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May 2017
EPA 402-R16-003
Revision 0

Rapid Method for Sodium Hydroxide Fusion of Asphalt Roofing Material Matrices Prior to Americium, Plutonium, Strontium, Radium, and Uranium Analyses

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Revision History

Revision 0	Original release.	05-01-2017

This report was prepared for the National Analytical Radiation Environmental Laboratory of the Office of Radiation and Indoor Air and the National Homeland Security Research Center of the U.S. Environmental Protection Agency's (EPA) Office of Research and Development. It was prepared by Environmental Management Support, Inc., of Silver Spring, Maryland, under contract EP-W-13-016, task order 014, managed by Dan Askren. This document has been reviewed in accordance with EPA policy and approved for publication. Note that approval does not signify that the contents necessarily reflect the views of the Agency. Mention of trade names, products, or services does not convey EPA approval, endorsement, or recommendation.

RAPID METHOD FOR SODIUM HYDROXIDE FUSION OF ASPHALT ROOFING MATERIAL MATRICES PRIOR TO AMERICIUM, PLUTONIUM, STRONTIUM, RADIUM, AND URANIUM ANALYSES

1. Scope and Application

    1.1. The method is applicable to the sodium hydroxide (NaOH) fusion of asphalt roofing material samples, prior to the chemical separation procedures described in the following procedures:

        1.1.1. Rapid Radiochemical Method for Americium-241 in Building Materials for Environmental Remediation Following Radiological Incidents (Reference 16.1).

        1.1.2. Rapid Radiochemical Method for Plutonium-238 and Plutonium-239/240 in Building Materials for Environmental Remediation Following Radiological Incidents (Reference 16.2).

        1.1.3. Rapid Radiochemical Method for Radium-226 in Building Materials for Environmental Remediation Following Radiological Incidents (Reference 16.3).

        1.1.4. Rapid Radiochemical Method for Total Radiostrontium (Sr-90) in Building Materials for Environmental Remediation Following Radiological Incidents (Reference 16.4).

        1.1.5. Rapid Radiochemical Method for Isotopic Uranium in Building Materials for Environmental Remediation Following Radiological Incidents (Reference 16.5).

    1.2. This general method applies to asphalt roofing material samples collected following a radiological or nuclear incident. The term “asphalt roofing materials” is used in this procedure to mean asphalt organic shingles or asphalt fiberglass shingles typically used for residential or commercial roofs. This roofing material procedure was validated with asphalt fiberglass shingles, which are likely more difficult to analyze due to the presence of fiberglass. Other roofing types will need to be validated by the laboratory.

        1.2.1. Asphalt fiberglass shingles have a base layer of glass fiber reinforcing mat. The mat is made from wet, random-laid fiberglass bonded with urea-formaldehyde resin. The mat is then coated with asphalt which contains mineral fillers and makes the fiberglass shingle waterproof.

        1.2.2. Organic asphalt shingles generally have thicker organic (paper) matting saturated with asphalt to make it waterproof, then a top coating of adhesive asphalt is applied and ceramic granules are then embedded. This method should work with asphalt organic shingles and other bitumen-based membrane roofing materials, but this will have to be tested and validated by the laboratory. The organic asphalt shingles aliquants will likely have less residual ash than fiberglass shingles after the furnace heating step that destroys the organic components in the aliquant. The fusion method is expected to process the smaller organic shingles residue successfully. It is recommended that labs validate the method for organic asphalt shingles as well.

1.3. The fusion method is rapid and rigorous, effectively digesting refractory radionuclide particles that may be present on asphalt roofing material samples. Asphalt roofing material samples should be cut into very small subsample pieces (<0.25 g) to get a representative as-received sample of 100 - 250 g prior to taking a representative aliquant for furnace heating and fusion. The asphalt roofing material sample should be cut into pieces small enough so that representative subsamples can be taken and analyzed. Material sampling techniques to combine asphalt roofing subsamples that represent a larger roof surface area may be appropriate.

1.4. Asphalt roofing material samples should be cut into very small pieces to get a representative as-received sample of 100 - 250 g prior to taking a representative aliquant for furnace heating and fusion. The asphalt roofing material sample should be cut into pieces small enough so that representative subsamples can be taken and analyzed. Material sampling techniques to combine asphalt roofing subsamples that represent a larger roof surface area may be appropriate.

1.5. After a homogeneous subsample is obtained, the asphalt roofing material aliquant is heated to destroy organics in the sample matrix. After heating, the sample aliquant is fused to digest the asphalt roofing material and sample matrix. Matrix removal steps are employed to collect and preconcentrate the radionuclides from the alkaline fusion matrix.

1.5.1. A subsample (~25 g or larger) of asphalt roofing material sample is taken to facilitate representative sampling of a larger roofing material surface area (a 25 g sample represents approximately a 15 × 5 × 0.06 cm surface area). The 25 g sample subsample is ashed in a furnace and homogenized as well as possible. A smaller representative aliquant of the ashed roofing material is taken for analysis.

1.5.2. As this method is a sample digestion and pretreatment technique to be used prior to other separation and analysis methods, the user should refer to those individual methods and any project-specific requirements for the determination of applicable measurement quality objectives (MQOs).

1.6. Application of this method by any laboratory should be validated by the laboratory using the protocols provided in *Method Validation Guide for Qualifying Methods Used by Radioanalytical Laboratories Participating in Incident Response Activities* (Reference 16.6), or the protocols published by a recognized standards organization for method validation.

1.6.1. In the absence of project-specific guidance, MQOs for asphalt roofing samples may be based on the analytical action levels (AALs), required method uncertainty (u_{MR}), and the required relative method uncertainty (φ_{mr}) found in the *Radiological Sample Analysis Guide for Incidents of National Significance — Radionuclides in Soil* (Reference 16.7).

2. Summary of Method

   2.1. The method is based on ashing a 25 g subsample of asphalt roofing material sample in a furnace to remove organic components, followed by taking a representative aliquant from the ashed sample. The 1–1.5 g aliquant of ashed sample is fused using NaOH fusion at 600 °C.

   2.2. Plutonium (Pu), uranium (U), and americium (Am) are separated from the alkaline matrix using an iron (Fe)/titanium hydroxide precipitation (enhanced with calcium phosphate precipitation) followed by a lanthanum fluoride matrix removal step.

   2.3. Strontium (Sr) is separated from the alkaline matrix using a phosphate precipitation, followed by a calcium fluoride precipitation to remove silicates.

   2.4. Radium (Ra) is separated from the alkaline matrix using a carbonate precipitation.

   2.5. The resulting solutions are subsequently processed using the methods referred to in Steps 1.1.1–1.1.5.

3. Definitions, Abbreviations and Acronyms

   3.1. Discrete Radioactive Particles (DRPs or "hot particles"). Particulate matter in a sample of any matrix where a high concentration of radioactive material is present as a very small particle (<millimeter [mm]).

   3.2. *Multi-Agency Radiological Analytical Laboratory Protocols (MARLAP) Manual* (Reference 16.8).

   3.3. The use of the term asphalt roofing materials throughout this method is intended to mean asphalt fiberglass shingles.

   3.4. ASTM D228 (Reference 16.9) lists several types of asphalt roofing material materials below. This method was validated with ASTM Type 1 asphalt roofing materials. While this method may be effective with other asphalt and bitumen-based roofing materials, this will have to be validated by the laboratory.

      3.4.1. Type 1: A single thickness of glass felt, coated with asphalt and mineral surfacing such as in Specifications D2178 (Reference 16.11), D3462 (Reference 16.12), D4601 (Reference 16.13), and D4897 (Reference 16.14).

      3.4.2. Type 2: A single thickness of asphalt-saturated felt coated with asphalt and mineral surfacing such as in specifications D225 (Reference 16.15), D2626 (Reference 16.16), and D6380 (Reference 16.17).

      3.4.3. Type 3: Similar to Type 2, but asphalt-coated and surfaced with mineral granules for part of one side of the saturated felt such as in Specification D6380 (Reference 16.17), Class WS.

      3.4.4. Type 4: This material is comprised of multiple layers bonded with a suitable adhesive, typically a bituminous material.

4. Interferences and Limitations

   4.1. The bitumen components, which may have affinity for the radionuclides, are destroyed in this method. Radionuclides deposited on the surface of the asphalt roofing material are effectively digested, including refractory radionuclide particles. A small amount of mineralized granules may remain after the fusion.

