

**September 2014 Update:** EPA has validated and published a rapid method for sodium hydroxide/sodium peroxide fusion of radioisotope thermoelectric generator materials in water and air filter matrices for analysis of plutonium-238 and plutonium-239. The method is summarized and accessible through the link provided below.

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### **Rapid Method for Sodium Hydroxide/Sodium Peroxide Fusion of Radioisotope Thermoelectric Generator Materials in Water and Air Filter Matrices Prior to Plutonium Analyses 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 Spectrophotometry

**Method Developed for:** Plutonium-238 and plutonium-239 in water and air filters

**Method Selected for:** SAM lists this method for qualitative analysis of concrete or brick building materials

**Description of Method:** This method is a pre-treatment technique, to be used prior to use of the rapid separation method “Rapid Radiochemical Method for Plutonium-238 and Plutonium 239/240 in Building Materials for Environmental Remediation Following Radiological Incidents” (Reference 16.1 in the method). The method is based on total dissolution of radioisotope thermoelectric generator (RTG) materials in water or air filter samples; it may be adapted for application to brick and concrete sample materials after destruction of organic matter, but must be validated by the laboratory prior to use. Air filters are fused using rapid sodium hydroxide/sodium peroxide at 700 °C. For water samples, refractory RTG particles are collected on a 0.45µm filter using a vacuum. RTG activity remaining in the aqueous filtrate is preconcentrated using calcium phosphate precipitation. Solid fractions and filtrate fractions are processed separately by fusing with sodium hydroxide/sodium peroxide prior to subsequent chemical separation and alpha spectrometric analysis. The fusion method requires approximately one hour per batch of twenty samples using multiple furnaces. Pre-concentration steps are needed to eliminate the alkaline fusion matrix and collect the radionuclides, and require approximately two hours. Plutonium is separated from the fusion matrix using a lanthanum/calcium fluoride matrix removal step in preparation for separation and analysis using the rapid separation method cited above. Assuming a 68 m<sup>3</sup> air volume, the method is capable of meeting a required minimum detectable concentration (MDC) of 0.003 pCi/m<sup>3</sup> or below for air filters. Assuming a 1L volume, the method is capable of meeting a required MDC of 0.23 pCi/L for water samples (filtered solids, filtrate, or combined result).

**Special Considerations:** Organic based materials, such as cellulose nitrate or cellulose acetate filters, may react vigorously upon addition of peroxide or during charring steps. Wet-ashing with nitric acid and hydrogen peroxide is needed to destroy organic constituents prior to fusion. Samples with elevated activity or samples that require multiple analyses may need to be split after dissolution. Analytical parameters, such as the duration of the sample count and the aliquant size, should be modified to achieve optimal throughput (see Section 4.3 of the method for examples). All plutonium must be reduced to plutonium (+3 or +4) before isotopic exchange with the tracer can be achieved with reasonable certainty. Additionally, only plutonium (+3 or +4) will precipitate in the lanthanum fluoride/calcium fluoride pre-concentration step. Although peroxide may reduce plutonium+6 to +4, the valence must be controlled with certainty (see Section 4.6 for valence control procedures). Valence controls also ensure that plutonium will be present in the plutonium +4 form prior to separation on TEVA™ resin. Although this method was validated using plutonium-242 tracer, plutonium-236 tracer may be used assuming traceable material can be obtained with sufficient purity.

**Source:** U.S. EPA, National Air and Radiation Environmental Laboratory (NAREL). April 2014. Rev 0 “Rapid Method for Sodium Hydroxide/Sodium Peroxide Fusion of Radioisotope Thermoelectric Generator Materials in Water and Air Filter Matrices Prior to Plutonium Analyses for Environmental Remediation Following Radiological Incidents,” EPA 402-R14-003.  
<http://www2.epa.gov/radiation/rapid-radiochemical-methods-selected-radionuclides>