eichrom® TECHNOLOGIES

A GCI COMPANY

npo eichrom

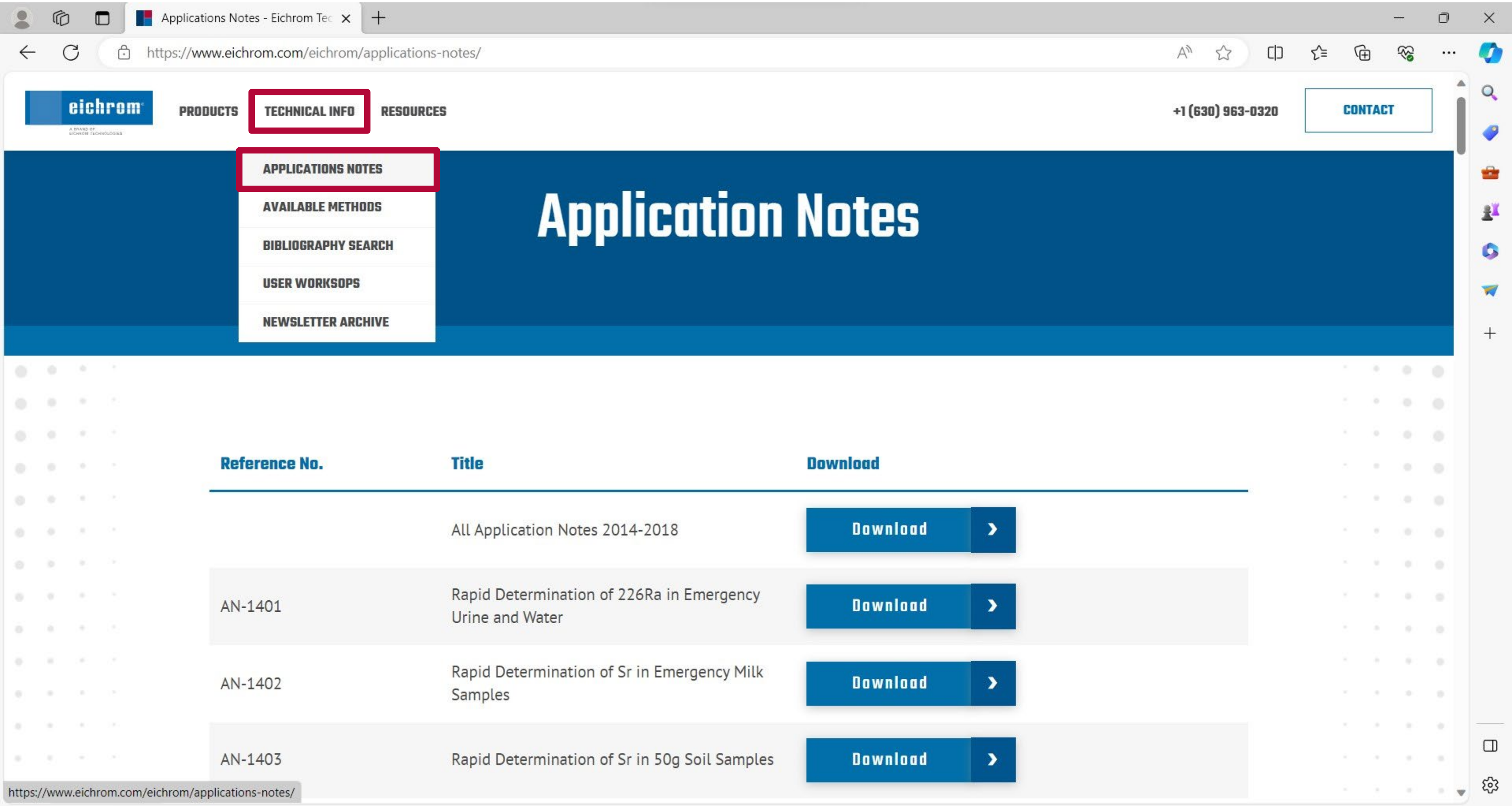
Resolve Filters: New Polyethylene Material