4.2. Information regarding the elemental composition of the sample may be helpful. For example, asphalt roofing materials may have native concentrations of U, Ra, thorium, stable Sr, or stable barium (Ba), all of which may have an effect on the chemical separations used following the fusion of the sample. In some cases (e.g., Sr analysis), elemental analysis of the digestate prior to chemical separations may be necessary to determine native concentrations of carrier elements present in the sample.

NOTE: In those samples where native constituents are present that could interfere with the determination of the chemical yield (e.g., Sr for ^{90}Sr analysis) or with the creation of a sample test source (e.g., Ba for ^{226}Ra analysis by alpha spectrometry), it may be necessary to determine the concentration of these 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.

4.3. The native stable Sr in asphalt shingles may have an impact on the Sr-90 method. Asphalt shingles may contain enough stable Sr to have a slight, yet significant impact on the Sr carrier chemical yield measurement used in the Sr-90 method. This will cause Sr carrier yields that are biased high and therefore Sr-90 measurements that are biased low. While a higher level Sr carrier (7 milligram [mg]) is used to mitigate this effect, the digestion and analysis of samples to determine stable Sr content or analysis of several shingle aliquots with no Sr carrier added may be needed to make reliable Sr carrier chemical yield measurements. The analysis of representative shingles aliquots with no carrier added may be the approach with the least impact on the laboratory during an emergency and the most efficient way to correct for the small amount of stable Sr in the asphalt shingle samples.

4.4. Matrix blanks for these matrices may not be practical to obtain. Efforts should be made to obtain independent, analyte-free materials that have similar composition as the samples to be analyzed. These blanks will serve as process monitors for the fusion, and as potential monitors for cross contamination during batch processing.

4.5. Uncontaminated asphalt roofing material may be acceptable blank material for Pu, Am, and Sr analyses, but this material will contain background levels of naturally occurring U and Ra isotopes.

4.5.1. If analyte-free blank material is not available and an empty crucible is used to generate a reagent blank sample, it is recommended that ~100–150 mg calcium (Ca) per sample aliquant be added as calcium nitrate ($Ca(NO_3)_2$) to the empty crucible as blank simulant. This step facilitates strontium phosphate and radium carbonate precipitations from the alkaline fusion matrix.

4.5.2. Tracer yields may be slightly lower for reagent blank matrices, since asphalt roofing material matrix components, such as Ca, typically enhance recoveries across the precipitation steps since Ca in the native asphalt materials facilitates analyte precipitation across the preconcentration steps used.

4.6. Samples with elevated activity or samples that require multiple analyses from a single aliquant may need to be split after dissolution. In these cases the initial digestate and the split fractions should be carefully measured to ensure that the sample aliquant for analysis is accurately determined.

4.6.1. Tracer or carrier amounts (added for yield determination) may be increased where the split allows for the normal added amount to be present in the subsequent aliquant. For very high activity samples, the addition of the tracer or carrier may need to be postponed until following the split, in which case special care must be taken to ensure that the process is quantitative until isotopic exchange with the yield monitor is achieved. This deviation from the method should be thoroughly documented and reported in the case narrative.

4.6.2. When this method is employed and the entire volume of fused sample is processed in the subsequent chemical separation method, the original sample size and units are used in all calculations, with the final results reported in the units requested by the project manager.

4.7. Batch blank samples, laboratory control samples (LCSs), and sample duplicates should be created as early in the process as possible, following the same tracer/carrier additions, digestion process, and sample splitting used for the field samples. In this method, quality control (QC) samples should be initiated at the point samples are aliquanted into crucibles for the fusion.

4.8. Zirconium crucibles used in the furnace ashing and fusion process may be reused.

4.8.1. Before reuse, the crucibles should be cleaned very well using soap and water, followed by warm nitric acid, HNO_3 (multiple rinses), and then water. Blank measurements should be monitored to ensure effective cleaning and control against cross-contamination.

4.8.2. Segregation of crucibles used for low and high activity samples is recommended to minimize the risk of cross-contamination while maximizing the efficient use of crucibles.

4.9. Centrifuge speeds of 3500 revolutions per minute (rpm) are recommended but lower rpm speeds may be used if 3500 rpm is not available. Longer centrifuge times may be needed with lower speeds.

4.10. Aluminum nitrate reagent typically contains trace levels of U contamination and may have a slight impact on the U isotopic method at very low levels. To achieve the lowest possible blanks for isotopic U measurements, some labs have removed the trace U by passing ~250 mL of the 2M aluminum nitrate reagent through ~7 mL TRU Resin, but this will have to be tested and validated by the laboratory.

4.11. It is very important to withdraw a representative subsample from as homogeneous and representative a sample aliquant of the asphalt roofing material as possible to allow reliable assessment of radiological contamination of the samples taken. The asphalt roofing sample, as received, is cut into small pieces (0.25 g or less if possible), combined, and pieces of the sample are randomly taken to comprise a 25 g sample aliquant (nominally 100-125 pieces, representing approximately a 15 × 5 ×

0.06 cm surface area) for analysis. A 1–1.5 g subsample aliquant of the ashed sample is taken for analysis. The mass used for calculation of results is corrected based on the relative mass of the ashed and as-received sample (i.e., percent ash). Enough shingles sample should be collected in the field so that 100-250 g of the asphalt shingles may be collected for sub-sampling and analysis. Replicate samples may be taken to minimize concerns about representative sampling.

4.12. The asphalt shingles should be cut into as small a pieces as possible (0.2 g or less) to facilitate homogenizing the residual ash for subsampling, in particular to minimize the size of the residual fiberglass so it can be effectively blended with the ash.

5. Safety

5.1. General

5.1.1. Refer to your laboratory's safety manual for concerns of contamination control, personal exposure monitoring and radiation dose monitoring.

5.1.2. Refer to your laboratory's chemical hygiene plan (or equivalent) for general safety rules regarding chemicals in the workplace.

5.2. Radiological

5.2.1. Hot particles (DRPs)

5.2.1.1. Hot particles, also termed "discrete radioactive particles" (DRPs), will likely be small, on the order of 1 mm or less. DRPs typically are not evenly distributed in the media and their radiation emissions are not uniform in all directions (anisotropic).

5.2.1.2. Asphalt roofing material media should be individually surveyed using a thickness of the solid sample that is appropriate for detection of the radionuclide decay particles.

NOTE: The information regarding DRPs should accompany the samples during processing as well as be described in the case narrative that accompanies the sample results.

5.2.1.3. It is assumed that radiological activity from surface deposition onto the asphalt roofing material is primarily in the homogenized ash material and not in any strands or clumps of fiberglass from the shingle matting remaining with the ash. Small amounts of the fiberglass may be analyzed, however, if desired.

5.3. Procedure-Specific Non-Radiological Hazards:

5.3.1. The furnace ashing and NaOH fusion are performed in a furnace at ~550 °C and 600 °C, respectively. The operator should exercise extreme care when using the furnace and when handling the hot crucibles. Long tongs are recommended. Thermal protection gloves are also recommended when performing this part of the procedure. The fusion furnace should be used in a well-ventilated area (hood, trunk exhaust, etc.).

5.3.2. Particular attention should be paid to the use of hydrofluoric acid (HF). HF is an extremely dangerous chemical used in the preparation of some of the reagents and in the microprecipitation procedure. Appropriate personal protective equipment (PPE) must be used in strict accordance with the laboratory safety program specification.

6. Equipment and Supplies

6.1. Adjustable temperature laboratory hotplates.

6.2. Balance, top loading or analytical, readout display of at least ± 0.01 g.

6.3. Beakers, Pyrex, 600 mL, 100 mL, 150 mL capacity.

6.4. Centrifuge able to accommodate 225 mL tubes. (250 mL tubes are acceptable)

6.5. Centrifuge tubes, 50 mL and 225 mL capacity.

6.6. Crucibles, 250 mL, zirconium, with lids.

6.7. 100 microliters (µL), 200 µL, 500 µL, and 1 mL pipets or equivalent and appropriate plastic tips.

6.8. 1-10 mL electronic/manual pipet(s).

6.9. Hot water bath or dry bath equivalent.

6.10. Metal snips or shears

NOTE: See Appendix A for a method for cutting and homogenization of asphalt roofing material samples.

6.11. Muffle furnace capable of reaching at least 600 ^{o}C.

6.12. Tongs for handling crucibles (small and long tongs).

6.13. Tweezers or forceps.

6.14. Vortex stirrer.

7. Reagents and Standards

NOTES: Unless otherwise indicated, all references to water should be understood to mean Type I reagent water (ASTM D1193; Reference 16.10).

