



Characterization Protocol for Radioactive Contaminated Soils

Office of Emergency and Remedial Response
Office of Radiation Programs, ANR-458

Quick Reference Fact Sheet

The Superfund Amendments and Reauthorization Act of 1986 (SARA) mandates that remediation at Superfund sites must utilize a permanent solution and alternative treatment technologies or resource recovery options to the maximum extent practicable. Treatment technologies that permanently and significantly reduce the mobility, toxicity, or volume of hazardous substances are preferred in this requirement. However, in most remedial actions conducted to date at radioactive sites, the radioactive soil has been excavated and stored in temporary above-ground containment facilities. To alleviate this storage situation the Office of Radiation Programs has developed an innovative soil characterization process applicable in the RI/FS stages of the Superfund process to support the development of technologies for on-site volume reduction of radioactive soils by physical separation^{1,2} technologies.

BACKGROUND

The volume reduction methods employed are based on physical/mechanical technologies that are common to the coal and ore processing industries. These common technologies have been adapted, modified, and directed toward the task of soil restoration. This soil characterization protocol is designed to demonstrate the suitability (or lack thereof) of various radioactivity contaminated soils for physical or chemical separation processes. These could potentially remove the radioactive fraction from the soil, thus producing a smaller volume requiring disposal. The protocol combines radiochemical and petrographic analysis of soil fractions, focusing on the contaminant waste and its particle size distribution in the host media. Soil remediation by volume reduction takes advantage of the fact that radionuclide contaminants concentrate generally in the smaller soil size fractions, and tend to selectively associate with materials that possess unique physical and/or chemical properties. The data obtained by following this protocol are used as the first phase of remediation assessment to determine if volume reduction is feasible.

CHARACTERIZATION DESCRIPTION

This soil characterization protocol examines the various size fractions of a representative sample of radioactive soil from a Superfund site, to provide the following information:

- Grain size distribution curve which relates weight percent versus particle size.
- Relationship of radioactivity to particle size.
- Identification of the mineral/material composition and physical properties of the radioactive contaminants for the various size fractions.
- Identification of the mineral composition and physical properties of the host material for the various size fractions.
- Additional information on contaminant and host material mineralogical and physical properties in support of feasible volume reduction techniques, e.g., magnetic properties.

These data are used to conceptualize a site-specific volume reduction process based on one or more of the following technologies:

- screening,
- classification,
- gravity separation,
- magnetic separation,
- flotation,
- chemical extraction,
- washing,
- scrubbing,
- surface de-bonding, and
- attrition.

The two-tiered soil characterization protocol, as shown in Figure 1, consists of feasibility analyses (Tier I), and optimization analyses (Tier II), as necessary, to cost-effectively maximize the volume reduction.

Pre-Tier I

Prior to Tier I laboratory tests, the representative contaminated soil samples obtained in compliance with EPA and DOE directives from a site^{3,4,5} are radiologically screened to assure that the activity levels are within laboratory license requirements and that proper safety practices will be applied. Additional chemical analyses should be performed on a portion of each soil sample for the presence of organic and heavy-metal constituents if that information has not been previously collected. This information not only identifies hazardous constituents (e.g., cyanide, heavy metals, chlorinated hydrocarbons), but also contributes to the mineralogical determination of the soil.

The remaining portions of each soil sample are oven dried at 60°C prior to weighing. The upper limit of 60°C is specified in order to maintain the mineral integrity of the soil by preventing the loss of water of hydration associated with the mineral structures which occur in some clays and other minerals at low temperatures.