Madeleine Eddy

1 November 2023

RRMC

https://www.eichrom.com/eichrom/applications-notes



Common Recipes for CeF_3 Microprecipitates

Analyte	Matrix	15 mPre-treatment*	Ce(ug)	HF (mL)
Ac	10 mL 0.35 M HNO3	None	100	1.0
Ac	15 mL 2 M HCl	None	100	1.0
Am/Cm	**15 mL 4 M HCl**	**Dilute 2x**	**50**	**3.0**
Am/Cm	15 mL 0.25 M HCl	None	50	1.0
Am/Cm	20 mL 1 M HCl	None	50	1.0
Np/Pu	10 mL 0.1 M NH4 bioxalate	None	50	1.0
Np/Pu	20 mL 0.1 M HCl – 0.01 M HF – 0.01 M TiCl3	None	50	1.0
Pu	25 mL 0.05 M HNO3 – 0.05 M HF – 0.02 M TiCl3	None	50	1.0
Th	**15 mL 9 M HCl**	**Dilute to 40 mL**	**40**	**3.0**
U	15 mL 1 M HCl	None	100	1.0
U	10 mL 0.1 M NH4 bioxalate	None	100	1.0
Y	15 mL 8 M HNO3	Dilute 2x	100	3.0

CeF_3 Microprecipitate Method Overview

1) Dilute samples as necessary and add Ce Carrier (See Table I).

2) **U Samples**, Add 0.5mL 10% $TiCl_3$

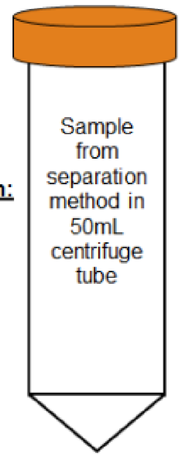
3) **Th, Np, Pu, Am/Cm samples requiring additional U decontamination:** Add 50 µL 30% H_2O_2.

4) Swirl to mix.

5) Add 1-3.5mL conc. HF (see Table I).

6) Swirl to mix.

7) Wait 20-30 minutes before filtration.



8) Ensure tight fit of filter assembly.

9) Engage vacuum.

10) Wet filter with 3-5mL 80% ethanol.

11) Wet filter with 3-5mL DI water.

12) Add sample.

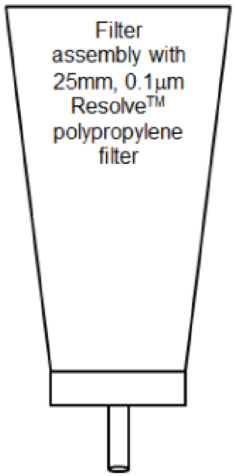
13) Rinse tube with 5mL DI water and add to filter assembly.

14) Allow all fluid to pass through filter.

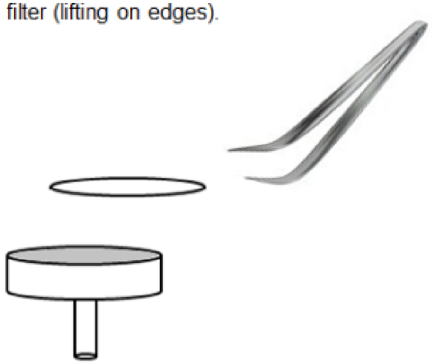
15) Rinse filter funnel with 3-5mL DI Water.

16) Rinse filter funnel with 2-3mL ethanol.

17) Filter until dry.



18) Remove filter (lifting on edges).



19) Mount filter to stainless steel planchet.

20) Place mounted filter into petri dish.

21) Dry under heat lamp.

22) Place lid on petri dish. Store until alpha measurement.

2022 Conclusions

- New Filter Material
  - Polyethylene is a viable replacement for Resolve® Filters
  - PE performance is equivalent to PP for standard actinide precipitate conditions
  - PE does experience slight curling
- Review of RE Precipitate Method
  - Precipitate quality is unaffected by different amounts of rare earth (25-200 ug) or HF (1-3 mL) added
  - Peroxide is detrimental to high HCl samples
  - Finishing with 100% ethanol reduces filter curling
  - Finishing/mounting/drying has little impact on spectra quality but does impact handling

Overview of Customer Comments/Questions

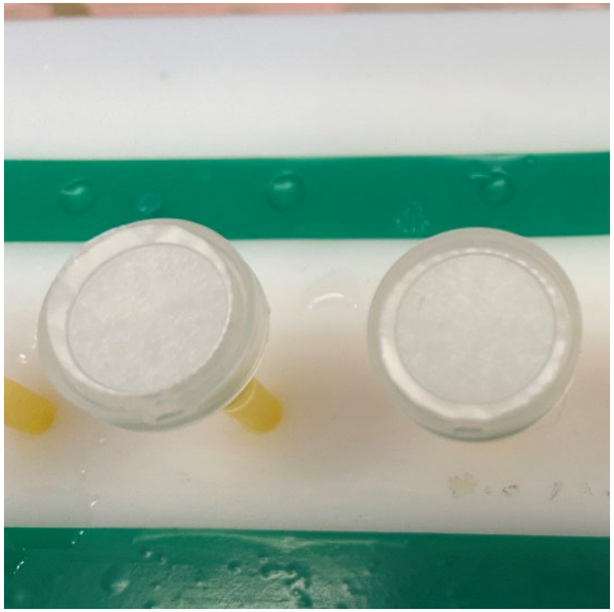
- Physical
  - Filters not centered on funnel
  - Oval-shaped filters
  - Wrinkling/ballooning of filters in funnel
- Chemical
  - High FWHM with tailing for Am/Cm samples
    - Specifically for TRU resin eluents
  - High FWHM for Th samples
- Kinetic
  - How long do precipitation reactions need?