All reagents are American Chemical Society grade or equivalent unless otherwise specified.

7.1. Type I reagent water as defined in ASTM Standard D1193 (Reference 16.10).

7.2. Aluminum (Al) nitrate solution, $Al(NO_3)_3$ (2M): Add 750 g of aluminum nitrate nonahydrate ($Al(NO_3)_3 \bullet 9H_2O$) to ~700 mL of water and dilute with water to 1 L.

7.3. Ammonium hydrogen phosphate (3.2M): Dissolve 106 g of $(NH_4)_2HPO_4$ in 200 mL of water, heat on low to medium heat on a hot plate to dissolve and dilute with water to 250 mL.

7.4. Boric Acid, H_3BO_3.

7.5. Calcium nitrate, $Ca(NO_3)_2$, (1.25M): Dissolve 147 g of calcium nitrate tetrahydrate ($Ca(NO_3)_2 \bullet 4H_2O$) in 300 mL of water and dilute with water to 500 mL.

7.6. Fe carrier (50 mg/mL): Dissolve 181 g of ferric nitrate ($Fe(NO_3)_3 \bullet 9H_2O$) dissolved in 300 mL water and dilute with water to 500 mL. This carrier does not need to be standardized.

7.7. Hydrochloric acid, HCl (12M): Concentrated HCl.

7.7.1. HCl (0.01M): Add 0.83 mL of concentrated HCl to 800 mL of water and dilute with water to 1 L.

7.7.2. HCl (1.5M): Add 125 mL of concentrated HCl to 800 mL of water and dilute with water to 1 L.

7.8. Hydrofluoric acid, HF (28M): Concentrated HF.

7.9. Lanthanum (La) carrier (1.0 mg La^{3+}/mL): Add 1.56 g lanthanum (III) nitrate hexahydrate ($La(NO_3)_3 \bullet 6H_2O$) in 300 mL water, dilute with water to 500 mL. This carrier does not need to be standardized.

7.10. Nitric acid, HNO_3 (16M): Concentrated HNO_3.

7.10.1. HNO_3 (3M): Add 191 mL of concentrated HNO_3 to 700 mL of water and dilute with water to 1 L.

7.10.2. HNO_3 (3M)– H_3BO_3 (0.25M) solution: Add 15.4 g of H_3BO_3 and 190 mL of concentrated HNO_3 to 500 mL of water, heat to dissolve, and dilute with water to 1 L.

7.10.3. HNO_3 (7M): Add 443 mL of concentrated HNO_3 to 400 mL of water and dilute with water to 1 L.

7.10.4. HNO_3 (8M): Add 506 mL of concentrated HNO_3 to 400 mL of water and dilute with water to 1 L.

7.11. Sodium carbonate, Na_2CO_3 (2M): Dissolve 212 g anhydrous Na_2CO_3 in 800 mL of water, then dilute with water to 1 L.

7.12. NaOH pellets.

7.13. Titanium (III) chloride solution ($TiCl_3$), 10 percent by mass (wt%) solution in 20–30 wt% HCl. (This reagent is typically available commercially in this concentration.)

7.14. Radioactive tracers/carriers (used as yield monitors) and spiking solutions. A radiotracer is a radioactive isotope of the analyte that is added to the sample to measure any losses of the analyte. A carrier is a stable isotope form of a radionuclide (usually the analyte) added to increase the total amount of that element so that a measureable mass of the element is present. A carrier can be used to determine the yield of the chemical process and/or to carry the analyte or radiotracer through the chemical process. Refer to the chemical separation method(s) to be employed upon completion of this dissolution technique. Tracers/carriers that are used to monitor radiochemical/chemical yield should be added to a 1-1.5 g aliquant of the ashed sample. This allows for monitoring and correction of chemical losses in the combined digestion process, as well as in the chemical separation method. Carriers used to prepare sample test sources but not used for chemical yield determination (e.g., cerium added for microprecipitation of Pu or U), should be added where indicated.

8. Sample Collection, Preservation, and Storage

Not Applicable.

9. Quality Control

9.1. Where the subsequent chemical separation technique requires the addition of carriers and radioactive tracers for chemical yield determinations, these are to be added prior to beginning the fusion procedure, unless there is good technical justification for doing otherwise (e.g., significantly elevated activity).

9.2. Quality control samples are generally specified in the laboratory's quality manual or in a project's analytical protocol specifications (APS). At the very minimum, the following are suggested:

9.2.1. A LCS, which consists solely of the reagents used in this procedure and a known quantity of radionuclide spiking solution, shall be run with each batch of samples. The concentration of the LCS should be at or near the action level or level of interest for the project.

9.2.2. One reagent blank shall be run with each batch of samples. The blank should consist solely of the reagents used in this procedure (including tracer or carrier from the analytical method added prior to the fusion process).

9.2.3. A sample duplicate that is equal in size to the original aliquant should be analyzed with each batch of samples. This provides assurance that the laboratory's sample size reduction and subsampling processes are reproducible.

9.3. Batch quality control results shall be evaluated and meet applicable APS prior to release of unqualified data. In the absence of project-defined APS or a project-specific quality assurance project plan (QAPP), the quality control sample acceptance criteria defined in the laboratory's Quality Manual and procedures shall be used to determine acceptable performance for this method.

10. Calibration and Standardization.

10.1. Refer to the individual chemical separation and analysis methods for calibration and standardization protocols.

11. Procedure

11.1. Fusion

11.1.1. In accordance with the data quality objectives (DQOs) and sample processing requirements stated in the project plan documents, remove extraneous materials from the asphalt roofing material sample using a clean forceps or tweezers.

11.1.2. Weigh a labeled 250 mL (or 400 mL) glass beaker. Record the tare weight.

11.1.3. Weigh out a representative ~25 g (or larger) subsample into a 250 ml or 400 mL glass beaker and record the beaker plus sample weight. See Appendix A for the asphalt roofing materials sampling procedure.

11.1.4. Place the labeled beakers in a furnace at ~200 °C using tongs, ramp temperature to 350 °C for ~30 minutes, ramp to 450 °C for ~15 minutes, and ramp to 550 °C-600 °C and heat for ~3 hours (or until black tar color is gone and a grey ash remains).

11.1.5. Remove the beakers using tongs and allow to cool.

11.1.6. Weigh each beaker and record the final weight.

11.1.7. Transfer the ashed sample to a small plastic sample container. Using a Teflon or stainless steel spatula or equivalent, loosen and transfer the ashed solids, scraping the beaker as needed to transfer the solids.

NOTE: Transfer the fiberglass that is present into the sample tube or container and mix well with the ashed solids. The sample container should have enough space so that effective mixing can occur when vortexing.

11.1.8. Cap and mix well with a vortex mixer to homogenize sample. Use a spatula (pressing and twisting) to grind up the fiberglass into the ash as well as possible.

11.1.9. Weigh out a representative 1–1.5 g aliquant of the ashed asphalt roofing sample into a 250 mL zirconium crucible.

NOTE: The weight loss on ashing will be used to calculate the equivalent weight of pre-ashed sample to allow the proper sample aliquant calculations. For example, if 1.5 g of ash is taken for assay and the weight loss was about 20%, the ashed samples will represent ~1.8 g of as received asphalt shingle. For Sr and Ra analyses, a reagent blank of 150 mg Ca (prepared by evaporating 3 mL of 1.25M $Ca(NO_3)_2$ for each sample) should be added to the crucible as a blank simulant to ensure the blank behaves like the asphalt roofing samples during the precipitation steps.

11.1.10. Add the proper amount of tracer or carrier appropriate for the method being used and the number of aliquants needed.

11.1.11. Place crucibles on a hot plate and heat to dryness on medium heat.

NOTE: Heat on medium heat to dry quickly but not so high as to cause splattering.

11.1.12. Add the following amounts of NaOH based on the aliquant size/analysis required.

1.5 g for Pu, Am, U, Sr:	15 g NaOH
1 g for Ra:	10 g NaOH

11.1.13. Place the crucibles with lids in the 600 °C furnace using tongs.

11.1.14. Fuse samples in the crucibles for ~20 minutes.

NOTE: Longer times may be needed for larger particles.

11.1.15. Remove hot crucibles from furnace very carefully using tongs, and transfer to hood.

11.1.16. Add ~25-50 mL of water to each crucible ~8 to 10 minutes (or longer) after removing crucibles from furnace, and heat on hotplate to loosen and dissolve solids.