Tier I

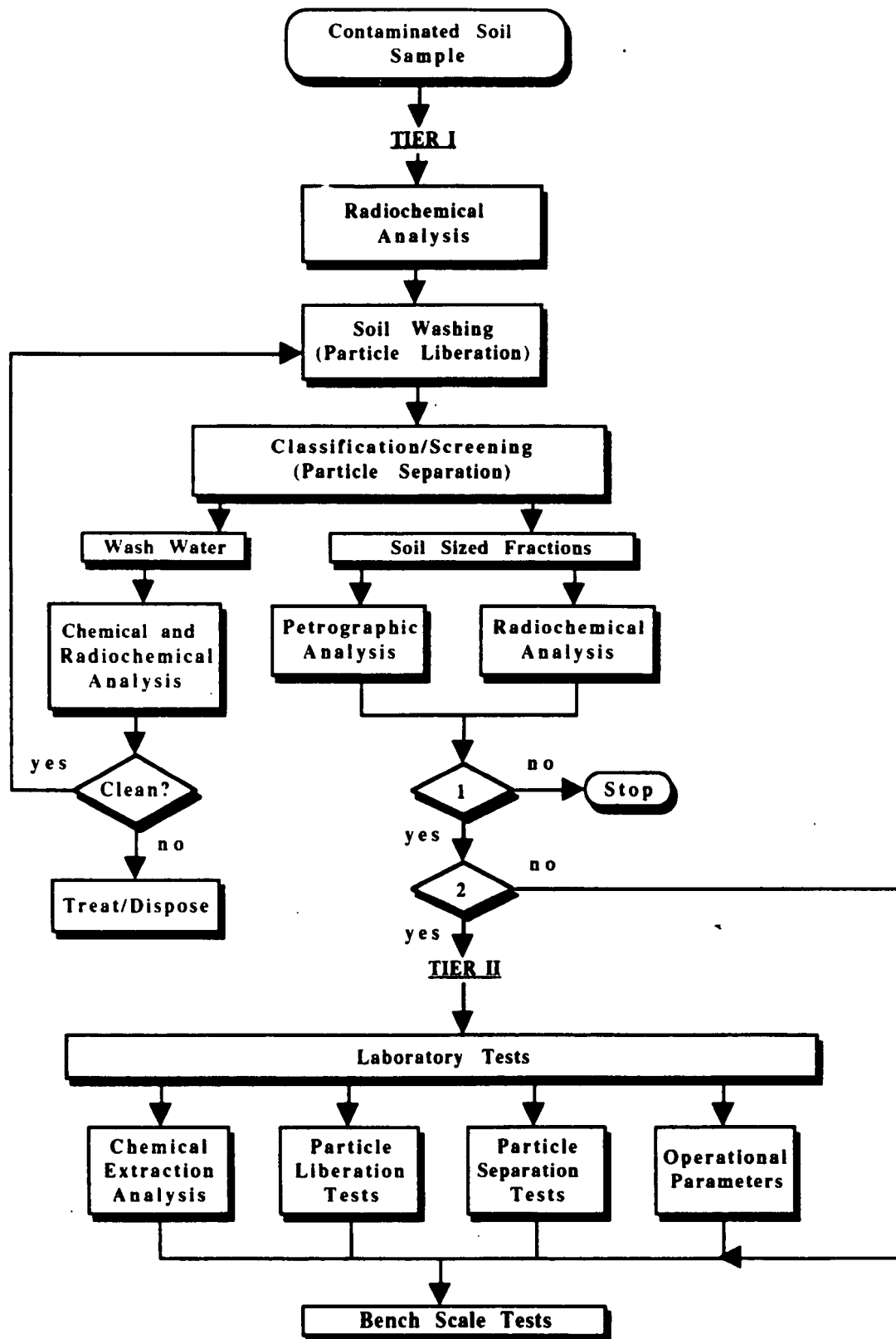
Tier I begins with radioanalysis of the dry soil samples by high-resolution gamma spectroscopy, and if necessary, alpha and beta spectroscopy analysis (using standard leaching/digestion and chemical methods⁶) to determine the level and type of activity present in each sample.