2023 Experiments

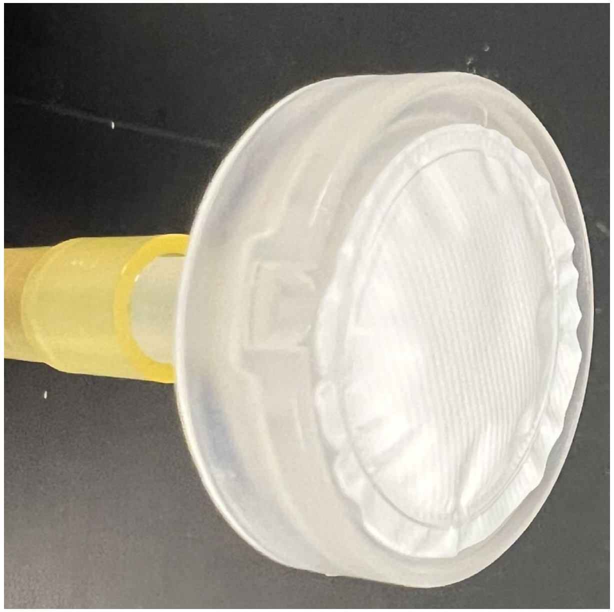
- Physical Handling
  - Wrinkled/off-center filter performance
  - Filter durability during long-term storage
- Aqueous Phase Chemistry
  - Clean acid vs. column eluent
  - Varied [acid], and acid neutralization
- Precipitation Kinetics
  - Filtration of Am/Cm samples at short time-points