11.1.17. If necessary for dissolution, add more water and warm as needed on a hotplate.

11.1.18. Proceed to Section 11.2 for the actinide preconcentration procedure, 11.3 for Sr preconcentration, or 11.4 for Ra preconcentration steps.

11.2. Preconcentration of Actinides (Pu, U, or Am) from Hydroxide Matrix

11.2.1. Pipet 1 mL of Fe carrier (50 mg/mL) into a labeled 225 mL centrifuge tube for each sample.

11.2.2. Pipet 1 mg La/mL to each tube as follows:

Pu, U: 5 mL, 1 mg, La/mL
Am: 3 mL, 1 mg, La/mL

11.2.3. Transfer each fused sample to a 225 mL centrifuge tube, rinse crucibles well with water, and transfer rinses to each tube.

NOTE: A final rinse of 5 mL 3M HNO_3 may be added to rinse each crucible and then added to tube.

11.2.4. Dilute each sample to ~180 mL with water.

11.2.5. Cool the 225 mL centrifuge tubes in an ice bath to approximately room temperature.

11.2.6. Pipet 1 mL 1.25M $Ca(NO_3)_2$ and 5 mL 3.2M $(NH_4)_2HPO_4$ into each tube.

11.2.7. Cap tubes and mix well.

11.2.8. Pipet 10 mL of 10 wt% $TiCl_3$ into each tube, and cap and mix immediately.

11.2.9. Cool 225 mL centrifuge tubes in an ice bath for approximately 10 minutes.

11.2.10. Centrifuge tubes for approximately 6 minutes at 3500 rpm or more or as needed.

11.2.11. Pour off the supernatant and discard to waste.

11.2.12. Add 1.5M HCl to each tube to redissolve each sample in a total volume of ~60 mL.

11.2.13. Cap and shake each tube to dissolve solids as well as possible.

11.2.14. Dilute each tube to ~170 mL with 0.01M HCl.

11.2.15. Pipet 1 mL of 1.0 mg La/mL into each tube. Cap and mix.

11.2.16. Pipet 5 mL of 10 wt% $TiCl_3$ into each tube. Cap and mix.

11.2.17. Add ~25 mL of concentrated HF into each tube. Cap and mix well.

11.2.18. Cool in ice bath for approximately 10 minutes to facilitate the precipitation.

11.2.19. Centrifuge for approximately 5 to 10 minutes at 3500 rpm or more or as needed.

11.2.20. Pour off supernatant, and discard to waste.

11.2.21. Pipet 6 mL of 3M HNO_3 – 0.25M H_3BO_3 into each tube.

11.2.22. Cap, mix and transfer contents of the tube into a labeled 50 mL centrifuge tube.

11.2.23. Pipet 7 mL of 7M HNO_3 and 8 mL of 2M $Al(NO_3)_3$ into each tube, cap and mix (shake or use a vortex stirrer), and transfer rinse to 50 mL centrifuge tube.

11.2.24. Pipet 3 mL of 3M HNO_3 directly into the 50 mL centrifuge tube.

11.2.25. Warm each 50 mL centrifuge tube in a hot water bath (or dry hot block) for a few minutes, swirling to dissolve.

11.2.26. Allow each 50 mL centrifuge tube to cool to room temperature

11.2.27. Centrifuge the 50 mL centrifuge tubes at 3500 rpm for 5 minutes to remove any traces of solids (may not be visible prior to centrifuging), and transfer solutions to labeled beakers or tubes for further processing. Discard any solids.

11.2.28. Proceed directly to the Am, Pu, or U methods listed in Steps 1.1.1, 1.1.2, or 1.1.5.

11.3. Preconcentration of ^{90}Sr from Hydroxide Matrix

11.3.1. Transfer each fused sample to a 225 mL centrifuge tube, rinse crucibles well with water, and transfer rinses to each tube.

11.3.2. Dilute each sample to ~150 mL with water.

11.3.3. Cool the 225 mL centrifuge tubes in an ice bath to approximately room temperature.

11.3.4. Pipet 1.5 mL 1.25M $Ca(NO_3)_2$, 1 mL 50 mg/mL Fe carrier and 5 mL 3.2M $(NH_4)_2HPO_4$ into each tube.

11.3.5. Cap tubes and mix well.

11.3.6. Allow 225 mL centrifuge tubes to sit for approximately 10 minutes.

11.3.7. Centrifuge tubes for approximately 6 minutes at 3500 rpm or more or as needed.

11.3.8. Pour off the supernatant and discard to waste.

11.3.9. Add 1.5M HCl to each tube to redissolve each sample in a total volume of ~60 mL.

11.3.10. Cap and shake each tube to dissolve solids as well as possible.

11.3.11. Dilute sample aliquant in 225 mL tube to ~170 mL with 0.01M HCl.

11.3.12. Cap and mix tubes.

11.3.13. Add ~25 mL of concentrated HF into each tube. Cap and mix well.

11.3.14. Allow 225 mL tubes to sit for approximately 10 minutes.

11.3.15. Centrifuge for approximately 5 to 10 minutes at 3500 rpm or more or as needed.

11.3.16. Pour off supernatant and discard to waste.

11.3.17. Pipet 5 mL of concentrated HNO_3 and 5 mL of 3M HNO_3 – 0.25M H_3BO_3 into each 225 mL centrifuge tube to dissolve precipitate.

11.3.18. Cap and mix well. Transfer contents of the tube into a labeled 50 mL centrifuge tube.

11.3.19. Pipet 7 mL of 3M HNO_3 and 7 mL of 2M $Al(NO_3)_3$ into each tube, cap tube and mix.

11.3.20. Transfer the rinse solutions to 50 mL centrifuge tubes and mix well (shake or use vortex stirrer).

11.3.21. Centrifuge the 50 mL tubes at 3500 rpm for 5 minutes to remove any traces of solids.

11.3.22. Transfer solutions to labeled beakers or new 50 mL centrifuge tubes for further processing.

11.3.23. If solids remain, add 5 mL 3M HNO_3 to each tube, cap and mix well by shaking or vortex stirrer, centrifuge for 5 minutes, and add the supernatant to the sample solution. Discard any residual solids.

11.3.24. Set aside for ^{90}Sr analysis using *Rapid Radiochemical Method for Total Radiostrontium (Sr-90) in Building Materials for Environmental Remediation Following Radiological Incidents* (Step 1.1.4).

11.4. Preconcentration of ^{226}Ra from Hydroxide Matrix

11.4.1. Transfer each sample to a 225 mL centrifuge tube, rinse crucibles well with water, and transfer rinses to each tube.

11.4.2. Dilute to ~150 mL with water.

11.4.3. Add 15 mL concentrated HCl to each tube.

11.4.4. Cap and mix each tube well.

11.4.5. Pipet 1 mL 1.25M $Ca(NO_3)_2$ into each tube.

11.4.6. Add 10 mL 2M Na_2CO_3 to each tube.

11.4.7. Cap tubes and mix.

11.4.8. Allow tubes to stand for approximately 10 minutes.

11.4.9. Centrifuge tubes for 6 minutes at 3500 rpm.

11.4.10. Pour off the supernatant and discard to waste.

11.4.11. Pipet 10 mL 1.5M HCl into each tube to dissolve precipitate. Cap and mix.

11.4.12. Transfer sample solution to a 50 mL centrifuge tube.

11.4.13. Pipet 10 mL 1.5M HCl into each 225 mL centrifuge tube to rinse. Cap and rinse well.

11.4.14. Transfer rinse solution to 50 mL tube and mix well by shaking well or vortex stirrer.

NOTE: Typically, the HCl added to dissolve the carbonate precipitate is sufficient to acidify the sample. If the precipitate was unusually large and suspended solids remain, additional acid may be needed. The pH can be checked to verify it is pH 1 or less. To acidify the pH <1, add 1 or 2 mL of concentrated hydrochloric acid to the solution and get it to clear. Tubes may be warmed in a water bath to help dissolve samples.

11.4.15. If solids remain, add 5 mL 1.5M HCl to each tube, cap and mix well, centrifuge for 5 minutes and add the supernatant liquid to the sample solution. Discard any residual solids.

11.4.16. Set aside for ^{226}Ra analysis using *Rapid Radiochemical Method for Radium-226 in Bitumen Aggregate, Stone or other Solid Samples for Environmental Remediation Following Radiological Incidents* (Step 1.1.3).

12. Data Analysis and Calculations

12.1. Equations for determination of final result, combined standard uncertainty, and radiochemical yield (if required) are found in the corresponding chemical separation and analysis methods, with the units being provided by the project manager.

