AP15

ANALYSIS OF POLONIUM-210 IN WATER, SOIL, AIR FILTERS, MISCELLANEOUS SAMPLES AND BIOTA

PART A

PRINCIPLE

Polonium in water is quantitatively deposited on a nickel disc from a hydrochloric acid solution. Polonium in air filters, soil, miscellaneous samples, and biota is leached using nitric acid and hydrogen peroxide. The samples are adjusted to $1\underline{M}$ HCl in the presence of ascorbic acid and hydroxylamine hydrochloride. The solution is stirred and heated to 80^OC and the polonium is spontaneously deposited onto a nickel disc. This is a very specific separation and can be carried out directly in the presence of many other radionuclides.

REFERENCES

1.   J.H. Harley, <u>HASL Procedures Manual, Suppl. 6</u>, 1978.

2.   F. B. Johns, et al., <u>Radiochemical Analytical Procedures for Analysis of Environmental Samples</u>, EMSL-LV-0539-17, March 1979.

3.   Beasley, T.M. and Osterberg, C.L., <u>Analytical Chemistry</u>, 41, #3, 541-43 (1969).

Certification Record for

AP15

ANALYSIS OF POLONIUM-210 IN
WATER, SOIL, AIR FILTERS,
MISCELLANEOUS SAMPLES AND BIOTA

CHECKPOINTS

1. **JOB HAZARD ANALYSIS** _____
2. **MSDS/HAZARDS DISCUSSED** _____
3. **SAMPLE DIGESTION** _____
4. **DEPOSITION** _____
5. **FINAL CALCULATIONS** _____

ANALYST'S SIGNATURE: _____

CERTIFIED BY: _____

DATE: _____

ANALYSIS VALUE: _____

KNOWN VALUE: _____

MEASURED/KNOWN: _____

See Task _____, Batch_____ for the original data.

COMMENTS:
_____
_____
_____
_____

PART B

1.0   PURPOSE AND SCOPE

This method is appropriate for the analysis of up to one gram soil or biota, one 10 cm (4-inch) diameter glass-fiber air filter, one liter of water, and other miscellaneous samples of various sizes.

2.0   REAGENTS

All chemicals are hazardous. **See Step 2.0 AP15 JHA.** See MSDS for specific precautions.

Ascorbic acid, saturated solution, prepared fresh daily; reagent grade

Acetone; reagent grade

Hydrochloric acid, (concentrated 12 $\underline{M}$); reagent grade

Hydrochloric acid (1 $\underline{M}$); Add 84 mL of 12 $\underline{M}$ hydrochloric acid to 900 mL of deionized water. Dilute to 1 L with deionized water

Hydroxylamine hydrochloride (50% w/v); Dissolve 50 g of reagent grade hydroxylamine hydrochloride in 100 mL of deionized water.

Nitric acid (concentrated 16 $\underline{M}$); reagent grade

Hydrogen peroxide, 35% (w/v); reagent grade

3.0   APPARATUS

Centrifuge tubes: 50 mL, disposable
Centrifuge
One inch diameter polished nickel discs
Hot Plate/Stirrer
Drill and drill bits
Rotary sander
Plastic twist ties
Stir bars

4.0   PROCEDURE

Before proceeding, you must be certified as indicated in QCP1 of this manual and Section 3 of the Quality Program (QP) Manual. See page 2 of this section for a copy of the certification record.

4.1   Water

    4.1.1   Measure water samples up to 1 L in a volumetric flask and transfer to an appropriate size beaker. Add enough concentrated hydrochloric acid to bring the HCl concentration to 0.5-1 $\underline{M}$ HCl. **Step 4.1.1 of AP15 JHA.**

    4.1.2   Add enough Po-209 tracer to obtain the desired counting statistics, typically 5-15 pCi. **Step 4.1.2 of AP15 JHA.**

    4.1.3   Evaporate to a volume of 20 mL and transfer to a 250 mL beaker. **Step 4.1.3 of AP15 JHA.**

    4.1.4   Add enough 1$\underline{M}$ HCl to fill the beaker 3/4 full. Add 2 mL saturated ascorbic acid and 2 mL 50% hydroxylamine hydrochloride. **Step 4.1.4 of AP15 JHA.**

    4.1.5   Prepare the nickel disc as follows: polish the nickel disc by sanding it with a rotary sander. Paint the unpolished side of the disc with an acid resistant acrylic paint and let dry. Drill a small hole near the edge of the disc with a 1/16" drill bit. **Step 4.1.5 of AP15 JHA.**

    4.1.6   Push a twist tie through the hole and twist each end to an uncoiled paper clip.

    4.1.7   Place the paper clip across the 250 mL beaker and heat to a temperature of 80°C and stir for 4 hours. **Step 4.1.7 of AP15 JHA.**

    4.1.8   Remove the disc, rinse with water air dry.

    4.1.9   Alpha count the disc. **Step 4.1.9 of AP15 JHA.**

4.2   Soil, Biota, Miscellaneous Samples, and Air Filters

    4.2.1   Weigh and transfer 0.5 to 1.0 g dried soil or biota, or an air filter, into a 250 mL beaker. For miscellaneous samples, use an appropriate sample quantity which may be the entire sample if the suspected activity is the result of surface contamination. Add enough Po-209 tracer to obtain the desired counting statistics, typically 5-15 pCi. Keep air filters as flat and close to bottom as possible. **Step 4.2.1 of AP15 JHA.**

    4.2.2   Add 10 mL 16 $\underline{M}$ nitric acid. Gently heat on a hot plate and carefully add 35% hydrogen peroxide dropwise. Gently swirl solution. Continue adding nitric acid and peroxide until sample digestion is achieved, usually after 1-2 hours. **Step 4.2.2 of AP15 JHA.**

