity in the soil is a white, clay-like, anthropogenic thorium substance that comprises three percent of the soil sample. This material is believed to be a thorium orthophosphate. Coal-fired, clinker slag comprising about 12 percent of the sample contributes residue ash of uranium and/or thorium that produces radioactivity above background levels.

Radioactive materials are believed to be contained in white glassy slag comprising four percent of the soil. Solid magnetic and nonmagnetic slag comprising, respectively, 7 and 13 percent of the soil sample, also might contain radioactivity above background levels. This material is suspected to be related to the industrial manufacture of thorium and may possibly contain radioactive residual products.

# 7.2.2 Composite 1016

Composite 1016 from Maywood is comprised of a variety of natural and anthropogenic materials. The natural rock and minerals comprise more than 45 percent of the soil and anthropogenic slags. Radioactive monazite and zircon are natural minerals that are present as significant sources of radioactivity. Both these minerals are found only in the fine sand and coarse silt-size fractions; however, the monazite comprises 3.33 percent and zircon 0.82 percent of the soil sample. The concentration level of these radioactive minerals suggests their presence is derived form ore minerals used to obtain thorium.

A white anthropogenic thorium substance comprises 8 percent of the soil sample tested. This material is probably an orthophosphate derived from the industrial process to obtain thorium metal. It is induced and contribution, or radioactivity in the soil sample.

Radioactivity is believed to be present in some of the slag materials that may be associated with the industrial process used to manufacture thorium metal. Individual fractions of white glassy slag comprising 5 percent of the soil; and solid magnetic and nonmagnetic slag, comprising 21 percent of the soil sample, should be examined to determine if this material is an important source of the radioactivity in the soil.

# 7.2.3 Dense and Magnetic Components

The results of the density and magnetics scoping study confirm the findings of the petrographic study that significant amounts of the radioactivity in Composites 1015 and 1016 are associated with dense (>2.89 g/cc) minerals. These results indicated the need for further characterization of the radioactive mineral associations using SEM/XRF and the need for additional laboratory studies on the composite samples to evaluate the potential for additional partitioning by specific gravity and magnetic susceptibility. The results of these studies are presented in Part II of this report.

# 7.3 Attrition

The attrition study on Composite 1015 did not indicate significant benefit from attrition to the -4/+100 mesh material. The results of the attrition test at 70 percent solids for Composite 1016 indicated discernable removal of radium and thorium. The use of a vigorous washing step before sieving the samples prior to attrition may have been sufficient to liberate the fine material from the coarse thus obscuring the benefit of such liberation. Any proposed process design would most certainly include some form of particle liberation as a precursor to any separation process.



7.4 Chemical Extraction

# 7.4.1 Composite 1015

The most effective extractant for both thorium and radium (Ra-228) was 3M HCl followed by 0.22M sodium hexametaphosphate, with removals of 80 and 63 percent, respectively. The resulting extracted soil concentrations of Ra-226, Ra-228, and Th-232, respectively, were 1.27, 4.48, and 1.40 pCMg. Only 6 percent of the soil was digested during the extraction.

While not as effective as 3M HCI/sodium hexametaphosphate, 0.1M EDTA/0.5M Na<sub>2</sub>CO<sub>3</sub> removed 62 and 24 percent of the thorium and radium, respectively. The resulting Ra-226, Ra-228, and Th-232 concentrations of the extracted soil were 1.63, 7.31, and 2.84 pCi/g, respectively, which would result in a TAR of less than one. Less than two percent of the soil was solubilized in this extraction.

# 7.4.2 Composite 1016

The most effective extractant for thorium, uranium, and radium (Ra-228) was 3M HNO<sub>3</sub>, with removals of 77, 73, and 78 percent, respectively. The resulting extracted soil concentrations of Ra-226, U-238, and Th-232, respectively, were 8.5, 3.4, and 25.7 pCi/g (TAR = 6.9). Approximately 6 percent of the soil was digested during the extraction.

While not as effective as  $HNO_3$ ; 3M HCl followed by 0.22M sodium hexametaphosphate removed 62, 67, and 71 percent of the thorium, uranium, and radium, respectively. The resulting Ra-226, U-238, and Th-232 concentrations of the extracted soil were 9.8, 4.2, and 43.4 pCi/g (TAR = 10.7), respectively.

If chemical extraction is believed to be a viable option for Maywood soil treatment, optimization studies will be required to determine ultimate removal potential.

# 7.5 Extended Petrographic Analyses

Detailed petrographic analyses conducted using Scanning Electron Microscopy coupled with Automated Image Analysis indicate that the only radioactive component observable via x-ray analysis under SEM was thorium. Thorium was found to occur primarily as a monazite-type particle in the heavy mineral sands. The amount of soil uncontaminated by the presence of heavy mineral sands was generally found to increase as particle size decreased. Also, the percentage of free heavy minerals contained in each sample was generally found to increase with decreasing particle size. In all cases, the 100x200 mesh size fraction seemed to stand out as the worst in terms of heavy mineral contamination.

In an attempt to relate the radionuclide analysis to the characterization data, the Total Activity Ratio (TAR) was compared to the heavy mineral content and the thorium mineral content of each size fraction. The TAR was found to correlate well with the thorium mineral content. A plot of TAR versus thorium mineral content indicated that these soils must be cleaned to a thorium mineral content of less than 0.06 percent in order to achieve a TAR of 1 or less. Based on the theoretical cut-off limit on thorium mineral content, the Composite 1015 sample was found to exhibit characteristics of a soil that would be amenable to physical separation. The Composite 1016 sample, on the other hand, appeared to contain far too much locked material to ever meet a TAR criteria of 1.

# 7.6 Laboratory Characterization Tests

# 7.6.1 Float-Sink Tests

Float-sink analyses were performed on the various size fractions of the Composite 1015 and 1016 soil samples. The test data confirmed the findings of the detailed petrographic results that indicated that remediation of the Composite 1016 sample that a TAR of 1 could not be achieved, although the total activity of the soil could be significantly reduced. For the Composite 1015 sample, the float-sink tests indicate that the largest weight percentage of the intermediate-sized soil particles are present in the 2.6 x 2.7 density fraction. These particles have Th-232 and Ra-226 (Pb-214) activities below 5 pCi/g.

### 7.6.2 Flotation Tests

Froth flotation tests conducted on the finer size fractions of the Composite 1015 soil indicate that flotation is capable of concentrating radionuclides in the froth product. For the minus 50 mesh flotation test, the Th-232 activity of the clean soil was reduced by 23 percent, while the concentrate Th-232 activity increased 63 percent. These values were changed to 20 percent and 320 percent, respectively, for the 50x200 mesh flotation test. The concentrate obtained for tests conducted on -200 mesh soil showed increases of up to 170 percent in Th-232 activity. Additional optimization tests are recommended based on these promising results.