12.2. An asphalt roofing subsample (~25 g) is ashed in a furnace at 550 °C. A 1–1.5 g aliquant of the ashed sample is taken for dissolution and analysis. The weight loss of the subsample is used to determine the amount of as-received sample contained in the 1 –1.5 g ashed aliquant. Tracers are not added until the 1–1.5 g sample aliquant is taken.

12.3. The following calculation is performed to determine the amount of as-received (or dried sample; see A12.1) asphalt roofing sample contained in the 1–1.5 g ashed aliquant.

$$\mathrm{M_c} = M_s \times \frac{\mathrm{M_i} - \mathrm{M_b}}{\mathrm{M_a} - \mathrm{M}_b} \tag{1}$$

M_b = empty beaker tare weight (g)
M_i = total weight of initial as-received sample weight + beaker (g)
M_a = total weight of ashed sample + beaker (g)
M_s = aliquant of ash taken for analysis (g)
M_c = aliquant corrected for weight loss (g)

12.3.1. If the sample aliquants taken for analysis are split or serially diluted during the fusion sample preparation method, the initial activity of tracer added to the sample aliquant is used for calculations. The aliquant size used for calculations, however, must be the effective amount of sample in the aliquant into which the tracer is added. It is calculated as follows:

$$W_a = W_s \times d_1 \times d_2 \times d_3 \qquad (2)$$

Where

$$d_1 = \frac{D_{a1}}{D_{s1}}, d_2 = \frac{D_{a2}}{D_{s2}}, and \;\; d_3 = \frac{D_{a3}}{D_{s3}} \qquad (3)$$

and

W_a = sample aliquant size, used for analysis, in the units designated by the project manager (e.g., kg, g, etc.).

W_s = initial size of the sample aliquant taken for fusion in the units designated in analytical protocol specifications (e.g., kg, g, etc.).

$D_{a\#}$ = mass or volume of the aliquant taken (i.e., the redissolved fusion cake or subsequent dilution thereof) where # denotes the respective number of the serial dilution from i to n, (e.g., 5.0 mL, etc.).

$D_{s\#}$ = mass or volume of the digestate from which $D_{a\#}$ is taken, where # denotes the number of the respective serial dilution from i to n (e.g., 20 mL, etc.).

NOTE: The $D_{a\#}$ and $D_{s\#}$ must use the same units of mass or volume. If fewer than three splits/dilutions are made, a factor of one (1) is substituted for the unneeded D_a and D_s terms. If no splits or dilutions are performed, $W_s = W_a$.

12.3.2. The actual activity of tracer added to the sample is used in the calculation of the final sample results as described in Step 12.3.1. If the sample has been split or diluted, the tracer activity used to calculate the radiochemical yield must be modified to reflect the activity of tracer that would be present theoretically in the final sample test source assuming 100% radiochemical yield. It is calculated as follows:

$$A_{t-yld} = A_t \times d_1 \times d_2 \times d_3 \qquad (4)$$

Where

$A_{t\text{-yld}}$ = theoretical activity in sample test source assuming 100% radiochemical yield

A_t = activity of the tracer added to the sample aliquant at the reference date/time for the tracer

and

d_1, d_2 and d_3 are defined as described in Step 12.3.1.

NOTE: The $D_{a\#}$ and $D_{s\#}$ must use the same units of mass or volume. If fewer than three splits/dilutions are made, a factor of one (1) is substituted for the unneeded D_a and D_s terms. If no splits or dilutions are performed, $A_{t\text{-yld}} = A_t$.

13. Method Performance

13.1. Method validation results are to be reported.

13.2. Expected turnaround time per sample.

13.2.1. For a representative 1 g aliquant of sample, the furnace heating and fusion steps should add approximately 2 hours per batch to the time specified in the individual chemical separation methods.

13.2.2. The preconcentration steps should add approximately 2 to 2.5 hours per batch.

NOTE: Processing times for the subsequent chemical separation methods are given in those methods for batch preparations.

14. Pollution Prevention

This method utilizes small volume (2 mL) extraction chromatographic resin columns. This approach leads to a significant reduction in the volumes of load, rinse and strip solutions, as compared to classical methods using ion exchange resins or solvent extraction techniques to separate and purify the radionuclide fractions.

15. Waste Management

15.1. Refer to the appropriate chemical separation methods for waste disposal information.

16. References

Cited References

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16.2. EPA 2013. *Rapid Radiochemical Method for Pu-238 and Pu-239/240 in Building Materials for Environmental Remediation Following Radiological Incidents*. Office of Air and Radiation, Washington, DC. Available here.

16.3. EPA. *Improved Rapid Radiochemical Method for Radium-226 in Building Materials for Environmental Remediation Following Radiological Incidents*. Office of Air and Radiation, Washington, DC. Not yet available.

16.4. EPA 2013. *Rapid Radiochemical Method for Total Radiostrontium (Sr-90) in Building Materials for Environmental Remediation Following Radiological Incidents*. Office of Air and Radiation, Washington, DC. Available here

16.5. EPA 2013. *Rapid Radiochemical Method for Isotopic Uranium in Building Materials for Environmental Remediation Following Radiological Incidents*. Office of Air and Radiation, Washington, DC. Available here.

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16.7. EPA 2012. *Radiological Laboratory Sample Analysis Guide for Incident Response — Radionuclides in Soil*. Revision 0. Office of Air and Radiation, Washington, DC. EPA 402-R-12-006, September. Available here. EPA 2004.

16.8. EPA 2004. *Multi-Agency Radiological Laboratory Analytical Protocols Manual* (MARLAP). 2004. Volumes 1 – 3. Washington, DC: EPA 402-B-04-001A-C, NUREG 1576, NTIS PB2004-105421, July. Available here.

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16.10. ASTM D1193. "Standard Specification for Reagent Water" ASTM Book of Standards 11.01, current version, ASTM International, West Conshohocken, PA.

16.11. ASTM D2178. "Specification for Glass Felt Asphalt Shingles Used in Roofing and Waterproofing" For Annual ASTM Book of Standards volume information, refer to the standard's Document Summary page on ASTM website at www.astm.org.

16.12. ASTM D3462. "Specification for Asphalt Shingles Made from Glass Felt and Surfaced with Mineral Granules" For Annual ASTM Book of Standards volume information, refer to the standard's Document Summary page on ASTM website at www.astm.org.

16.13. ASTM D4601. "Specification for Asphalt-Coated Glass Fiber Base Sheet Used in Roofing" For Annual ASTM Book of Standards volume information, refer to the standard's Document Summary page on ASTM website at www.astm.org.

16.14. ASTM D4897. "Specification for Asphalt Shingles (Organic Felt) Surfaced with Mineral Granules" For Annual ASTM Book of Standards volume information, refer to the standard's Document Summary page on ASTM website at www.astm.org.

16.15. ASTM D225. "Specification for Glass Felt Asphalt Shingles Used in Roofing and Waterproofing" For Annual ASTM Book of Standards volume information, refer to the standard's Document Summary page on ASTM website at www.astm.org.

16.16. ASTM D2626. "Specification for Asphalt-Saturated and Coated Organic Felt Base Sheet Used in Roofing" For Annual ASTM Book of Standards volume information, refer to the standard's Document Summary page on ASTM website at www.astm.org.

16.17. ASTM D6380. "Specification for Asphalt Roll Roofing (Organic Felt)" For Annual ASTM Book of Standards volume information, refer to the standard's Document Summary page on ASTM website at www.astm.org.

