September 2013 Update: EPA has validated and published a rapid method for analysis of radium in soil matrices by incorporating fusion procedures with counting methods. The method is summarized and accessible through the link provided below, and replaces use of ASTM D3084-05 for analysis of radium-226 in soil samples when using the methods listed in SAM.

Rapid Method for Radium in Soil Incorporating the Fusion of Soil and Soil-Related Matrices with the Radioanalytical Counting Method for Environmental Remediation Following Radiological Incidents

Analyte(s)	CAS RN
Radium-226	13982-63-3

Analysis Purpose: Qualitative analysis
Technique: Alpha spectrometry

Method Developed for: Radium-226 in soil samples
Method Selected for: SAM lists this method for qualitative analysis of soil/sediment samples

Description of Method: The method is based on the complete fusion of a representative, finely ground 1-g aliquot of dried sample with no insoluble residue remaining after dissolution of the fused melt in acid. For organic soil matrices, the sample is dry-ashed at 600 ^{o}C in an appropriate vessel prior to fusion, then dissolved in a crucible with hydrofluoric acid and evaporated to dryness on a hotplate at medium to high heat (~300 ^{o}C). Dry flux mix (equal weight of dried sodium carbonate, potassium carbonate and boric acid) is added and the crucible is warmed under a flame until a reaction initiates. The crucible is then heated under full flame until the reaction subsides and the melt is completely liquid and homogeneous. After cooling, the solidified melt is dissolved in hydrochloric acid, and the dissolved sample is transferred to an appropriately sized digestion container while rinsing the crucible with 6 M of HCl to ensure a quantitative transfer of material. The barium content is determined for a small aliquot of the dissolved flux by ICP-AES. Based on the barium content, add a sufficient amount of barium is added to the sample so that the final mass of barium is not more than 90 µg. A manganese (IV) solution and phenolphthalein indicator is added to this mixture and the pH is adjusted with sodium hydroxide until the solution just turns pink. Hydrogen peroxide is slowly added causing the solution to foam and form black insoluble manganese (II) oxide. After mixing and centrifuging, the manganese (IV) oxide precipitate is rinsed with water and dissolved in the manganese (IV) oxide stripping agent. Ascorbic acid is added to reduce any iron (III) to iron (II) and actinium and thorium are removed by passing the solution through a column of Diphonix® resin, and the column is rinsed with hydrochloric acid. The solution is poured through the resin column and rinsed with hydrochloric acid. The rinse solution is collected in a centrifuge tube, ammonium sulfate and isopropanol are added, and the centrifuge tube is placed in a cold water ultrasonic bath. After 20 minutes, the solution is filtered (the precipitate will not be apparent). The sample container and filter apparatus is rinsed with an isopropanol solution of ammonium sulfate; all rinsate is run through the filter. The filter is then placed in a Petri dish and dried under a heat lamp for several minutes. The filter is stored for at least 24 hours to allow sufficient astatine-217 (third progeny of radium-225) to ingrow into the sample test source. The sample is then counted by alpha spectrometry.

Special Considerations: If the sample may contain discrete radioactive particles (DRPs) or particles larger than a nominal size of 150 µm, additional sample preparation may be necessary as described in Sections A4 and A5.2.3 of the method (Interferences and Hot Particles, respectively). Soils with high silica content may require either additional fusing reagent and boric acid or a longer fusion melt. Platinum crucibles must be used, in this method, when digesting samples with hydrofluoric acid. If platinum crucibles are not available, an effective, alternate method is available that uses zirconium

crucibles [see *Rapid Method for Sodium Hydroxide Fusion of Concrete Matrices prior to Am, Pu, Sr, Ra, and U Analyses* (www2.epa.gov/radiation/incident-guides) and *Rapid Radiochemical Method for Total Radiostrontium (Sr-90) in Building Materials for Environmental Remediation Following Radiological Incidents* (www2.epa.gov/radiation/incident-guides)].

Source: EPA, Office of Radiation and Indoor Air National Air and Radiation Environmental Laboratory (NAREL). August 2012. Rev 0. "Rapid Method for Radium in Soil Incorporating the Fusion of Soil and Soil-Related Matrices with the Radioanalytical Counting Method for Environmental Remediation Following Radiological Incidents," EPA-600_R-12-635. http://www2.epa.gov/radiation/rapid-radiochemical-methods-selected-radionuclides