### 7.7 Conceptual Flowsheet Development

Test data obtained from the petrographic and laboratory characterization studies were used to develop a conceptual flowsheet for the Maywood FUSRAP site. The flowsheet was developed using spreadsheet-based mass balancing and process simulation programs that were developed specifically for this project. For the Composite 1015 soil sample, the simulation results indicate that primary screening followed by hydroclassification (Circuit I) is capable of meeting a Th-232 activity of 5 pCi/g at a soil recovery of 52 percent by weight. By adding a low-cost density separator (Circuit II) to retreat the coarse product underflow from the hydroclassifier, achieving a Th-232 activity of 5 pCi/g also gives a soil recovery of 52 percent by weight. However, with just a slight decrease in the allowable Th-232 activity, the data show a significant decrease in soil recovery for the two circuits. For example, at a 4.5 pCi/g Th-232 activity the soil recoveries are 25 and 50 percent for Circuit I and Circuit II, respectively. These data indicate that operating Circuit I at a 5 pCi/g Th-232 activity for the final product will have little margin for error. This conclusion is supported in Figure 6-19 where the "knee" of the curve occurs around 4.8 pCi/g Th-232 with a sharp decline in soil recovery to around a 4.5 pCi/g Th-232 activity. This figure also shows that the decline in soil recovery is more subtle with Circuit II. Therefore, the Circuit II configuration will provide a "safety-net" around the clean-up level of 5 pCi/g, and thus is the circuit of choice. Because of the importance of Th-232 removal in meeting the clean-up criteria, an additional series of optimization runs were performed in which the soil recovery was maximized at different levels of Th-232 activity. The results of these simulations are also shown in Figure 6-19. Again, the simulation data show that only about 52 percent of the soil can be recovered at a Th-232 activity of 5 pCi/g. However, the mass recovery of clean soil can be increased to about 63 percent by increasing the allowable Th-232 activity to 13.5 pCi/g. A further increase in Th-232 activity is not technically feasible with the Circuit I or Circuit II configurations. To recover additional mass, the secondary classifier cut-point  $(D_{so})$  must decrease. This is due to 36 percent of the feed soil is below 200 mesh. However, there are no separation unit operations that will achieve a cut-point below 200 mesh, especially at the  $\beta$  and  $R_f$  values that are needed. However, the data suggest that the best operating point for reducing the Th-232 activity while maximizing soil recovery would be to produce a clean soil with a Th-232 activity of approximately 5 to 13 pCi/g. These values correspond to just above the "knee" in the recovery-activity plots. Figure 6-19 shows that these operating points would result in mass recoveries of approximately 55 and 63 percent. These values could be achieved using either Circuit I or II.

A preliminary cost analysis was conducted to examine the economic feasibility of the proposed circuitry. The cost analysis included an estimation of both capital and operating costs for the installation of processing facilities capable of treating as little as 62,100 tons and as much as

550,845 tons of contaminated soil. For the smaller volume, the estimated cost for remediation varied from \$198.42 to \$162.48 per ton of dry feed soil, depending on whether the cleanup target was set to 5 pCi/g or 15 pCi/g, respectively. These values represent a cost savings of more than \$8.4 million as compared to complete excavation and disposal of soil from the Maywood site. For the greater tonnage of 550,845 tons, the estimated remediation cost was found to vary from \$179.69 to \$143.73 per ton of dry feed soil for the cleanup targets of 5 pCi/g or 15 pCi/g, respectively. In this case, a total cost savings of more than \$85 million may be realized by combining soil treatment and disposal at the Maywood site.

# 8.0 REFERENCES

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# GLOSSARY

Anthropogenic	Man-made material. In this document it refers to the association of naturally occurring radioactive material with a manufacturing process waste.
Characterization	A study conducted to determine the basic characteristics, such as particle size, density, and magnetic susceptibility of soil. While its purpose is to determine if a treatment process is applicable to the remediation of a particular soil, the study is not conducted to specifically evaluate the effectiveness of a given technology.
Composite	Sample formed by the selective addition of other samples.
Head Sample	The sample fraction upon which experimentation is conducted (beginning sample fraction).
Hydroclassification	The separation of solid particles into fractions based on their differential rates of flow or settling through water.
Mesh Size	Number of openings per linear inch in a sieve used to determine particle size.
Monazite	Mineral consisting of phosphates of the rare earth metals, including thorium.
Orthophosphate	A salt of orthophosphoric acid containing the trivalent , negative radical $PO_4$ .
Petrography Study	Examination conducted to identify and classify minerals and materials in a soil sample.
TAR	Total Activity Ratio. The sum of the radium-226 and thorium-232 concentrations, divided by five, summed with the uranium-238 concentration, divided by 50.
Treatability Study	A study conducted for the purpose of determining if a treatment technology is applicable to the remediation of a contaminated soil.
Zircon	A natural mineral which may have incorporated uranium or thorium.

- b. If all contractually required peaks were not bbrary searched, the designated representative could request these data from the laboratory.
- 3. FIC results which are not sufficiently above the level in the blank should not be reported. (Dilutions and sample size must be taken into account when comparing the amounts present in blanks and samples.)
- 4. When a compound is not found in any blanks, but is a suspected artifact of common laboratory contaminant, the result may be flagged as unusable (R).
- 5. In deciding whether a library search result for a TIC represents a realistic identification, professional judgment must be exercised. If there is more than one reasonable match, the result may be reported as "either compound X or compound Y." If there is a lack of isomer specificity, the TIC result may be changed to a non-specific isomer result (1,3,5-trimethyl benzene to trimethyl benzene isomer) or to a compound class (2-methyl, 3-ethyl benzene to substituted aromatic compound).
- 6. The reviewer may elect to report all similar isomers as a total. (All alkanes may be summarized and reported as total hydrocarbons.)
- 7. Other Case factors may influence TIC judgments. If a sample TIC match is poor but other samples have a TIC with a good library match, similar relative retention time and the same ions, identification information may be inferred from the other sample TIC results.
- 8. Physical constants, such as boiling point, may be factored into professional judgment of TIC results.

### XIL SYSTEM PERFORMANCE

During the period following Instrument Performance QC checks (e.g. blacks, tuning, calibration), changes may occur in the system that degrade the quality of the data. While this degradation would not be directly shown by QC checks until the next required series of analytical QC runs, a thorough review of the ongoing data acquisition can yield indicators of instrument performance.

Some examples of instrument performance indicators for various factors are as follows:

- 1. Abrupt, discrete shifts in reconstructed ion chromatogram (RIC) baseline may indicate gain or threshold changes.
- 2. Poor chromatographic performance affects both qualitative and quantitative results. Indications of substandard performance include:
  - a. High RIC background levels or shifts in absolute retention times of internal standards.
  - b. Excessive baseline rise at elevated temperature.

### PESTICIDES PROCEDURE.

The requirements to be checked in validation are listed below. ("CCS" indicates that the <u>contract</u> requirements for these items will also be checked by CCS; CCS requirements are not always the same as the data review criteria.)