Other References

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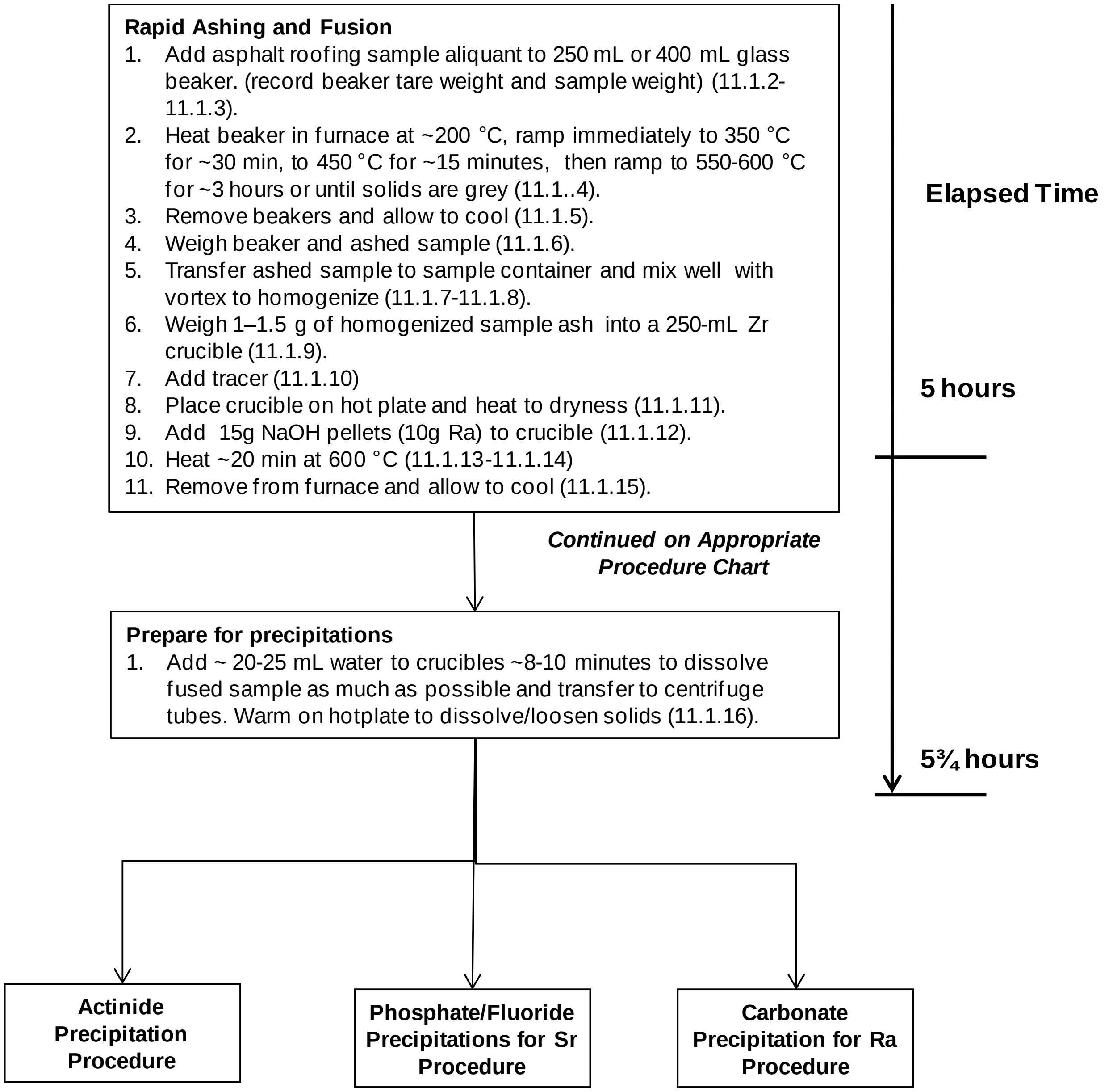
17. Tables, Diagrams, and Flow Charts

17.1. Fusion Flow Chart

Timeline for Rapid Fusion and Preparation of Asphalt Roofing Samples for Precipitation and Analysis



Rapid Ashing and Fusion

1. Add asphalt roofing sample aliquant to 250 mL or 400 mL glass beaker. (record beaker tare weight and sample weight) (11.1.2-11.1.3).
2. Heat beaker in furnace at ~200 °C, ramp immediately to 350 °C for ~30 min, to 450 °C for ~15 minutes, then ramp to 550-600 °C for ~3 hours or until solids are grey (11.1..4).
3. Remove beakers and allow to cool (11.1.5).
4. Weigh beaker and ashed sample (11.1.6).
5. Transfer ashed sample to sample container and mix well with vortex to homogenize (11.1.7-11.1.8).
6. Weigh 1–1.5 g of homogenized sample ash into a 250-mL Zr crucible (11.1.9).
7. Add tracer (11.1.10)
8. Place crucible on hot plate and heat to dryness (11.1.11).
9. Add 15g NaOH pellets (10g Ra) to crucible (11.1.12).
10. Heat ~20 min at 600 °C (11.1.13-11.1.14)
11. Remove from furnace and allow to cool (11.1.15).

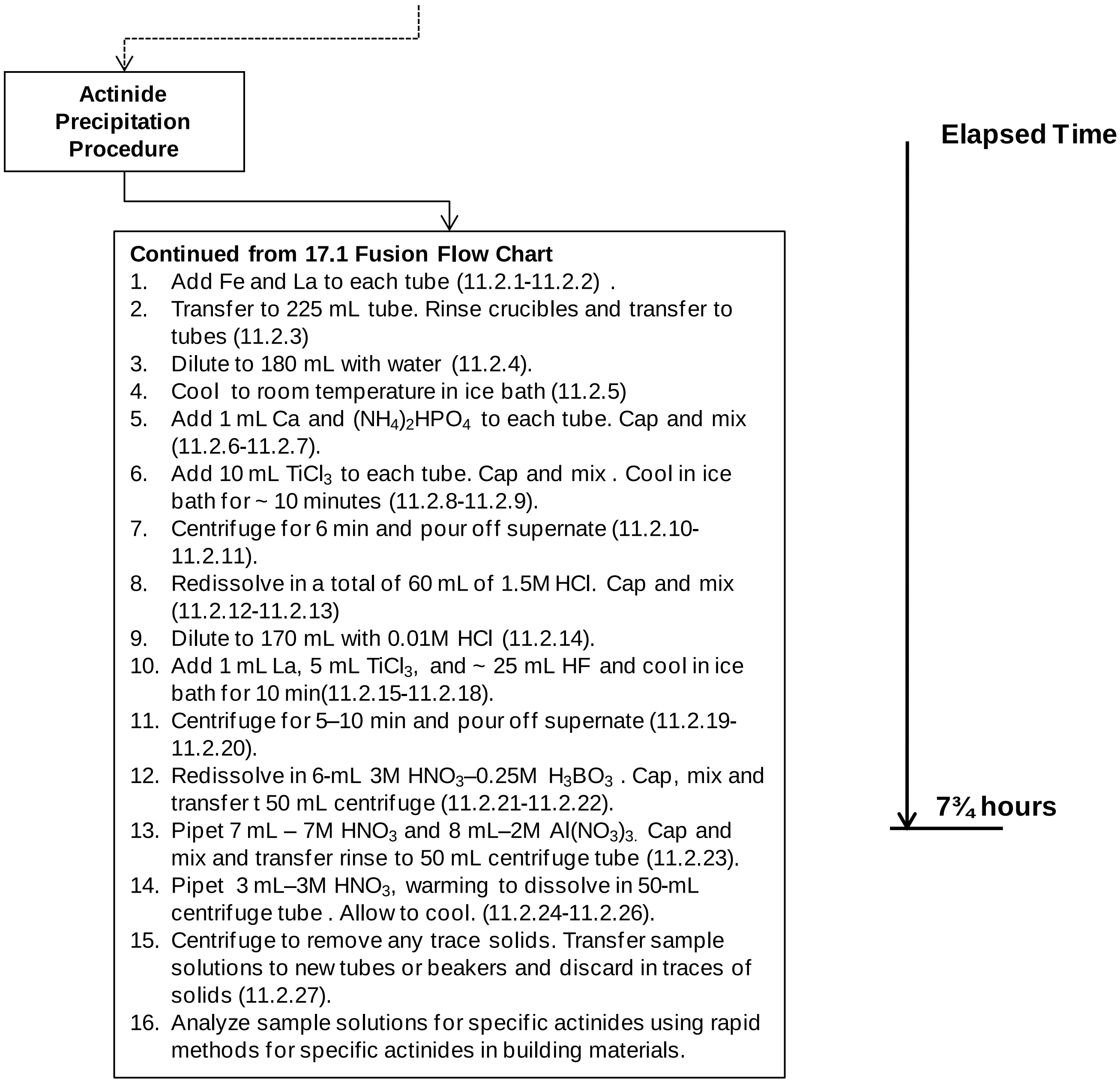
Continued on Appropriate Procedure Chart

Prepare for precipitations

1. Add ~ 20-25 mL water to crucibles ~8-10 minutes to dissolve fused sample as much as possible and transfer to centrifuge tubes. Warm on hotplate to dissolve/loosen solids (11.1.16).

