September 2014 Update: EPA has validated and published a rapid method for building material matrices for analysis of plutonium-238 and plutonium-239. The method is summarized and accessible through the link provided below.

Rapid Radiochemical Method for Plutonium-238 and Plutonium-239/240 in Building Materials for Environmental Remediation Following Radiological Incidents

Analyte(s)	CAS RN
Plutonium-238	13981-16-3
Plutonium-239	15117-48-3

Analysis Purpose: Qualitative analysis
Technique: Alpha spectrometry

Method Developed for: Plutonium-238 and plutonium-239 in building materials
Method Selected for: SAM lists this method for qualitative analysis of plutonium-238 and -239 in concrete or brick building materials

Description of Method: This method is based on the use of TEVA® resin (Aliquat 336 extractant-coated resin) to isolate and purify plutonium by removing interfering radionuclides as well as other components of the sample matrix in order to prepare the plutonium fraction for counting by alpha spectrometry. The method utilizes vacuum-assisted flow to improve the speed of the separations. The sample may be fused using the procedure "Rapid Method for Sodium Hydroxide Fusion of Concrete and Brick Matrices Prior to Americium, Plutonium, Strontium, Radium, and Uranium Analyses," Revision 0, EPA 402-R14-004 (Reference 16.3 of the method); with the plutonium isotopes then removed from the fusion matrix using iron hydroxide and lanthanum fluoride precipitation steps. Plutonium-242 or plutonium-236 tracer, added to the sample, is used as a yield monitor. The sample test source is prepared by microprecipitation with cerium (III) fluoride. The method is capable of achieving a required method uncertainty of 0.25 pCi/g for plutonium-238, -239/240, at an analytical action level of 1.89 pCi/g. To attain the stated measurement quality objectives (MQOs), a sample weight of approximately 1 gram and count time of at least 3 to 4 hours are recommended.

Special Considerations: Alpha-emitting radionuclides with irresolvable alpha energies, such as plutonium-238 (5.50 MeV), americium-241 (5.48 MeV), and thorium-228 (5.42 MeV) must be chemically separated to enable measurement. This method separates these radionuclides effectively. The significance of peak overlap will be determined by the individual detector's alpha energy resolution characteristics and the quality of the final precipitate that is counted. Vacuum box lid and holes must be cleaned frequently to prevent cross-contamination of samples. Non-radiological interferences include very high levels of anions such as phosphates which may lead to lower yields due to competition with active sites on the resin and/or complexation with plutonium ions. Aluminum is added in the column load solution to complex interfering anions such as fluoride and phosphate.

Source: U.S. EPA, National Air and Radiation Environmental Laboratory (NAREL). April 2014. Rev 0 "Rapid Radiochemical Method for Plutonium-238 and Plutonium-239/240 in Building Materials for Environmental Remediation Following Radiological Incidents," EPA 402-R14-006.
http://www.epa.gov/sam/pdfs/EPA-402-R14-006.pdf