- 1. Holding Times (CCS Lab holding times only)
- II. Pesticides Instrument Performance (CCS)
- III. Calibration
  - o Initial (CCS)
  - o Analytical Sequence (CCS)
  - o Continuing (CCS) .
- IV. Blanks (CCS)
- V. Surrogate Recovery

VL Matrix Spike/Matrix Spike Duplicate (CCS)

VIL Field Duplicates

VIIL Compound Identification

IX. Compound Quantitation and Reported Detection Limits

X. Overall Assessment of Data for a Case

#### B. Criteria

1 DD1 Refention Time

DDT must have retention time on packed columns (except OV-1 and OV-101) greater than or equal to 12 minutes.

2. Retention Time Windows

The laboratory must report retention time window data on the Pesticide/PCB Standards Summary (Form IX) for each GC column used to analyze samples.

3. DDT/Endrin Degradation Check

The total percent breakdown for neither DDT nor endrin may exceed 20%. The percent breakdown is the amount of decomposition that endrin and  $4_4$ -DDT undergo when analyzed by the chromatographic system.

- a. For endrin, the percent breakdown is determined by the presence of endrin aldehyde and/or endrin ketone in the GC chromatogram.
- b. For 4,4'-DDT, the percent breakdown is determined from the presence of 4,4'-DDD and/or 4,4'-DDE in the GC chromatogram.
- c. A combined percent breakdown must be calculated if there is evidence of a peak at the retention time of endrin aldehyde/4,4'-DDD, which co-elute on the OV-1 packed column (or an equivalent column).
- d. Percent breakdown is calculated using the following equations:

& Desekdaye	Degradation Peak Areas (endrin aldehyde + endrin ketone) x 100 Peak Area (endrin + endrin aldehyde + endrin ketone)				
for eadrin					
	Note 1:	Peak area of endrin aldehyde must be measured during the degradation check to verify system performance. Endrin aldehyde is not reported on Form 1 because it is removed by alumina cleanup.			
	Note 2:	The term "peak height" may be substituted for the term "peak area".			
Combined =	L	Total degradation peak areas (DDE + DDD + endrin aldehyde + endrin ketone)			
% Breakdown	(DDT	Total DDT and endrin peak areas DDE + DDD + endrin + endrin aldehyde + endrin ketone)			

- a For the affected samples, check to see if chromatograms contain any peaks within an expanded window surrounding the experied retention time window of the pesticide of interest. If no peaks are propert either within or close to the retention time window of the deviant target pesticide compound, there is usually no effect on the data (Non-detected values can be considered valid.)
- b. If the affected sample chromatograms contain peaks which may be of concern (i.e., above the CRQL and either close to or within the expected retention time window of the pesticide of interest), then two options are available to the reviewer to determine the extent of the effect on the data.
  - 1) If no additional effort is warranted by the reviewer, flag all positive results and quantitation limits as unusable (R). The narrative should emphasize the possibility of either false negatives or false positives, as appropriate.
  - 2) In some cases, additional effort is warranted by the reviewer (e.g., if the data are needed on a priority basis and if the peak(s) present might represent a level of concern for that particular pesticide). In these situations, the reviewer may undertake the following additional efforts to determine a usable retention time window for affected samples:
    - (a) The reviewer should examine the data package for the presence of three or more standards containing the pesticide of interest that were run within a 72-hour period during which the sample was analyzed.
    - (b) If three or more such standards are present, the mean and standard deviation of the retention time window can be reevaluated.
    - (c) If all standards and matrix spikes fall within the revised window, the valid positive or negative sample results can be determined using this window.
    - (d) The marrative should identify the additional efforts taken by the reviewer and the resultant impact on data usability. In addition, the support documentation should contain all calculations and comparisons generated by the reviewer.
- 3. DDT/Endrin Degradation Check
  - a. If DDT breakdown is greater than 20%, beginning with the samples following the last <u>in-control</u> standard:
    - 1) Flag all quantitative results for DDT as estimated (J). If DDT was not detected, but DDD and DDE are positive, then flag the quantitation limit for DDT as unusable (R).
    - 2) Flag results for DDD and/or DDE as presumptively present at an estimated quantity (NJ).

where,

o = Standard Deviation

- CF = Mean Calibration Factor
- Note: The 10% RSD linearity check is required only for columns which are used for quantitative determinations. Quantitation of the surrogate requires the use of a column shown to meet the 10% linearity criterion. Columns used only to provide qualitative confirmation are not required to meet this criterion.
- 2. Analytical Sequence
  - a. Primary Analysis

At the beginning of each 72-hour period all standards must be analyzed.

- b. Confirmation Analysis
  - 1) Evaluation Standard Mix A, B, and C are required for the curve.
  - 2) Only the standards containing the compound(s) to be confirmed are required. These standards must be repeated after every five samples.
  - 3) Evaluation Mix B is required after every ten samples.
- 3. Continuing Calibration

The calibration factor for each standard must be within 15% of the standard at the beginning of the analytical sequence on quantitation columns (20% on confirmation columns).

#### C. Evaluation Procedure

- 1. Initial Calibration
  - a. Inspect the Pesticide Evaluation Standards Summary (Form VIII) and verify agreement with the raw GC data (chromatograms and data system printouts).
  - b. Check the raw data and recalculate some of the calibration factors and the percent relative standard deviations (%RSD) for aldrin, endrin, 4,4'-DDT, and dibutylchlorendate at the three calibration concentrations.

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#### IV. IILANKS

#### A. Objective

The assessment of blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply to any blank associated with the samples. If problems with <u>any</u> blank exist, all data associated with the Case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the Case, or the problem is an isolated occurrence not affecting other data.

#### B. Criteria

No contaminants should be present in the blank(s).

#### C. Evaluation Procedure

- 1. Review the results of all associated blank(s), Form I(s) and raw data (chromatograms, quantitation reports or data system printouts).
- 2. Verify that the method blank analysis(es) contains less than the Contract Required Quantitation Limits (CRQL) of any Pesticide/PCB or interfering peak.
- 3. Verify that method blank analysis has been reported per matrix, per concentration level, for each GC system used to analyze samples, and for each extraction batch.

#### D. Action

Action in the case of unsuitable blank results depends on the circumstances and the origin of the blank. No positive sample results should be reported unless the concentration of the compound in the sample exceeds 5 times the amount in the blank. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. The results must not be corrected by subtracting the blank value. Specific actions are as follows:

- 1. If a Pesticide/PCB is found in the blank but <u>not</u> found in the sample(s), no action is taken.
- 2. Any Pesticide/PCB detected in the sample and also detected in any associated blank, must be qualified when the sample concentration is less than 5 times the blank concentration.

The reviewer should note that the blank analyses may not involve the same weights, volumes or dilution factors as the associated samples. These factors must be taken into consideration when applying the Sx criteria, such that a comparison of the total amount of contamination is actually made.

Additionally, there may be instances where little or no contamination was present in the associated blanks, but qualification of the sample was deemed

#### B. Criteria

Sample and blank recoveries of dibutyleblorendate must be within limit, is perapplicable SOW (Form 11).

#### C. Evaluation Procedure

- 1. Check raw data (i.e., chromatograms, quant list, etc.) to verify the recoveries on the Surrogate Recovery (Form II).
- 2. If recoveries are not within limits, check raw data for possible interferences which may have affected surrogate recoveries.

