Bulk Dissolution Standard Operating Procedure

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Details required by EHS:
Experiment performed in rooms 012 and 024 of Green Earth Science Building. Eyewashes/safety showers/hoods/lab equipment are regularly checked and maintained, by EHS as well as lab users. Do not raise fume hoods further than maximum height. HF safety information is posted next to the First Aid kit (near lab computer) and calcium glauconate is stored in the refrigerator in the Chemistry Lab (012). During all HF work, please wear 1) safety goggles, 2) long-sleeved apron, 3) double gloved, 4) lab coat and 5) closed toe shoes and long pants under lab coat. Please never work with HF alone. All researchers have Tier 1, Tier 2 and Tier 3 training and have been lectured on HF first aid procedures and risks associated with this chemical.

Purpose:
This method is utilized to dissolve sediment, bones and soils, bringing all components into solution (usually for further analysis). Total experiment time is 3 days. This experiment is not performed regularly. It is common for a researcher to go through 3 or 4 batches (10-20 samples) in a row and then not run any samples for several months.

Chemicals Used:

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Concentration</th>
<th>Safety Procedures</th>
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</thead>
<tbody>
<tr>
<td>Hydrofluoric Acid (HF)</td>
<td>40%</td>
<td>Vinyl lab coat covering neck to wrist to ankle, double gloved, safety goggles, lab coat, closed toe shoes/pants, fume hood down.</td>
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<tr>
<td>Nitric Acid</td>
<td>8 N and 1 N</td>
<td>Lab coat, gloves, safety goggles.</td>
</tr>
<tr>
<td>Hydrogen Peroxide</td>
<td>30%</td>
<td>Lab coat, gloves, safety goggles.</td>
</tr>
<tr>
<td>Hydrochloric Acid</td>
<td>30%</td>
<td>Lab coat, gloves, safety goggles.</td>
</tr>
</tbody>
</table>

Procedure:
1) (Steps 1-5 are in room 012). Sample (please see above) is added to an 8 ml teflon beaker. For hydrofluoric acid (HF) work (next step) protective gear includes safety goggles, HF apron (8 mm vinyl) which reaches from neck to wrists and down to feet and double gloved in nitrile.
2) Inside fume hood, beakers in secondary containment are arranged into row, with caps loosened. The secondary containment is clearly labeled “Danger: Contains Hydrofluoric Acid and 8 N Nitric Acid”. The fume hood door is lowered so there is just room for arms to manipulate the pipette.
3) 1 ml 8 N Nitric Acid is added, and caps are screwed on tightly.
4) 2 ml of 40% HF (all HF is this concentration) is added by pipette. The cap is lifted off each sample, the 1 ml is added and the cap is set on again (this is done twice). The HF is in a 20ml container (not the 500 ml container it comes in). The pipette tip is disposed of and exchanged for a new one (sitting ready in hood).
5) Beakers are placed in sonicator, which has been transported into the hood for this experiment. Sonication for 1 hour. Samples are transported back into hood in secondary containment. Beakers are opened and 2 ml of 30% hydrogen peroxide is added. This time, each beaker is opened and the reagent is added, then the beaker is closed.

6) Beakers are transported in secondary containment to an oven at 90°C, and placed on top of a sheet of aluminum foil in a secondary containment tray. The oven should be inside the fume hood. Samples remain in oven overnight.

7) Samples are placed in secondary containment and carried to the clean lab (room 024, a 20 foot travel). Here the samples are placed on a hot plate in a fume hood and dried down until almost all the liquid is gone. In the clean lab, I double glove and wear safety glasses, but do not have an HF apron. Determine if beakers are dry by holding beaker up to hood. Then 1 ml of 8 N Nitric Acid is added and dried down. At this point, if sample has come out of solution 1 ml of 40% Hydrochloric Acid is added. Sample is dried down again and 1 ml of 8 N Nitric Acid is added and dried down. Samples are removed from the hot plate at the end of 3 dry downs and reconstituted in 1 N nitric acid. Samples are then added to centrifuge tubes (15-50 ml). Samples are removed at a later date for analysis.

8) Teflon beakers are rinsed in DI water and soap and individually scrubbed. They are placed in a 25% Aqua Regia solution (12.5% Nitric Acid and 12.5% Hydrochloric acid) for 1 day, then in 25% Nitric Acid for 1 night, rinsed and dried.

9) Waste Disposal: As the procedure is described here, there is not waste…all of the sample is retained for analysis. However, once sample needs to be disposed of, the 1 N Nitric Acid is added to a waste container (wearing gloves, goggles and lab coat) and containers are disposed of.