USTUR 1010: Leaching Procedure for Radioanalysis of Incinerator Ash

Purpose	Incinerator Ash Analysis	**Method Number**	USTUR 1010
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Revision Number	1	**Approved By**	James T. Elliston
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1. Principle of Method

1.1. The pH of the incinerator ash is determined using a mixture of de-ionized water and dilute hydrochloric acid.

1.2. An appropriate extraction fluid, based on the ash pH, is used to leach an aliquot of ash for 18 hours.

1.3. A reagent blank is made with each batch of samples.

1.4. An aliquot of the leachate is counted by liquid scintillation and compared with the reagent blank to determine if radionuclides are present.

2. Apparatus

2.1. Hotplates: magnetic stirring

2.2. Hotplate thermometer

2.3. Watch glasses: Pyrex, plain to fit beakers

2.4. Beakers: 250 mL

2.5. Magnetic stir bars

2.6. Graduated Cylinders: various sizes

2.7. Erlenmeyer flasks: 250 mL

2.8. Stoppers: to fit Erlenmeyer flasks, non-reactive with extraction fluid used

2.9. Suction filter apparatus

2.10. Vacuum pump

2.11. Filters: glass fiber, 0 .6-0.9 um

2.12. Scintillation vials: 20 mL, glass, with poly-cone cap

2.13. Pipettes: various sizes

2.14. Balance: top-loading, capable of 0.01 g.

2.15. Spatulas

2.16. pH paper with range of ±0.5 from pH 5.

2.17. Tri-Carb 1900 CA Liquid Scintillation Analyzer

3. Reagents

3.1. 18 M Ω de-ionized water (D.I. Water)

3.2. Hydrochloric acid (concentrated 36.5 – 38% reagent grade)

3.3. Hydrochloric acid (1 M). Add 83 mL of concentrated HCl to 917 mL of D.I. water.

3.4. Glacial acetic acid (reagent grade)

3.5. Ecosint Scintillation cocktail

3.6. Sodium hydroxide (NaOH, reagent grade)

3.7. Sodium hydroxide (1 M). Weigh out 49,9 g of sodium hydroxide, dissolve in ~500 mL D.I. water and then bring to a total volume of 1000 mL with D.I. water.

3.8. Extraction Fluid #1: Add 5.7 mL glacial acetic acid to 500 mL D.I. water. Add 64.3 mL of 1 M NAOH and dilute to total volume of 1000 mL with D.I. water.

3.9. Extraction Fluid #2: Add 5.7 mL glacial acetic acid to D.I. water and bring to total volume of 1000 mL with D.I. water.

4. Procedure

4.1. Incinerator ash pH determination

4.1.1. Weigh out 5.0 g of ash in a 250 mL beaker.

4.1.2. Add 96.5 mL of D.I. water to beaker and a magnetic stir bar.

4.1.3. Stir vigorously on stirring hotplate for 5 minutes (with no heat)

4.1.4. Add 3.5 mL of 1 M HCl to solution, slurry briefly, cover with watch glass, heat to 50°C and hold at 50°C for 10 minutes.

4.1.5. Let the solution cool to room temperature and record pH, using pH paper.

4.1.6. If pH is <5 use extraction fluid #1 (see 3.8) in section 4.2.

4.1.7. If pH is >5 use extraction fluid #2 (see 3.9) in section 4.2.

4.2. Leaching

4.2.1. Weigh out 5.0 g of incinerator ash into a 250 mL Erlenmeyer flask.

NOTE: This is a new aliquot of ash and not the same aliquot in 4.1.

4.2.2. Add 100.0 mL of the appropriate extraction fluid (determined in 4.1.6) to the flask.

NOTE: Make a reagent blank to run with each batch of sample.

4.2.3. Add a magnetic stir bar to flask and stopper the flask.

4.2.4. Stir solution at a moderate speed on stirring hotplate (no heat) for 18 ± 2 hours.

4.2.5. After 1 hour check that no pressure is building in the stoppered flask. If there is it should be periodically relieved by loosening stopper.

4.2.6. After 18 ± 2 hours remove the sample from the stirring hotplate and separate leachate from the ash as per section 4.3.

4.3. Leachate Preparation

4.3.1. Set-up suction filter apparatus with secondary flask to catch any moisture from sample.

4.3.2. Filter leachate/ash mixture with filter apparatus. If necessary, change filter if it becomes clogged with ash, so all of the mixture is filtered.

4.3.3. Take a 4.0 mL aliquot of filtered leachate with a glass pipet or equivalent and add to glass scintillation vial.

4.3.4. Add 16.0 mL of Ecosint scintillation cocktail to scintillation vial and shake vigorously.

4.3.5. Allow vial to set in the dark until solution is clear and a single phase.

4.4. Counting

4.4.1. Count scintillation vials using protocol 25 (see addendum) Tri-Carb Scintillation Analyzer.

4.4.2. Put the reagent blank in the first slot and label the caps appropriately of each sample vial.

4.4.3. Wipe vials down with ethyl alcohol to ensure no fingerprints or other marks are on the glass portion of the vial.

4.5. Radionuclide Determination

4.5.1. Compare the counts per minute (cpm) of each sample in both the alpha and beta channel printouts to those of the reagent blank.

4.5.2. If cpm of samples are less then twice that of the reagent blank, the sample is considered non-radioactive. If cpm counts are high, re-count sample and blank to verify results.

4.5.3. Make a copy of the counting printouts with sample numbers identified and report to the Radiation Safety Office.

Reference

1. EPA SW 846 Method 1311: Toxicity Characteristic of Leaching Procedure.

ADDENDUM

Protocol # =	25	Copy Protocol #?	no
Protocol Name?	ustur acid waste		
Cycles?	1		
Count Time?	60.00		
2 Sigma Coincidence?	no		
# Counts/Vial?	1		
# Vials/Standard?	1	# Vials/Sample?	1
1st Vial Backgroung?	No		
Radionuclide?	Manual		

	LL	UL	Bkg	2 Sigma%	LCR
Region A:	2.0	50.0	0.00	.00	0
Region B:	51.0	500.	0.00	.00	0
Region C:	2.0	2000	0.00	.00	0

Qip?	tSIE	ES Terminator?	Count
% of Reference?	No		
Data Mode?	Cpm		