#### D. Actioa

If pesticide surrogate recoveries are outside of advisory windows, the following guidance is suggested:

- 1. If low recoveries are obtained, flag associated positive results and quantitation limits as estimated (J).
- 2. If high recoveries are obtained, professional judgment should be used to determine appropriate action. A high bias may be due to co-eluting interferences.
- 3. If zero pesticide surrogate recovery is reported, the reviewer should examine the sample chromatogram to determine if the surrogate may be present, but slightly outside its retention time window. If this is the case, in addition to assessing surrogate recovery for quantitative bias, the overriding consideration is to investigate the qualitative validity of the analysis. If the surrogate is not present, flag all negative results as unusable (R).

#### VL MATRIX SPIKE/MATRIX SPIKE DUPLICATE

A. Objective

These data are generated to determine long-term precision and accuracy of the analytical method on various matrices. These data alone cannot be used to evaluate the precision and accuracy of individual samples.

#### B. Criteria

- 1. Advisory limits are established for spike recovery limits in the appropriate SOW and on Form III.
- 2. Advisory limits are established for relative percent difference between matrix spike and matrix spike duplicate recoveries in the appropriate SOW and on Form 111.

#### VIII. COMPOUND IDENTIFICATION

#### A. Objective

Qualitative criteria for compound identification have been established to minimize the number of erroneous identifications of compounds. An erroneous identification can either be a false positive (reporting a compound present when it is not) or a false negative (not reporting a compound that is present).

#### B. Criteria

- 1. Retention times of reported compounds must fall within the calculated retention time windows for the two chromatographic columns.
- 2. GC/MS confirmation is required if the concentration of a compound exceeds 10 ng/uL in the final sample extract.
- C. Evaluation Procedure
  - 1. Review Form L the associated raw data (chromatograms and data system printouts) and the Pesticide/PCB Identification Summary (Form X). Confirm reported positive detects, using appropriate retention times and retention time windows, and verify that the compounds listed as "not detected" are correct.
  - 2. Verify that positive identifications have dissimilar column analysis. (The 3% OV-1 column cannot be used for confirmation if both dieldrin and DDE are identified.)
  - 3. For multipeak pesticides (chlordane and toxaphene) and PCBs, the retention times and relative peak height ratios of major component peaks should be compared against the appropriate standard chromatograms.
  - 4. Verify that GC/MS confirmation was performed for pesticides/PCB concentrations in the final sample extract which exceeded 10 ng/uL.

#### D. Actioa

- 1. If the qualitative criteria for two-column confirmation were not met, all reported positive detects should be considered non-detects. The reviewer should use professional judgment to assign an appropriate quantitation limit using the following guidance:
  - a. If the misidentified peak was sufficiently outside the target pesticide retention time window, then the CRQL can be reported.
  - b. If the misidentified peak poses an interference with potential detection of a target peak, then the reported value should be considered and flagged as the estimated quantitation limit (UJ).
- 2. If PCBs or multipeak pesticides exhibit marginal pattern-matching quality, professional judgment should be used to establish whether the differences are attributable to environmental "weathering". If the presence of a

#### N OVERALL ASSESSMENT OF DATA FOR A CASE

It is appropriate for the data reviewer to make professional judgments and express concerns and comments on the validity of the overall data package for a Case. This is particularly appropriate for Cases in which there are several QC criteria out of specification. The additive nature of QC factors out of specification is difficult to assess in an objective manner, but the reviewer has a responsibility to inform users concerning data quality and data limitations in order to assist that user in avoiding inappropriate use of the data, while not precluding any consideration of the data at all. The data reviewer would be greatly assisted in this endeavor if the data quality objectives were provided.

### GLOSSARY B

#### Other Lenus

131 <sup>2</sup> 13	Reomothiorobenzene – volatile tuning compound
BNA	Base/Neutral/Acid Compounds - compounds analyzed by semivolatile technique
Case	A finite, usually predetermined number of samples collected over a given time period for a particular site. A case consists of one or more Sample Delivery Group(s).
ссс	Calibration Check Compound
ccs	Contract Compliance Screening - process in which SMO inspects analytical data for contractual compliance and provides results to the Regions, laboratories and EMSL/LV.
CF	Calibration Factor
CRQL	Contract Required Quantitation Limit
DFTPP	Decasluorotriphenylphosphine — semivolatile tuning compound
DPO	Deputy Project Officer
EICP	Extracted Ion Current Profile
GC/EC	Gas Chromatography/Electron Capture Detector
GC/MS	Gas Chromatograph/Mass Spectrometer
GPC	Gel Permeation Chromatography – A sample clean-up technique that separates compounds by size and molecular weight. Generally used to remove oily materials from sample extracts.
IS	Internal Standards - Compounds added to every VOA and BNA standard, blank, matrix spike duplicate, and sample extract at a known concentration, prior to instrumental analysis. Internal standards are used as the basis for quantitation of the target compounds.
MS/MSD	Matrix Spike/Matrix Spike Duplicate
m/z	The ratio of mass (m) to charge (z) of ions measured by GC/MS
OADS	Organic Analysis Data Sheet (Form 1)
ORDA	Organic Regional Data Assessment
PCB	Polychlorinated biphenyl

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VISE Validated time of Sample Receipt = time of sample receipt at the laborators is recorded on the shipper's delivery receipt and Sample Traffie Deport

o Standard Deviation Estimate (of a sample)

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### LABORATORY DATA VALIDATION

# FUNCTIONAL GUIDELINES FOR EVALUATING INORGANICS ANALYSES

Prepared for the

HAZARDOUS SITE EVALUATION DIVISION U.S. ENVIRONMENTAL PROTECTION AGENCY

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July 1, 1988

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#### INTRODUCTION

This document is designed to offer guidance in laboratory data evaluation and validation. In some aspects, it is equivalent to a Standard Operating Procedure (SOP). In other, more subjective areas, only general guidance is offered due to the complexities and uniqueness of data relative to specific samples. These Guidelines have been updated to include all requirements in the 7/87 Statement of Work (SOW) for Inorganics, Amendment 1 and December 1987 Revisions.

Those areas where specific SOPs are possible are primarily areas in which definitive performance requirements are established. These requirements are concerned with specifications that are not sample dependent; they specify performance requirements on matters that should be fully under a laboratory's control. These specific areas include blanks, calibration standards, calibration verification standards, laboratory control standards, and interference check standards. In particular, mistakes such as calculation and transcription errors must be rectified by resubmission of corrected data sheets.

This document is intended for technical review. Some areas of overlap between technical review and Contract Compliance Screening (CCS) exist however, determining contract compliance is not intended to be a goal of these guidelines. It is assumed that the CCS is available and can be utilized to assist in the data review procedure.