Physical separation of the soil particles is accomplished by mixing at least 250 grams of each soil sample with water to produce a liquid-to-solid (L/S) ratio of 5/1, agitating the mixture with a vigorous motion for 30 minutes at ambient temperature, and wet screening⁷ through a set of nested sieves. In some site specific cases it may be advantageous to perform a less vigorous wash because of the nature of the constituents. The standard sieves include at least mesh sizes 4 (4.75 mm), 50 (0.30 mm), 100 (0.15 mm), and 200 (0.075 mm). Each soil fraction is dried at 60°C, weighed, and analyzed for radionuclide activity. From this procedure the weight and radionuclide distribution by particle size is determined. A similar separation is also performed using hydroclassification methods. The results of these tests indicate the compatibility of the soil to remediation by particle-size hydroseparation techniques.

[NOTE: All water used must be collected and analyzed since it may contain transferred radioactive contaminants, Target Analyte List metals, volatile organic solvents, and/or pesticides. The analytical results will determine if the water can be recycled, safely disposed down a drain, or if it must be treated as a hazardous waste.]

Petrographic analysis is conducted on each of the size fractions to identify the mineral/material composition and physical properties of the radioactive contaminants and host materials. Petrographic procedures^{8,9,10} include the use of binocular and petrographic microscopes to provide a statistical point count of all materials larger than silt-size to 0.038 mm (400 mesh size), and x-ray diffraction analysis of fines less than 0.038 mm size. Density separations are made on sand and silt size fractions (0.30 to 0.045 mm) to concentrate heavy particles greater than 3.0 specific gravity using sodium polytungstate as the separating liquid. The heavy fractions, in many cases, provide focus on radioactive particles which tend to concentrate in minerals or anthropogenic radioactive materials of the heavy fractions. The degree of weathering, presence of coatings, particle shape, surface texture,

Figure 1: Soil Characterization Flow Chart



LEGEND



Volumetric reduction feasible?



Additional physical and chemical data recommended

hardness, magnetism, and degree of aggregation or homogeneous nature are also physical properties examined for interpretations that relate to adsorption, waste form, and potential physical separation methods.

Tier I Report

Tier I tests results are gained from the petrographic and radiochemical analysis of the size fractions, as depicted in Figure I, to assess the feasibility of using volume reduction as a remediation technology. The test results include a grain size distribution curve of weight percent versus particle size, graphic data on activity level versus particle size, and tables and graphs on complete physical and mineralogic descriptions. This data is instrumental to the interpretation of the radioactive contaminants concentration in specific size ranges and the physical similarity and difference of the contaminants in relation to host materials.

It is assumed that the petrography and radiochemistry will be performed by personnel who are qualified by education and experience to employ the methodology specified and that recommendations for additional tests to validate key parameters for future tests will be incorporated in the report, e.g., recommend analysis of diagnostic elements that constitute chemical signatures to radioactive compounds. Radiochemical data should also be correlated with mineralogic data for interpretations, e.g., secular equilibrium of radionuclides to validate natural radioactive mineral assemblages reported or in the event of non-secular equilibrium of radionuclides, to reflect on anthropogenically enhanced radioactive waste forms in the radioactive soil. Any historic data on the ore minerals used and chemical processes used to convert the radionuclides to anthropogenic compounds should also be reported for the forensic data it might provide to support the list of radioactive compounds reported in the Tier I testing.

The Tier I report will provide an assessment of the technical feasibility of using one or more of the volume reduction technologies. Based on the feasibility of the most promising alternative, the Tier I report will also provide recommendations on further testing (Tier II) focusing on the validation of key factors that affect volume reduction. On the other hand, an evaluation of the test data could lead to the preliminary conclusion that volume reduction is not technically feasible.

Tier II

If the Tier I test data indicates the soil is satisfactory for remediation consideration Tier II testing is conducted. Tier II tests are designed to collect additional data for further characterization of contaminated soils. For example, additional soil fractions may be tested to focus on the mineral phase of opaque constituents, particle coatings, or special materials requiring more precise instrumentation for validation of particles than was made available for Tier I tests. Additional tests may also be necessary to provide optimum soil separation sizes. These tests can be performed with small soil volumes. The results are to be used to plan bench-scale tests that are designed to take advantage of unique physical and chemical characteristics of radioactive contaminants and host soil constituents. Tier II tests to be considered are in support of one of the following general categories of treatment technologies:

- Particle separation,
- Particle liberation, and
- Chemical extraction.