17.2. Actinide Precipitation Flow Chart

Actinide Precipitation Procedure



Continued from 17.1 Fusion Flow Chart

1. Add Fe and La to each tube (11.2.1-11.2.2) .
2. Transfer to 225 mL tube. Rinse crucibles and transfer to tubes (11.2.3)
3. Dilute to 180 mL with water (11.2.4).
4. Cool to room temperature in ice bath (11.2.5)
5. Add 1 mL Ca and $(NH_4)_2HPO_4$ to each tube. Cap and mix (11.2.6-11.2.7).
6. Add 10 mL $TiCl_3$ to each tube. Cap and mix . Cool in ice bath for ~ 10 minutes (11.2.8-11.2.9).
7. Centrifuge for 6 min and pour off supernate (11.2.10-11.2.11).
8. Redissolve in a total of 60 mL of 1.5M HCl. Cap and mix (11.2.12-11.2.13)
9. Dilute to 170 mL with 0.01M HCl (11.2.14).
10. Add 1 mL La, 5 mL $TiCl_3$, and ~ 25 mL HF and cool in ice bath for 10 min(11.2.15-11.2.18).
11. Centrifuge for 5–10 min and pour off supernate (11.2.19-11.2.20).
12. Redissolve in 6-mL 3M HNO_3–0.25M H_3BO_3 . Cap, mix and transfer t 50 mL centrifuge (11.2.21-11.2.22).
13. Pipet 7 mL – 7M HNO_3 and 8 mL–2M $Al(NO_3)_{3.}$ Cap and mix and transfer rinse to 50 mL centrifuge tube (11.2.23).
14. Pipet 3 mL–3M HNO_3, warming to dissolve in 50-mL centrifuge tube . Allow to cool. (11.2.24-11.2.26).
15. Centrifuge to remove any trace solids. Transfer sample solutions to new tubes or beakers and discard in traces of solids (11.2.27).
16. Analyze sample solutions for specific actinides using rapid methods for specific actinides in building materials.

17.3. Strontium Precipitation Flow Chart

Strontium Precipitation Procedure

$Ca_3(PO_4)_2$ / CaF_2 Precipitation for Sr Procedure

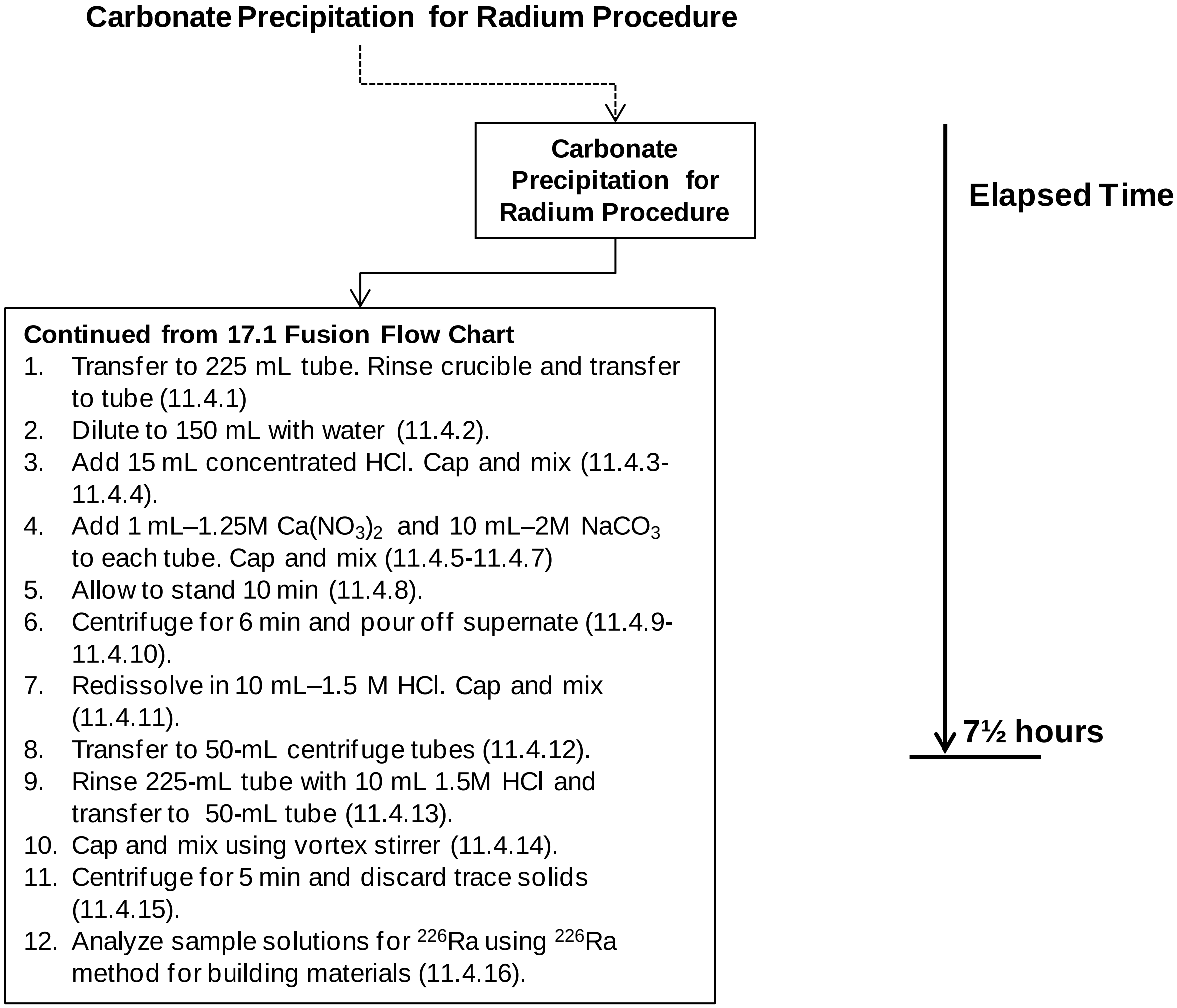
Elapsed Time

Continued from 17.1 Fusion Flow Chart

1. Transfer to 225 mL tube. Rinse crucible and transfer to tube (11.3.1)
2. Dilute to 150 mL with water (11.3.2).
3. Cool in ice bath to room temperature (11.3.3).
4. Add 1.5 mL 1.25 M $Ca(NO_3)_2$, 1 mL Fe, and 5 mL 3.2M $(NH_4)_2HPO_4$ to each tube . Cap and mix (11.3.4-11.3.5).
5. Allow 225-mL tubes to sit for ~10 min (11.3.6).
6. Centrifuge for 6 min and pour off supernate (11.3.7-11.3.8).
7. Redissolve in 1.5M HCl to a total of 60 mL. Cap and mix (11.3.9-11.3.10).
8. Dilute to 170 mL with 0.01M HCl. Cap and mix (11.3.11-11.3.12).
9. Add 25 mL concentrated HF and wait ~10 min (11.3.13-11.3.14).
10. Centrifuge for 5–10 min and pour off supernate (11.3.15-11.3.16).
11. Redissolve in 5 mL 3M HNO_3-0.25M H_3BO_3 and 5 mL concentrated HNO_3. Cap, mix and transfer to 50 mL centrifuge tube (11.3.17-11.3.18)
12. Pipet 7 mL 2M $Al(NO_3)_3$ + 7 mL–3M HNO_3. Cap and mix well. Transfer rinse solution to 50 mL tubes and mix well (11.3.19-.11.3.20).
13. Centrifuge for 5 min and discard trace solids. Transfer to 50 mL tubes (11.3.21-11.3.22).
14. Analyze sample solutions for ^{90}Sr using ^{90}Sr method for building materials.

7¾ hours

17.4. Radium Precipitation Flow Chart



Continued from 17.1 Fusion Flow Chart

1. Transfer to 225 mL tube. Rinse crucible and transfer to tube (11.4.1)
2. Dilute to 150 mL with water (11.4.2).
3. Add 15 mL concentrated HCl. Cap and mix (11.4.3-11.4.4).
4. Add 1 mL–1.25M $Ca(NO_3)_2$ and 10 mL–2M $NaCO_3$ to each tube. Cap and mix (11.4.5-11.4.7)
5. Allow to stand 10 min (11.4.8).
6. Centrifuge for 6 min and pour off supernate (11.4.9-11.4.10).
7. Redissolve in 10 mL–1.5 M HCl. Cap and mix (11.4.11).
8. Transfer to 50-mL centrifuge tubes (11.4.12).
9. Rinse 225-mL tube with 10 mL 1.5M HCl and transfer to 50-mL tube (11.4.13).
10. Cap and mix using vortex stirrer (11.4.14).
11. Centrifuge for 5 min and discard trace solids (11.4.15).
12. Analyze sample solutions for ^{226}Ra using ^{226}Ra method for building materials (11.4.16).