At times, there may be an urgent need to use data which do not meet all contract requirements and technical criteria. Use of these data does <u>not</u> constitute either a new requirement standard or full acceptance of the data. Any decision to utilize data for which performance criteria have not been met is strictly to facilitate the progress of projects requiring the availability of the data. A contract laboratory submitting data which are out of specification may be required to rerun or resubmit data even if the previously submitted data have been utilized due to urgent program needs; data which do not meet specified requirements are never fully acceptable. The only exception to this requirement is in the area of requirements for individual sample analysis; if the nature of the sample itself limits the attainment of specifications, appropriate allowances must be made. The overriding concern of the Agency is to obtain data which are technically valid and legally defensible.

All data reviews must have, as a cover sheet, the Inorganic Regional Data Assessment (IRDA) form. (A copy is attached at the end of this document.) If mandatory actions are required, they should be specifically noted on this form. In addition, this form is to be used to summarize overall deficiencies requiring attention, as well as general laboratory performance and any discernible trends in the quality of the data. (This form is not a replacement for the data review.) Sufficient supplementary documentation must accompany the form to clearly identify the problems associated with a Case. The form and any attachments must be submitted to the Contract Laboratory Program Quality Assurance Coordinator (CLP QAC), the Regional Deputy Project Officer (DPO), and the Environmental Monitoring Systems Laboratory in Las Vegas (EMSL/LV).

It is the responsibility of the data reviewer to notify the Regional DPO concerning problems and shortcomings with regard to laboratory data. If there is an urgent requirement, the DPO may be contacted by telephone to expedite corrective action. It is recommended that all items for DPO action be presented at one time. In any case, the Inorganic Regional Data Assessment form must be completed and submitted.

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- II. Calibration
  - o Initial (CCS)
  - o Initial and Continuing Calibration Verification (CCS)
- III. Blanks (CCS)
- IV. ICP Interference Check Sample (CCS)
- V. Laboratory Control Sample (CCS)
- VI. Duplicate Sample (CCS)
- VII. Matrix Spike Sample (CCS)
- VIII. Furnace Atomic Absorption QC (CCS)
- IX. ICP Serial Dilution (CCS)
- X. Sample Result Verification (CCS 10%)
- XI. Field Duplicates
- XII. Overall Assessment of Data for a Case

#### I. HOLDING TIMES

A. Objective

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of analysis.

Note: The holding time is based on the date of collection, rather than verified time of sample receipt, and date of digestion/distillation. It is a technical evaluation rather than a contractual requirement.

B. Criteria

Technical requirements for sample holding times have only been established for water matrices. The following holding time and preservation requirements were established under 40 CFR 136 (Clean Water Act) and are found in Volume 49, Number 209 of the Federal Register, page 43260, issued on October 26, 1984.

METALS:	6 months; preserved to $pH < 2$
MERCURY:	28 days; preserved to $pH < 2$
CYANIDE:	14 days; preserved to $pH > 12$

2) The correlation coefficient must be  $\geq 0.995$ .

Note: The correlation coefficient of 0.995 is a technical criterion and not contractual.

- c. Mercury Analysis
  - 1) A blank and at least four standards must be used in establishing the analytical curve.
  - 2) The correlation coefficient must be  $\geq 0.995$ .
- d. Cyanide Analysis
  - 1) A blank and at least three standards must be used in establishing the analytical curve.
  - 2) A midrange standard must be distilled.
  - A correlation coefficient ≥0.995 is required for photometric determination.
- 2. Initial and Continuing Calibration Verification (ICV and CCV)
  - a. Analysis results must fall within the control limits of 90 -110 percent Recovery (%R) of the true value for all analytes except mercury and cyanide.
  - b. Analysis results for mercury must fall within the control limits of 80-120%R.
  - c. Analysis results for cyanide must fall within the control limits of 85-115%R.
- C. Evaluation Procedure
  - 1. Verify that the instrument was calibrated daily and each time the instrument was set up using the correct number of standards and blank.
  - 2. Verify that the correlation coefficient is  $\geq 0.995$
  - 3. Check the distillation log and verify that the midrange CN standard was distilled.
  - 4. Recalculate one or more of the ICV and CCV %R per type of analysis (1CP, GFAA, etc.) using the following equation and verify that the recalculated value agrees with the laboratory reported values on Form IIA. Due to possible rounding discrepancies, allow results to fall within 1% of the contract windows (e.g., 89-111%).

$$%R = \frac{Found}{True} \times 100$$

Where.

Found = concentration (in ug/L) of each analyte measured in the analysis of the ICV or CCV solution

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#### C. Evaluation Procedures

Review the results reported on the Blank Summary (Form III) as well as the raw data (ICP printouts, strip charts, printer tapes, bench sheets, etc.) for all blanks and verify that the results were accurately reported.

D. Action

Action in the case of unsuitable blank results depends on the circumstances and origin of the blank. Sample results > IDL but <5 times the amount in any blank should be gualified as (U).

Any blank with a negative result whose absolute value is > IDL must be carefully evaluated to determine its effect on the sample data.

<u>Note</u>: The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil sample results reported on Form 1 will not be on the same basis (units, dilution) as the <u>calibration</u> blank data reported on Form III. The reviewer may find it easier to work from the raw data when applying 5X criteria to soil sample data/calibration blank data.

In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. The results must <u>not</u> be corrected by subtracting any blank value.

#### IV. ICP INTERFERENCE CHECK SAMPLE (ICS)

A. Objective

The ICP Interference Check Sample verifies the contract laboratory's interelement and background correction factors.

### B. Criteria

- 1. An ICS must be run at the beginning and end of each sample analysis run (or a minimum of twice per 8 hour working shift, whichever is more frequent).
- 2. Results for the ICS solution AB analysis must fall within the control limits of  $\pm 20\%$  of the true value.
- C. Evaluation Procedure
  - I. Recalculate from the raw data (ICP printout) one or more of the recoveries using the following equation (%R) and verify that the recalculated value agrees with the laboratory reported values on Form IV.

ICS %R = Found Solution AB x 100 True Solution AB
considered only as estimated values, since the exact value of any analytical system is instrument specific. Therefore, estimate the concentration produced by an interfering element. If the estimate is >2X CRDL and also greater than 10% of the reported concentration of the affected element, qualify the affected results as estimated (J).

#### V. LABORATORY CONTROL SAMPLE (LCS)

#### A. Objective

The laboratory control sample serves as a monitor of the overall performance of all steps in the analysis, including the sample preparation.

- B. Criteria
  - 1. All aqueous LCS results must fall within the control limits of 80-120%R, except Sb and Ag which have no control limits.
  - 2. All solid LCS results must fall within the control limits established by the EPA. This information is available from EMSL/LV.
- C. Evaluation Procedure
  - 1. Review Form VII and verify that results fall within the control limits.
  - 2. Check the raw data (ICP printout, strip charts, bench sheets) to verify the reported recoveries on Form VII. Recalculate one or more of the recoveries (%R) using the following equation:

$$LCS \%R = \frac{LCS Found}{LCS True} \times 100$$

Where,

- LCS Found = concentration (in ug/L for aqueous; mg/kg for solid) of each analyte measured in the analysis of LCS solution
- LCS True concentration (in ug/L for aqueous; mg/kg for solid) of each analyte in the LCS source
- D. Action
  - 1. Aqueous LCS
    - a. If the LCS recovery for any analyte falls within the range of 50 79% or >120%, qualify results > IDL as estimated (J).
    - b. If results are < IDL and the LCS recovery is greater than 120%, the data are acceptable.
    - c. If results are < IDL and the LCS recovery falls within the range of 50-79%, qualify the data for the affected analytes as estimated (UJ).

