

# Determination of $^{227}\text{Ac}$ in Geological Samples

**Summary of Method** Soil or rock samples are pulverized to <1mm and dissolved, either by acid digestion or sodium hydroxide fusion.  $^{227}\text{Ac}$  is separated from matrix ions using a ferric hydroxide precipitation step. Following dissolution in 4M HCl,  $^{227}\text{Ac}$  is separated from radiometric impurities using a 2mL cartridge of DGA, Normal resin.  $^{227}\text{Ac}$  is prepared for measurement using a  $\text{CeF}_3$  microprecipitation onto Resolve<sup>(R)</sup> Filters. An  $^{225}\text{Ac}$ ( $^{229}\text{Th}$ ) tracer is used to measure chemical recovery of actinium. After a 30 minute ingrowth time, the  $^{225}\text{Ac}$  tracer yield is measured via alpha spectrometry using the  $^{221}\text{Fr}$  and  $^{217}\text{At}$  daughters of  $^{225}\text{Ac}$ .  $^{227}\text{Ac}$  is measured via its  $^{227}\text{Th}$  and  $^{223}\text{Ra}$  daughters after a longer period of ingrowth (30-90 days). Ac yields are typically 70-90%. MDA for  $^{227}\text{Ac}$  was 0.05Bq/kg for 3 day count times after 90 days ingrowth period.

## Reagents

DGA Resin, 2mL Cartridges (Eichrom DN-R50-S)  
 Iron Carrier (50mg/mL Fe, as ferric nitrate)  
 Cerium Carrier (10mg/mL)  
 $^{229}\text{Th}$ ( $^{225}\text{Ac}$ ) tracer  
 Hydrofluoric Acid (49%) or Sodium Fluoride  
 Boric acid                                     $\text{HNO}_3$  (70%)  
 HCl (37%)                                        NaOH  
 Deionized Water                                $\text{H}_2\text{O}_2$  (30%)  
 Optional for additional Th/U removal:  
 TRU Resin, 2mL cartridges (Eichrom TR-R50-S)

## Equipment

Vacuum Box (Eichrom AR-24-BOX or AR-12-BOX)  
 Cartridge Reservoir, 20mL (Eichrom AR-200-RV20)  
 Inner Support Tubes-PE (Eichrom AR-1000-TUBE-PE)  
 Yellow Outer Tips (Eichrom AR-1000-OT)  
 Resolve Filters in Funnel (Eichrom RF-DF25-25PP01)  
 50mL and 250mL Centrifuge Tubes  
 Alpha Spectrometry System  
 Ball mill grinder or equivalent  
 Centrifuge                                        Vacuum Pump  
 Heat Lamp                                         Analytical Balance

## Fusion Option

250mL Zirconium crucibles with zirconium lids  
 Muffle Furnace

## Digestion Option

Hot Plate  
 Teflon Beakers

## Sample Preparation

0.25-50g Soil or Rock

Pulverize to <1mm.

Aliquot Sample. Add  $^{229}\text{Th}$ ( $^{225}\text{Ac}$ ) tracer.

## Acid Digestion Option

Digest in Teflon beaker on hotplate with 2:1 conc.  $\text{HNO}_3$ :HF to near dryness.

Digest in Teflon beaker on hotplate with conc.  $\text{HNO}_3$  + Boric Acid.

Dissolve Residue in 4M HCl + 0.25M Boric acid. If solids remain. Repeat digestion. Proceed to ferric hydroxide precipitation.

## Fusion Option

In Zr crucible. Add 10-15g NaOH

Muffle at 600°C for 15-30 minutes.

Cool. Dissolve fusion cake with 50mL water. Heat as necessary. Rinse crucible with 50mL 4M HCl. Proceed to ferric hydroxide precipitation.

## Ferric Hydroxide Precipitation

Transfer sample to 250mL centrifuge tube.

Dilute to 150mL with water.

Add 25mg Fe carrier. Mix well.

Centrifuge 2500 rpm for 10 minutes.

Decant Supernate.

Rinse ppt with 50mL water. Centrifuge.

Decant Supernate.

Dissolve precipitate with 10mL conc. HCl.

Dilute to 30mL.

## Ac Separation on DGA

(1) Precondition 2mL DGA with 10mL 4M HCl.\*

(2) Load sample solution.

(3) Rinse sample tube with 5mL 4M HCl. Add tube rinse to DGA. (If TRU cartridge is used, remove following this step.)

(4) Rinse DGA with 10mL 3M HNO<sub>3</sub>.

(5) Rinse DGA with 20mL 0.5M HNO<sub>3</sub>.

(6) Strip Ac from DGA with 20mL 2M HCl. (2M HCl is used to achieve additional decontamination from Th.)

\* A 2mL cartridge of TRU resin may be added above DGA for additional decontamination from U/Th.



(7) Add 0.5mL 30% H<sub>2</sub>O<sub>2</sub> to samples.

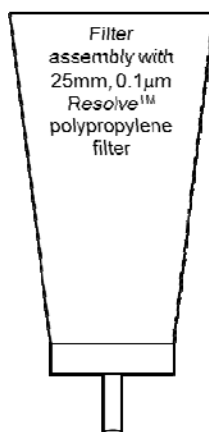
(8) Add 50ug Ce carrier to samples. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 minutes.

(9) Set up Resolve® Filter Funnel on vacuum box.

(10) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(11) Filter sample.

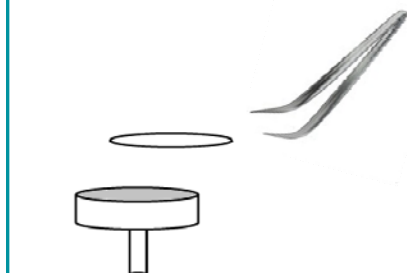
(12) Rinse sample tube with 5mL DI water and add to filter.



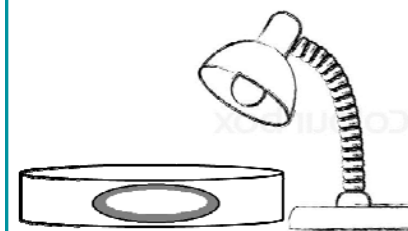
(13) Rinse filter funnel with 3mL DI water and 2mL 100% ethanol.

(14) Draw vacuum until filter is dry.

(15) Remove filter from funnel assembly and mount filter on stainless steel planchet with 2-sided tape.



(16) Dry filter under heat lamp for 3-5 minutes.



(17) Measure actinium or daughters by alpha spectrometry.

### Method Performance

Rock Standard	<sup>227</sup> Ac	<sup>227</sup> Ac	% Bias	Tracer Recovery
	Measured Bq/kg	Reference Bq/kg		
BCR-2	0.955 ± 0.083	0.967	-1.2	83
BHVO-1	0.299 ± 0.017	0.283	5.7	71
HK-018	0.965 ± 0.009	0.948	1.8	86
HK-019	0.962 ± 0.073	0.966	-0.4	91
HK-021	0.559 ± 0.055	0.572	-2.3	80
HK-022	0.887 ± 0.080	0.862	2.9	68
SAV B6	0.677 ± 0.067	0.680	-0.4	66

## References

1) H. Dulaiova, K.W.W. Sims, M.A. Charette, J. Prytulak, J.S. Blusztajn "A new method for the determination of actinium-227 in geological samples," *J. Radioanal. Nucl. Chem.*, 296, 279-283 (2013).