Particle separation is the separation of a mixture of various particles into two or more portions. For example, magnetic separation separates a mixture of soil particles based on the difference in magnetic susceptibilities.

Particle liberation is the physical de-bonding of contaminated particles or coatings from clean particles. For example, attrition removes friable coatings from soil particles.

When performing chemical extraction, the soil is immersed in a solvent that has been carefully chosen to preferentially extract the contaminant.

Selected chemical extraction tests may be performed in Tier II (as shown in Figure 1) to determine the potential for remediation by simple chemical extraction. Chemical extraction tests are designed to remove contaminants from selected particle-size fractions or from whole soil if it proves to be unsuitable for remediation by physical separation techniques. For example, the latter possibility exists for soils with uniform radionuclide distribution among the various particle sizes.

The chemical extraction tests are conducted on 100

gram samples of selected soil fractions or whole soil. On a sample in which the nature of the contaminant is poorly known, extractions are performed at 90°C with water and each of four extracting reagents known to be effective in removing various radionuclides from contaminated soils. These reagents include dilute solutions of hydrochloric acid, nitric acid, sodium chloride with hydrochloric acid, and sodium hexametaphosphate. With foreknowledge of the presence of a contaminant in a particular mineral form, one or two other select extracting reagents specific for the mineral are also included in these preliminary tests. The results of these tests provide information about the potential of chemical extraction as a complement or alternative to remediation.

Along with Tier I results, data from the Tier II tests can be used to select bench-scale test equipment for conducting remediation tests of contaminated soils. The initiation of bench-scale testing is based on the preliminary information provided by soil characterization which assesses the differences in physical properties between the waste form and host materials. For example, for physical volume reduction the applicable information relating to the differences in the waste form from the host material may be classified as follows:

- Relationship of radioactivity to particle sizes.
- Relationship of radioactivity to particle densities.
- Relationship of radioactivity to particle wettabilities.
- Relationship of radioactivity to particle shapes.
- Relationship of radioactivity to particle magnetic properties.
- Relationship of radioactivity to friability of particles or of particle coatings.
- Solubility of contaminants.

The most important information is the relationship of radioactivity to particle sizes. The information on the other physical properties such as density is

obtained by identifying the waste form and host matrix using petrographic techniques. It is important to develop this petrographic information for various ranges of particle size. And, based on a careful analysis of this information, a preliminary bench-scale test can be designed using batch applications of physical methods if a difference in the physical properties stated exists between the radioactive contamination and the host materials.

Tier II Report

The Tier II report consists of the test data generated in the categories depicted in Figure I. In most cases, except for the chemical extraction tests, the Tier I recommendations provided focus on amplification of specific objectives that appear in tables and graphs in the report. Tier II tests results, just like Tier I tests results, are evaluated to assess the feasibility of using volume reduction, and if so, to what degree. The evaluation has focus on the physical differences previously cited between the waste form and host materials for design of bench-scale tests that will provide more realistic quantification of degree of separation possible by volume reduction equipment. The nature of the site specific soil drives the testing performed so that, while no standard format is presented, it is assumed that the test objectives will be governed by qualified personnel skilled in the state of the art of quality beneficiation testing. The report data can thus generate preliminary cost and time assessments that relate to the feasibility of volume reduction for the particular site.

SUMMARY

The characterization protocol described above for radioactive contaminated soils depends mainly upon the physical, chemical, and mineralogical characteristics of the soil and radioactive particles with respect to grain size. The intent is to return the "clean" soil fractions, which can be a major portion of the soil (by volume), to the ground, preferably on-site.

Supplemental information concerning this protocol may be obtained from James Neihsel or Mike Eagle at (202) 260-9630, ANR 461, U.S. Environmental Protection Agency, 401 M Street SW, Washington, D.C. 20460.

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