2. If the field blank was used for duplicate analysis, all other QC data must be carefully checked and professional judgment exercised when evaluating the data.

Note: This information must be included on the IRDA form.

#### VII. MATRIX SPIKE SAMPLE ANALYSIS

#### A. Objective

The matrix spike sample analysis provides information about the effect of each sample matrix on the digestion and measurement methodology.

- B. Criteria
  - 1. Samples identified as field blanks cannot be used for spiked sample analysis.
  - 2. Spike recovery (%R) must be within the limits of 75-125%. However, spike recovery limits do not apply when sample concentration exceeds the spike concentration by a factor of 4 or more.
- C. Evaluation Procedure
  - 1. Review Form V and verify that results fall within the specified limits.
  - 2. Check raw data and recalculate one or more %R using the following equation to verify that results were correctly reported on Form V.

$$%R = \frac{(SSR - SR)}{SA} \times 100$$

Where,

SSR = Spiked Sample Result SR = Sample Result SA = Spike Added

- 3. Verify that the field blank was not used for spike analysis.
- D. Action
  - 1. If the spike recovery is >125% and the reported sample results are < IDL, the data is acceptable for use.
  - 2. If the spike recovery is >125% or <75% and the sample results are > IDL, gualify the data for these samples as estimated (J).
  - 3. If the spike recovery falls within the range of 30-74% and the sample results are < 1DL, qualify the data for these samples as estimated (UJ).
  - 4. If spike recovery results fall <30% and the sample results are < 1DL, qualify the data for these samples as unusable (R).

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- 6. If sample absorbance is <50% of the post digestion spike absorbance then:
  - a. If the furnace post digestion spike recovery is not within 85-115%, qualify the sample results > IDL as estimated (J).
  - b. If the furnace post digestion spike recovery is not within 85-115%, qualify the sample results < IDL as estimated (UJ).
- 7 If Method of Standard Additions (MSA) is required but has not been done, qualify the data as estimated (J).
- 8. If any of the samples run by MSA have not been spiked at the appropriate levels, qualify the data as estimated (J).
- 9. If the MSA correlation coefficient is <0.995, qualify the data as estimated (J).

#### IX. ICP SERIAL DILUTION

A. Objective

The serial dilution determines whether significant physical or chemical interferences exist due to sample matrix.

B. Criteria

If the analyte concentration is sufficiently high (concentration in the original sample is minimally a factor of 50 above the IDL), an analysis of a 5-fold dilution must agree within 10% Difference (%D) of the original results.

- C. Evaluation Procedures
  - 1. Check the raw data and recalculate the %D using the following equation to verify that the dilution analysis results agree with results reported on Form IX.

$$\%D = \frac{II-SI}{I} \times 100$$

Where,

I = Initial Sample Result

- S = Serial Dilution Result (Instrument Reading x 5)
- 2. Check the raw data for evidence of negative interference, i.e., results of the diluted sample are significantly higher than the original sample.
- D. Action
  - 1. When criteria are not met, qualify the associated data as estimated (J).
  - 2. If evidence of negative interference is found, use professional judgment to qualify the data.

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#### C. Evaluation Procedures

Samples which are field duplicates should be identified using EPA Sample Traffic Reports or sample field sheets. The reviewer should compare the results reported for each sample and calculate the Relative Percent Difference (RPD), if appropriate.

#### D. Action

Any evaluation of the field duplicates should be provided with the reviewer's comments.

#### XII. OVERALL ASSESSMENT OF DATA FOR A CASE

It is appropriate for the data reviewer to make professional judgments and express concerns and comments on the validity of the overall data for a Case. This is particularly appropriate when there are several QC criteria out of specification. The additive nature of QC factors out of specification is difficult to assess in an objective manner, but the reviewer has a responsibility to inform the user concerning data quality and data limitations in order to assist that user in avoiding inappropriate use of the data, while not precluding any consideration of the data at all. If qualifiers other than those used in this document are necessary to describe or qualify the data, it is necessary to thoroughly document/explain the additional qualifiers used. The data reviewer would be greatly assisted in this endeavor if the data quality objectives were provided. The cover form and supplementary documentation must-be included with the review.

#### GLOSSARY B

#### Additional Terms

Any sample related to a particular QC analysis. Associated Samples For example: For ICV, all samples run under the same calibration curve. For duplicate RPD, all SDG samples digested/distilled of the same matrix. Atomic Absorption AA A plot of absorbance versus concentration of Calibration Curve standards A finite, usually predetermined number of samples Case collected in a given time period for a particular site. A Case consists of one or more Sample Delivery Groups. Continuing Calibration Blank - a deionized water CCB sample run every ten samples designed to detect any carryover contamination. Contract Compliance Screening - process in which CCS SMO inspects analytical data for contractual compliance and provides EMSL/LV, laboratories, and the Regions with their findings. Continuing Calibration Verification - a standard run CCV every ten samples designed to test instrument performance. Contract Laboratory Program CLP Contract Required Detection Limit CRDL Coefficient of Variation CV **Deputy Project Officer** DPO Environmental Monitoring System EMSL/LV Laboratory/ Las Vegas (P.O. Box 15027. Las Vegas. Nevada 89114) Field blanks are intended to identify contaminants Field Blank that may have been introduced in the field. blanks, travel blanks. trip Examples are rinsate blanks, and decontamination blanks.

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Sample Delivery Group - defined by one of the following, whichever occurs first:

- case of field samples
- each twenty field samples in a Case
- each 14-day calendar period during which field samples in a Case are received, beginning with receipt of the first sample in the SDG.

SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work

SDG

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# **RISK ASSESSMENT**

By: Scott Rowden Engineering Science

Presented at:

Clemson University, Continuing Engineering Education Seminar, February 20, 1991 DEFINITION OF "RISK ASSESSMENT"

CHARACTERIZATION OF THE POTENTIAL ADVERSE HEALTH EFFECTS OF HUMAN EXPOSURES TO ENVIRONMENTAL HAZARDS. INCLUDES:

- DESCRIPTION OF POTENTIAL ADVERSE HEALTH
   EFFECTS BASED ON RESEARCH RESULTS; human of an and a second sec
- EXTRAPOLATION OF RESEARCH RESULTS TO GIVEN SET OF CONDITIONS;
- JUDGEMENTS ON NUMBERS OF PERSONS EXPOSED AND TYPE OF EXPOSURES;
- SUMMARY OF OVERALL PUBLIC HEALTH
   PROBLEM; AND
- CHARACTERIZATION OF UNCERTAINTIES.



