September 2014 Update: EPA has validated and published a rapid method for sodium hydroxide fusion of concrete and brick matrices for analysis of americium-241, plutonium-238, plutonium-239, radium-226, strontium-90, uranium-234, uranium-235 and uranium-238. The method is summarized and accessible through the link provided below.

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

Analyte(s)	CAS RN
Americium-241	14596-10-2
Plutonium-238	13981-16-3
Plutonium-239	15117-48-3
Radium-226	13982-63-3
Strontium-90	10098-97-2
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: Americium-241, plutonium-238, plutonium-239, radium-226, strontium-90, uranium-234, uranium-235 and uranium-238 in concrete and brick samples Method Selected for: SAM lists this method for qualitative analysis of americium-241, plutonium-238, plutonium-239, radium-226, strontium-90, uranium-234, uranium-235 and uranium-238 in concrete or brick building materials

Description of Method: Concrete and brick samples may be received as core samples, pieces of various sizes, dust or particles (wet or dry) from scabbling, or powder. Concrete or brick samples should be ground to at least 50 - 100 mesh size prior to fusion, if possible. The method is based on the rapid fusion, in zirconium crucibles, of a representative, finely ground 1 - 1.5 gram aliquant using rapid sodium hydroxide fusion at 600 °C. Plutonium, uranium, and americium are separated from the alkaline matrix using an iron/titanium hydroxide precipitation (enhanced with calcium phosphate precipitation) followed by a lanthanum fluoride matrix removal step. Strontium is separated from the alkaline matrix using a carbonate precipitation, followed by a calcium fluoride precipitation. The method is applicable to the sodium hydroxide fusion of concrete and brick samples, prior to the chemical separation procedures described in the following procedures:

- Rapid Radiochemical Method for Americium-241 in Building Materials for Environmental Remediation Following Radiological Incidents
- Rapid Radiochemical Method for Plutonium-238 and Plutonium-239/240 in Building Materials for Environmental Remediation Following Radiological Incidents
- Rapid Radiochemical Method for Radium-226 in Building Materials for Environmental Remediation Following Radiological Incidents
- Rapid Radiochemical Method for Total Radiostrontium (Strontium-90) in Building Materials for Environmental Remediation Following Radiological Incidents
- Rapid Radiochemical Method for Isotopic Uranium in Building Materials for Environmental Remediation Following Radiological Incidents

Special Considerations: Large amounts of extraneous debris (pebbles larger than one quarter inch, nonsoil related debris) are not generally considered to be part of a concrete or brick matrix. When consistent with data quality objectives (DQOs), these materials should be removed from the sample, prior to drying. In those samples where native constituents may be present that could interfere with the determination of the chemical yield (e.g., strontium for strontium-90 analysis) or with the creation of a sample test source (e.g., barium for radium-226 analysis by alpha spectrometry), it may be necessary to determine the concentration of the native constituents in advance of chemical separation (using a separate aliquant of fused material) and make appropriate adjustments to the yield calculations or amount of carrier added. Re-used zirconium crucibles should be cleaned very well using soap and water, followed by warm nitric acid, and a final water rinse. Blank measurements should be monitored to ensure effective cleaning. Additional information regarding potential interferences and procedures for addressing the interferences is provided in Section 4 of the method.

Source: U.S. EPA, National Air and Radiation Environmental Laboratory (NAREL). April 2014. Rev 0 "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," EPA 402-R-14-004. <u>http://www.epa.gov/sam/pdfs/EPA-402-R14-004.pdf</u>