Appendix A:

Rapid Technique for Sampling Asphalt Roofing Material

A1. Scope and Application

A1.1. The goal is to obtain representative sample aliquants from the asphalt roofing material received.

A1.2. This method describes one approach for the rapid preparation of asphalt roofing materials samples to yield representative aliquants for radiochemical analysis of non-volatile radionuclides.

A1.3. The method is designed to be used as a preparatory step for the attached methods for furnace heating and fusion of asphalt roofing materials for measurement of ^{241}Am, ^{238}Pu, $^{239/240}$Pu, U, ^{90}Sr, and ^{226}Ra.

A2. Summary of Methods

A2.1. This method uses disposable equipment or materials to contact the sample, where possible, minimizing the risk of contamination and cross-contamination and eliminating concerns about adequate cleaning of equipment.

A2.2. The asphalt roofing material, as-received is cut into as small pieces as possible to allow a ~25 g subsample to be taken, which will be ashed in a furnace, homogenized, as well as possible, and a smaller aliquant of the ashed sample is taken. Additional replicate analyses may also be performed to provide further assurance that analytical results are representative of asphalt roofing material samples received.

A3. Definitions, Abbreviations, and Acronyms

A3.1. Discrete Radioactive Particles (DRPs or “hot particles”). Particulate matter in a sample of any matrix where a high concentration of radioactive material is present as a very small particle (<millimeter [mm]).

A3.2. *Multi-Agency Radiological Analytical Laboratory Protocols (MARLAP) Manual* (Reference A16.1).

A4. Interferences

A4.1. Radiological Interferences

A4.1.1. This method assumes that radioactivity in the sample is primarily adsorbed onto the surface of particles, as opposed to being present as a hot particle. (See discussion of DRPs in A4.1.2.)

A4.1.2. If the sample was collected near the epicenter of a radiological dispersal device (RDD) or improvised nuclear device (IND) explosion, it may contain millimeter- to micrometer-sized particles of contaminant referred to as DRPs. DRPs may consist of small pieces of the original radioactive source and thus may have very high specific activity. They may also consist of chemically intractable material and present special challenges in the analytical process.

A5. Safety

A5.1. General

A5.1.1. Refer to your laboratory's safety manual for concerns of contamination control, personal exposure monitoring, and radiation dose monitoring.

A5.1.2. Refer to your laboratory's chemical hygiene plan (or equivalent) for general safety rules regarding chemicals in the workplace.

A5.2. Radiological

A5.2.1. Refer to your radiation safety manual for direction on working with known or suspected radioactive materials.

A5.2.2. This method has the potential to generate airborne radioactive contamination. The process should be carefully evaluated to ensure that airborne contamination is maintained at acceptable levels. This should take into account the activity level, and physical and chemical form of contaminants possibly present, as well as other engineering and administrative controls available.

A5.2.3. Hot particles (DRPs)

A5.2.3.1. Hot particles, also termed "discrete radioactive particles" (DRPs), will usually be small, on the order of 1 mm or less. DRPs typically are not evenly distributed in the media and their radiation emissions are not uniform in all directions (anisotropic).

A6. Equipment and supplies

A6.1. Balance, top-loading, range to accommodate sample size encountered, readability to ±1%, or better.

A6.2. Drying oven, at 110±10 ºC.

A6.3. Disposable scoop, scraper, tongue depressor or equivalent.

A6.4. Metal snips to cut the asphalt roofing material samples.

A6.5. Plastic bag.

A6.6. Steel paint cans and lids (pint, quart, 2 quart, 1 gallon, as needed), used to dry asphalt roofing material if needed.

A7. Reagents and Standards

No reagents needed.

A8. Sample Collection, Preservation and Storage

A8.1. Samples should be collected in appropriately sized plastic bags or other containers.

A9. Quality Control

A9.1. Batch quality control results shall be evaluated and meet applicable analytical protocol specifications (APSs) prior to release of unqualified data. In the absence of project-defined APS or a project-specific quality assurance project plan (QAPP), the quality control sample acceptance criteria defined in the laboratory quality manual and procedures shall be used to determine acceptable performance for this method.

A10. Procedure

A10.1. Prepare the asphalt roofing sample for subsampling.

NOTE: Asphalt roofing material samples do not typically contain large amounts of moisture. Large pieces of asphalt roofing material will require cutting into smaller pieces.

A10.1.1. Spread a large piece of plastic in a hood (or alternate paper material which can be discarded to waste later).

A10.1.2. Remove the asphalt roofing sample for the sample container.

A10.1.2.1. Take representative samples randomly from different areas of the asphalt roofing material by cutting the asphalt shingle material into smaller sections using metal snips (unless already cut into smaller pieces).

A10.1.2.2. Cut enough of the asphalt roofing material from the sections or larger pieces into very small subsample pieces (<0.5 g) to get a representative as-received sample of 100–250 g.

NOTE: Clean the metal snips between use with different samples.

A10.2. If the asphalt roofing material is visibly wet (or if requested), place the cut pieces of asphalt shingles into a can or other container that can be heated (without lid) in an oven at 110±10 ºC and dry the asphalt roofing material.

NOTES: Asphalt roofing material samples will typically be dry enough such that drying prior to taking the subsample aliquant is not required, however it may be received wet due to weather conditions. In the event samples are received that contain moisture, the samples may be dried in a drying oven at 110±10 ºC prior to taking the aliquant. It may be difficult to obtain a constant weight with continued volatility of organics upon heating.

A10.3. Weigh the combined mass of the can/container, sample, and lid. If the percent solids are required calculations. Remove can from oven and allow to cool. Place lid on can for storage and weigh with lid on container.

A10.4. Store the asphalt roofing material sample in a properly labeled sample can, jar, or bag.

A11. Calibration and Standardization

A11.1. Balances used shall be calibrated using National Institute of Standards and Technology (NIST)-traceable weights according to the process defined by the laboratory’s quality manual.

A12. Data Analysis and Calculations

NOTE: Drying of asphalt roofing materials is not required unless the material received is visibly wet or damp due to weather conditions. It may be difficult to dry to a constant weight. It may be difficult to obtain a constant weight with continued volatility of organics upon heating.

A12.1. The percent solids (dry-to-as-received mass ratio) for each sample is calculated from data obtained during the preparation of the sample as follows:

$$\%\ \text{Solids} = \frac{M_{\text{dry}} - M_{\text{tare}}}{M_{\text{as rec}} - M_{\text{tare}}} \times 100$$

where:

M_{dry} = mass of dry sample + labeled can + lid (g)
M_{tare} = tare mass of labeled can + lid (g)
$M_{\text{as rec}}$ = mass of sample as received + labeled can + lid (g)

A12.2. If dried, convert the equivalent mass of sample, as received, to dry mass. Dry mass is calculated from a measurement of the total as received mass of the sample received as follows:

$$\text{Dry Sample Equivalent} = M_{\text{total-as rec.}} \times \frac{\%\ \text{Solids}}{100}$$

where:

$M_{\text{total-as rec}}$ = total mass of sample, as received (g)

A12.3. Results Reporting

A12.3.1. The result for percent solids and the approximate total mass of sample as received should be reported for each result.

A13. Method Performance

A13.1. Results of method validation performance are to be archived and available for reporting purposes.

A13.2. Expected turnaround time for these sample preparation steps is about 3 hours for an individual sample and about 4 hours per batch.

A14. Pollution Prevention.

Not applicable.

A15. Waste Management

A15.1. All radioactive and other regulated wastes shall be handled according to prevailing regulations.

A16. References

A16.1. EPA 2004. *Multi-Agency Radiological Laboratory Analytical Protocols Manual* (MARLAP). 2004. Volumes 1 – 3. Washington, DC: EPA 402-B-04-001A-C, NUREG 1576, NTIS PB2004-105421, July. Available here.

A16.2. International Union of Pure and Applied Chemistry (IUPAC) 1997. Compendium of Chemical Terminology, 2nd ed. (the “Gold Book”). Compiled by A. D. McNaught and A. Wilkinson. Blackwell Scientific Publications, Oxford. XML on-line corrected version: http://goldbook.iupac.org/C01265.html (2006-) created by M. Nic, J. Jirat, B. Kosata; updates compiled by A. Jenkins. Last update: 2010-12-22.