# DATA COLLECTION

- **MODELLING PARAMETERS**
- DEFINING BACKGROUND **CONCENTRATIONS** 
  - . . . comples

, not related to site incread

- **NATURALLY OCCURRING**
- THROPOGENIC LEVELS
- **RAGS MANUAL SUGGESTS APPLICATION OF RIGOROUS STATISTICAL ANALYSIS ONLY UNDER** LIMITED CIRCUMSTANCES NOTE: 10% OF TOTAL SAMPLES - SOMETIMES

**RULE OF THUMB** 

- **PRELIMINARY IDENTIFICATION OF POTENTIAL HUMAN EXPOSURE** 
  - MEDIA OF CONCERN
  - **TYPES OF EXPECTED CONTAMINANTS**
  - **POTENTIALLY EXPOSED POPULATIONS**
  - **POTENTIAL CONTAMINANT TRANSPORT ROUTES**

## DATA EVALUATION

- APPROPRIATE ANALYTICAL METHODS
- SAMPLE QUANTIFICATION LIMITS (SQLS)
  - Some samples contain a contaminant
  - 1/2 SQL
  - NOT ZERO
- EVALUATION OF QUALIFIED DATA
  - TABLE FOR USE OF QUALIFIED DATA: RAGS MANUAL
  - ESTIMATED VALUES (J)- SAME AS POSITIVE RESULT
  - REJECTED VALUES (R)- ELIMINATE
  - CONTAMINANTS FOUND IN BLANKS (B)-
    - × 10 X'S: COMMON LABORATORY CONTAMINANTS
    - **¤** 5 X'S: NOT COMMON LABORATORY CONTAMINANTS

## **EXPOSURE ASSESSMENT**

## THREE STEP PROCESS:

# **1. CHARACTERIZE EXPOSURE SETTING**

- PHYSICAL ENVIRONMENT--FUTURE AND CURRENT
- POTENTIALLY EXPOSED POPULATION

## 2. IDENTIFY EXPOSURE PATHWAYS

- CHEMICAL SOURCE/RELEASE
- EXPOSURE POINT
- EXPOSURE ROUTE to receptor

## 3. QUANTIFY EXPOSURE

- · MONITORING DATA les data
- MODELING

### Note: The 95% upper confidence limit (UCL) on the arithmetic average concentration most often used as estimate of exposure concentration.

QUANTIFICATION OF EXPOSURE: ESTIMATION OF CHEMICAL INTAKE (CONTINUED)

- INGESTION OF CONTAMINATED FRUITS
   AND VEGETABLES
- INGESTION OF CONTAMINATED MEAT, EGGS, AND DAIRY PRODUCTS

NOTE: SEE ATTACHMENT 1 FOR DETAILS CONCERNING CALCULATION OF CHEMICAL INTAKES

# GENERIC EQUATION FOR CALCULATING CHEMICAL INTAKES:

I = C X CR X EFDBW X AT

WHERE:

- I = CHEMICAL INTAKE (I.E., MG/KG-DAY)
- C = CHEMICAL CONCENTRATION (E.G., MG/LITER)

- EFD = EXPOSURE FREQUENCY AND DURATION EF = EXPOSURE FREQUENCY (DAYS/YEAR) ED = EXPOSURE DURATION (YEARS)
- BW = BODY WEIGHT (E.G., 70 KG)

AT = AVERAGING TIME = (DAYS)

## **TOXICITY ASSESSMENT--**CONTINUED

- WEIGHT-OF-EVIDENCE CLASSIFICATION:

### **GROUP CLASSIFICATION**

Α	HUMAN CARCINOGEN
B1 OR B2	PROBABLE HUMAN CARCINOGEN
С	POSSIBLE HUMAN CARCINOGEN
D	NOT CLASSIFIABLE AS TO HUMAN

CARCINOGENICITY

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E EVIDENCE OF NONCARCINOGENICITY FOR HUMANS

## **RISK CHARACTERIZATION**

# [CANCER RISKS]

### LINEAR LOW-DOSE CANCER RISK:



 $RISK_{i} = THE RISK ESTIMATE FOR THE i^{TH}$ SUBSTANCE. ATTACHMENT 1 EXPOSURE SCENARIOS EXTRACT FROM "RISK ASSESSMENT GUIDANCE FOR SUPERFUND," EPA/540/1-89/002 DECEMBER 1989

### RESIDENTIAL EXPOSURE: INGESTION OF CHEMICALS IN DRINKING WATER ' (AND BEVERAGES MADE USING DRINKING WATER)



### RESIDENTIAL EXPOSURE: DERMAL CONTACT WITH CHEMICALS IN WATER

Equatio	00:					
	Absorbed	Dose (mg/kg-day)	) = <u>CW x S</u>	A x PC x ET x F BW x AT	F x ED x CF	
Where:					,,,,,,,,,,,,	
CW = SA = PC = ET = EF = CF = BW = AT =	CW = Chemical Concentration in Water (mg/liter) SA = Skin Surface Area Available for Contact (cm <sup>2</sup> ) PC = Chemical-specific Dermal Permeability Constant (cm/br) ET = Exposure Time (hours/day) EF = Exposure Frequency (days/year) ED = Exposure Duration (years) CF = Volumetric Conversion Factor for Water (1 liter/1000 cm <sup>3</sup> ) BW = Body Weight (kg) AT = Averaging Time (period over which exposure is averaged — days)					
Variable Va	ilues:					
	CW: Site-s	pecific measured o	r modeled v	alue		
	SA:		.•			
	50th Percent	tile Total Body Sur	face Area (	m²) (EPA 1989d	<u>1985a)</u>	
		AGE (YRS)	MALE	FEMA	LE	
		3 < 6	0.728	0.711		
		6 < 9	0.931	0.919		
		9 < 12 12 < 15	1.16	1.10		
		15 < 18	1.75	1.60		
		Adult	1.94	1.69		
	50th Percent	tile Body Part-spec	cific Surfac	e Areas for Male	rs (m²) (EPA 1989d. )	1 <u>985a)</u>
	AGE (YRS)	ARMS	•	HANDS	LEGS	
	3<4	0.096		0.040	0.18	
	• < 7 • < 10	0.11		0.057	0.24	
	Adult	0.23-		0.082	0.55	
			· · · · · · · · · · · · · · · · · · ·			

### **RESIDENTIAL EXPOSURE:**

## INGESTION OF CHEMICALS IN SOIL

### Equation:

## $\frac{\text{Intake (mg/kg-day)} = \frac{\text{CS x IR x CF x FI x EF x ED}}{\text{BW x AT}}$

### Where:

CS = IR = CF = FI = ED = BW = AT =	Chemical Concentration in Soil (mg/kg) Ingestion Rate (mg soil/day) Conversion Factor (10 <sup>-4</sup> kg/mg) Fraction Ingested from Contaminated Source (unitless) Exposure Frequency (days/years) Exposure Duration (years) Body Weight (kg) Averaging Time (period over which exposure is averaged — days)				
12112010 14	1003.				
	CS:	Site-specific measured value			
	IR:	200 mg/day (children, 1 through 6 years old; EPA 1989g) 100 mg/day (age groups greater than 6 years old; EPA 1989g)			
		NOTE: IR values are default values and could change based on site-specific or other information. Research is currently ongoing to better define ingestion rates. IR values do not apply to individuals with abnormally high soil ingestion rates (i.e., pica).			
	CF:	10 <sup>-6</sup> kg/mg			
	FI:	Pathway-specific value (should consider contaminant location and population activity patterns)			
	EF:	365 days/year			
	ED:	70 years (lifetime; by convention) 30 years (national upper-bound time (90th percentile) at one residence; EPA 1989d) 9 years (national median time (50th percentile) at one residence; EPA 1989d)			
	BW:	70 kg (adult, average; EPA 1989d) 16 kg (children 1 through 6 years old, 50th percentile; EPA 1985a)			
	AT:	Pathway-specific period of exposure for noncarcinogenic effects (i.e., ED x 365 days/year), and 70 year lifetime for carcinogenic effects (i.e., 70 years x 365 days/year).			

