September 2014 Update: EPA has validated and published a rapid method for building material matrices for analysis of uranium-234, uranium-235, and uranium-238. The method is summarized and accessible through the link provided below.

Rapid Radiochemical Method for Isotopic Uranium in Building Materials for Environmental Remediation Following Radiological Incidents

Analyte(s)	CAS RN
Uranium-234	13966-29-5
Uranium-235	15117-96-1
Uranium-238	7440-61-1

Analysis Purpose: Qualitative analysis
Technique: Alpha spectrometry

Method Developed for: Uranium-234, uranium-235, and uranium-238 in concrete and brick samples
Method Selected for: SAM lists this method for qualitative analysis of uranium-234, uranium-235, and uranium-238 in concrete or brick building materials

Description of Method: This method is based on the use of extraction chromatography resins to isolate and purify uranium isotopes by removing interfering radionuclides as well as other components of the sample matrix in order to prepare the uranium fraction for counting by alpha spectrometry. The method utilizes vacuum-assisted flow to improve the speed of the separations. Uranium-232 tracer, added to the building materials sample, is used as a yield monitor. A one gram (approximately) sample is fused using the procedure "Rapid Method for Sodium Hydroxide Fusion of Concrete and Brick Matrices Prior to Americium, Plutonium, Strontium, Radium, and Uranium Analyses for Environmental Remediation Following Radiological Incidents" (Reference 16.3 of the method). The uranium isotopes are then removed from the fusion matrix using iron hydroxide and lanthanum fluoride precipitation steps. The sample test source is prepared by microprecipitation with cerium (III) fluoride. The method is capable of achieving a method uncertainty for uranium-234, uranium-235, and uranium-238 of 1.9 pCi/g at an analytical level of 14.7 pCi/g. To attain the stated measurement quality objectives (MQOs), a sample weight of approximately 1 g and count time of at least 3 to 4 hours are recommended.

Special Considerations: Alpha-emitting radionuclides with peaks at energies that cannot be adequately resolved from the tracer or analyte (e.g., polonium-210 (5.304 MeV), thorium-228 (5.423 MeV, 5.340 MeV), and americium-243 (5.275 MeV, 5.233 MeV)) must be chemically separated to enable radionuclide-specific measurements. This method separates these radionuclides effectively (See Section 4 of the method for procedures to remove specific interferences). Nonradiological anions such as fluoride and phosphate that complex uranium ions may cause lower chemical yields. Aluminum that is added in the column load solution complexes fluoride, as well as any residual phosphate that may be present. Lanthanum, added to preconcentrate uranium from the sample matrix as lanthanum fluoride, can have a slight adverse impact on uranium retention on TRU resin, but this impact is minimal at the level added. Iron (3+) can also have an adverse impact on uranium retention on TRU resin, but the residual iron levels after preconcentration steps are acceptable.

Source: U.S. EPA, National Air and Radiation Environmental Laboratory (NAREL). April 2014. Rev 0 "Rapid Radiochemical Method for Isotopic Uranium in Building Materials for Environmental Remediation Following Radiological Incidents," EPA 402-R14-005.
http://www2.epa.gov/radiation/rapid-radiochemical-methods-selected-radionuclides