    4.2.3   Slowly evaporate the solution to a volume of 5 mL. Do not allow the sample to go to dryness. **Step 4.2.3 of AP15 JHA.**

4.2.4   Transfer sample to centrifuge tube using 1<u>M</u> HCl. Centrifuge at 2000 rpm for five minutes. Decant and pour the supernate into a 250 mL beaker. Wash the sample by adding 10 mL 1<u>M</u> HCl to the centrifuge tube and vortex. Centrifuge at 2000 rpm for five minutes. Decant and pour the supernate into a 250 mL beaker. **Step 4.2.4 of AP15 JHA.**

4.2.5   Proceed with step 4.1.4.

5.0   <u>CALCULATIONS</u>

All necessary data is recorded and reduced using the following calculations:

$$Concentration = \frac{G - B}{T \cdot E \cdot Q \cdot Y \cdot 2.22}$$

$$2\sigma \ Error = \frac{1.96\sqrt{G + B}}{T \cdot E \cdot Y \cdot Q \cdot 2.22} = pCi/unit$$

$$2\sigma \ TPU = C \cdot 1.96 \sqrt{\frac{G + B}{((G - B)^2} + RE^2 + RY^2 + RQ^2} = pCi/unit$$

$$MDC = \frac{3 + 4.65\sqrt{B}}{T \cdot E \cdot Y \cdot Q} = pCi/unit$$

where:

B = background counts
C = concentration
E = detector efficiency, counts/disintegration
G = gross counts
MDC = minimum detectable concentration
Q = sample quantity
RE = 1σ relative error of the efficiency
RQ = 1σ relative error of the quantity
RY = 1σ relative error of the yield
T = length of count, minutes
TPU = total propagated uncertainty
Y = chemical yield
2.22 = conversion of dpm to pCi

6.0   RECORDS

    6.1   Reference QP Manual for record requirements.

    6.2   The raw count data are saved during the weekly backup of the Liquid Scintillation
            Analyzer to the ORISE network disks.

    6.3   Hard copies of assignment and calculation sheets are maintained in the archived task
            file. Electronic copies of assignment and calculation sheets are saved during the daily
            incremental backup of the network system. The following data sheets should be
            completed and retained:

            • Po-210 Analysis Assignment Form
            • Po-210 Lab Data Sheet
            • Po-210 Concentration and Uncertainty Report (This report may be generated
              using approved Excel spreadsheets or from the database, if available.)

AP15(Rev 1) - Po-210 ANALYSIS ASSIGNMENT FORM

Assigned To: _____ **Date:** _____ **Batch:** _____

Task #: _____ **LWR #:** _____ **Activity Lev*:** _____

Sample #'s: _____

_____

_____

ANALYSIS REQUIRED:

| Initials |

☐ Po Tracer # _____ Volume _____ Units _____ ☐

Pipet # _____ Weight _____ Volume _____

QC REQUIRED:

Blank ☒

LCS ☒ Po Std # _____ Quantity _____ Units _____ ☐ (Initials)

Replicate ☐ Sample # _____ # Replications _____

Matrix Spk ☐ Sample # _____

Po Std # _____ Volume _____ Units _____

SPECIAL INSTRUCTIONS: _____

_____

_____

_____

* If Activity Level is indicated as Moderate or High, perform area survey

AP15(Rev 1) - Po-210 ANALYSIS ASSIGNMENT FORM

Sample #								
Sample Quantity								
Quant. Units								

Sample #								
Sample Quantity								
Quant. Units								

Sample #								
Sample Quantity								
Quant. Units								

* If Activity Level is indicated as Moderate or High, perform area survey

** Indicate sample type (A=Ashed Soil, S=Unashed Soil, W=Water, X=Other, indicate)

batch #:   Acq Date:

AVERAGE EFFICIENCY: _____		YIELD: _____		CORRECTED TRACER: _____		SAMPLE QUANTITY _____	
DETECTOR #:							
SAMPLE ID	ISOTOPE	Conc. (pCi/g)	TPU (pCi/g)	MDC (pCi/g)	ENERGY (KEV)	SAMPLE COUNTS	DETECTOR BKG

AVERAGE EFFICIENCY: _____		YIELD: _____		CORRECTED TRACER: _____		SAMPLE QUANTITY _____	
DETECTOR #:							
SAMPLE ID	ISOTOPE	Conc. (pCi/g)	TPU (pCi/g)	MDC (pCi/g)	ENERGY (KEV)	SAMPLE COUNTS	DETECTOR BKG

AVERAGE EFFICIENCY: _____		YIELD: _____		CORRECTED TRACER: _____		SAMPLE QUANTITY _____	
DETECTOR #:							
SAMPLE ID	ISOTOPE	Conc. (pCi/g)	TPU (pCi/g)	MDC (pCi/g)	ENERGY (KEV)	SAMPLE COUNTS	DETECTOR BKG

LCS	LCS Known Activity (pCi)	LCS Known Uncertainty	Meas./Known Activity Ratio	Meas./Known Uncertainty Ratio
Po-210				

BLK CORRECT? YES[ ] NO[ ]
LCS CORRECT? YES[ ] NO[ ]
YIELDS CORRECT? YES[ ] NO[ ]
IF NO, SPECIFY REASON:

QC Review: _____
(Initials/Date)

_____ Analyst Review   _____ Date

_____ Reviewed By   _____ Date

_____ Given To: _____ Date

_____ QC Entered By   _____ Date