### RESIDENTIAL EXPOSURE: INHALATION OF AIRBORNE (VAPOR PHASE) CHEMICALS

Equation: Intake  $(mg/kg-day) = CA \times IR \times ET \times EF \times ED$ BW x AT Where: CA =Contaminant Concentration in Air (mg/m<sup>3</sup>) IR = Inhalation Rate (m<sup>3</sup>/hour) ET =Exposure Time (hours/day) EF = Exposure Frequency (days/year) ED = Exposure Duration (years) BW = Body Weight (kg) AT = Averaging Time (period over which exposure is averaged - days) Variable Values: CA: Site-specific measured or modeled value IR: 30 m<sup>2</sup>/day (adult, suggested upper bound value; EPA 1989d) 20 m³/day (adult, average; EPA 1989d) Hourly rates (EPA 1989d) Age-specific values (EPA 1985a) Age, sex, and activity based values (EPA 1985a) 0.6 m<sup>3</sup>/hr -- showering (all age groups; EPA 1989d) ET: Pathway-specific values (dependent on duration of exposure-related activities) 12 minutes - showering (90th percentile; EPA 1989d) 7 minutes — showering (50th percentile; EPA 1989d) EF: Pathway-specific value (dependent on frequency of showering or other exposure-related activities) ED: 70 years (lifetime; by convention) 30 years (national upper-bound time (90th percentile) at one residence; EPA 1989d) 9 years (national median time (50th percentile) at one residence; EPA 1989d) BW: 70 kg (adult, average; EPA 1989d) Age-specific values (EPA 1985a, 1989d) AT: Pathway-specific period of exposure for noncarcinogenic effects (i.e., ED x 365 days/year), and 70 year lifetime for carcinogenic effects (i.e., 70 years x 365 days/year).

### RESIDENTIAL EXPOSURE: FOOD PATHWAY --INGESTION OF CONTAMINATED FISH AND SHELLFISH

Equation:

## Intake (mg/kg-day) = CF x IR x FI x EF x ED BW x AT

Where:

CF =	Cont	aminant Concentration in Fish (mg/kg)			
FI = EF = ED =	IR = Ingestion Rate (kg/meal) FI = Fraction Ingested from Contaminated Source (unitless) EF = Exposure Frequency (meals/year) ED = Furgeoute Function (meals/year)				
BW =	Body	Weight (kg)			
AT =	AT = Averaging Time (period over which exposure is averaged days)				
Variable Va	lues:				
	CF:	Site-specific measured or modeled value			
·	IR:	0.284 kg/meal (95th percentile for fin fish; Pao <i>et al.</i> 1982) 0.113 kg/meal (50th percentile for fin fish; Pao <i>et al.</i> 1982)			
		<ul> <li>132 g/day (95th percentile daily intakes averaged over three days for consumers of fin fish; Pao et al. 1982)</li> <li>38 g/day (50th percentile daily intake, averaged over three days for consumers of fin fish; Pao et al. 1982)</li> <li>6.5 g/day (daily intake averaged over a year; EPA 1989d. NOTE: Daily intake values should be used in conjunction with an exposure frequency of 365 days/year.)</li> <li>Specific values for age, sex, race, region and fish species are available (EPA 1989d, 1989h)</li> </ul>			
	FI:	Pathway-specific value (should consider local usage patterns)			
	EF:	Pathway-specific value (should consider local population patterns if information is available) 48 days/year (average per capita for fish and shellfish; EPA Tolerance Assessment System in EPA 1989h)			
	ED:	70 years (lifetime; by convention) 30 years (national upper-bound time (90th percentile) at one residence; EPA 1989d) 9 years (national median time (50th percentile) at one residence; EPA 1989d)			
	BW:	70 kg (adult, average; EPA 1989d) Age-specific values (EPA 1985a, 1989d)			
	AT:	Pathway-specific period of exposure for noncarcinogenic effects (i.e., ED x 365 days/year), and 70 year lifetime for carcinogenic effects (i.e., 70 years x 365 days/year).			

### RESIDENTIAL EXPOSURE: FOOD PATHWAY --INGESTION OF CONTAMINATED MEAT, EGGS, AND DAIRY PRODUCTS<sup>a</sup>

Equation: Intake  $(mg/kg-day) = CF \times IR \times FI \times EF \times ED$ BW x AT Where: CF Contaminant Concentration in Food (mg/kg) = IR = Ingestion Rate (kg/meal) FI Fraction Ingested from Contaminated Source (unitless) = EF = Exposure Frequency (meals/year) ED = Exposure Duration (years) BW = Body Weight (kg) AT. = Averaging Time (period over which exposure is averaged - days) Variable Values: CF: Site-specific measured or modeled value. Based on soil concentrations, plant (feed) accumulation factors, and feed-to-meat or feed-to-dairy product transfer coefficients IR: 0.28 kg/meal --- beef (95th percentile; Pao et al. 1982) 0.112 kg/meal --- beef (50th percentile; Pao et al. 1982) Specific values for other meats are available (Pao et al. 1982) 0.150 kg/meal - eggs (95th percentile; Pao et al. 1982) 0.064 kg/meal - eggs (50th percentile; Pao et al. 1982) Specific values for milk, cheese and other dairy products are available (Pao et al. 1982) Pathway-specific value (should consider location and size of contaminated FI: area relative to that of residential areas, as well as anticipated usage patterns) EF: Pathway-specific value (should consider anticipated usage patterns) ED: 70 years (lifetime; by convention) 30 years (national upper-bound time (90th percentile) at one residence; EPA 1989d) 9 years (national median time (50th percentile) at one residence; ÉPA1989d) BW: 70 kg (adult, average; EPA 1989d) Age-specific values (EPA 1985a, 1989d) Pathway-specific period of exposure for noncarcinogenic effects AT: (i.e., ED x 365 days/year), and 70 year lifetime for carcinogenic effects (i.e., 70 years x 365 days/year).

### ATTACHMENT NO. 2 CASE STUDY NO. 1 CERCLA LANDFILL SITE

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