Examples of Distorted Filters

Oval-shaped and wrinkled loose filters



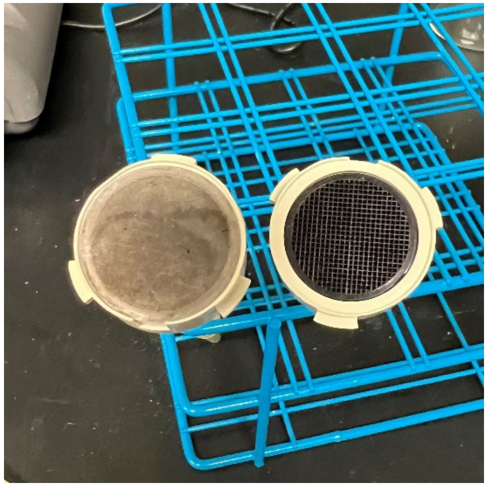
Filters placed off-center on funnels



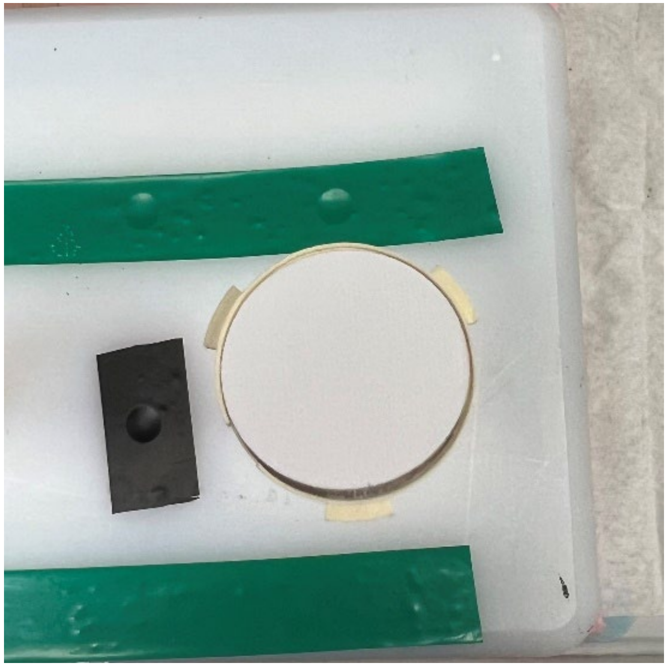
Ballooned filter removed from funnel

Resolve Funnels vs. Pall Gelman Funnels

Comparison of filter edge width



Pall Gelman filter with (L) and without (R) mesh insert

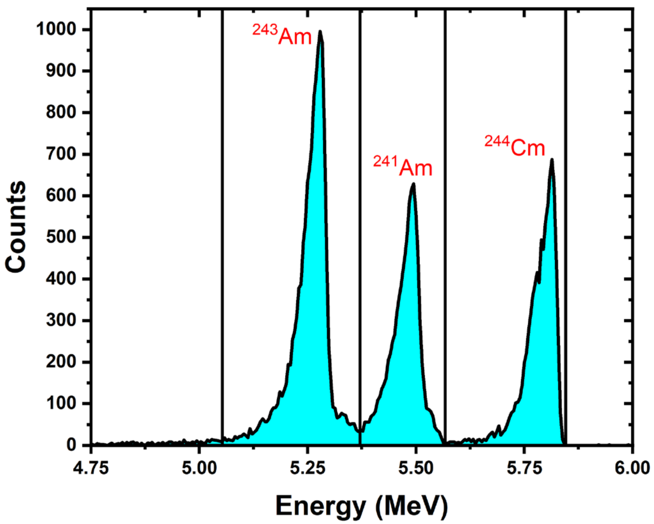


Oval-cut filter on Pall Gelman

Physical Handling "Worst Case Scenario" Tests

Experimental Conditions:

- 200 dpm ^{243}Am, 100 dpm ^{241}Am, and ^{244}Cm
- 10 mL 1 M HCl
- 50 ug Ce
- 1 mL conc. HF
- 20 min precipitation time



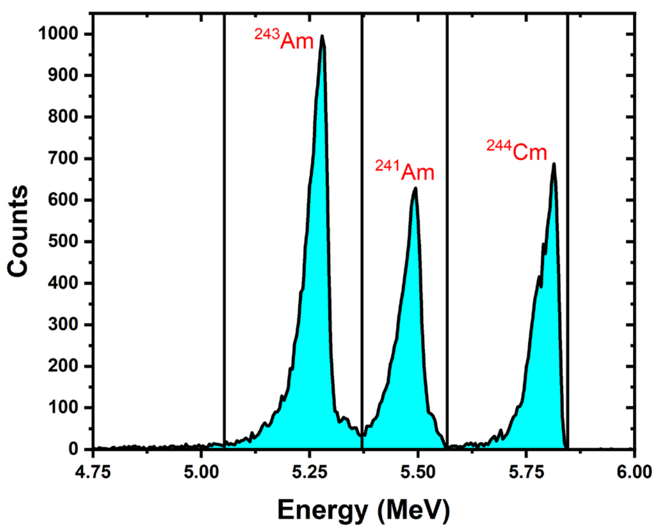
Relative Percent (%) recoveries of physically handled sample sets vs. control group (center-placed PP fliters)

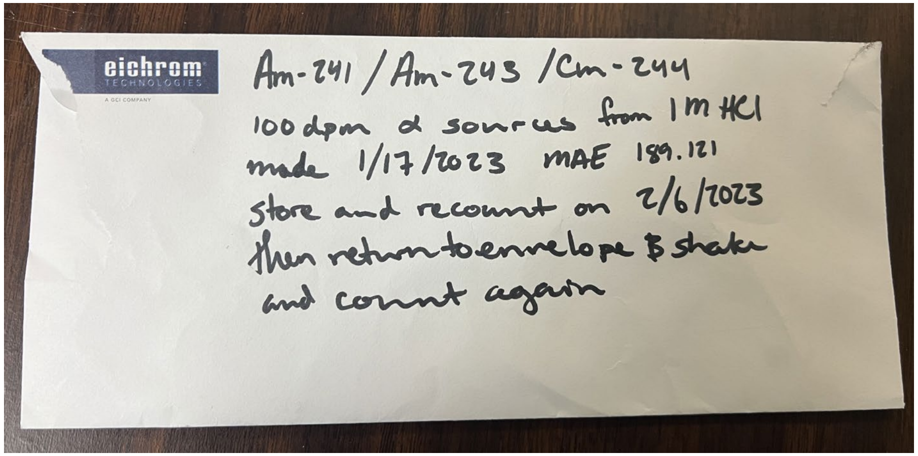
Sample Set	^{243}Am	^{241}Am	^{244}Cm
PE – off-center	120 ± 20	105 ± 4	102 ± 4
PP – dropped	105 ± 3	105 ± 2	104 ± 4
PE – dropped	104 ± 3	103 ± 2	102 ± 2
PP – Pall Gelman	100 ± 3	98 ± 2	100 ± 3
PE – Pall Gelman	102 ± 5	93 ± 4	95 ± 4
PE – Pall Gelman w/mesh	102 ± 2	98 ± 3	100 ± 2

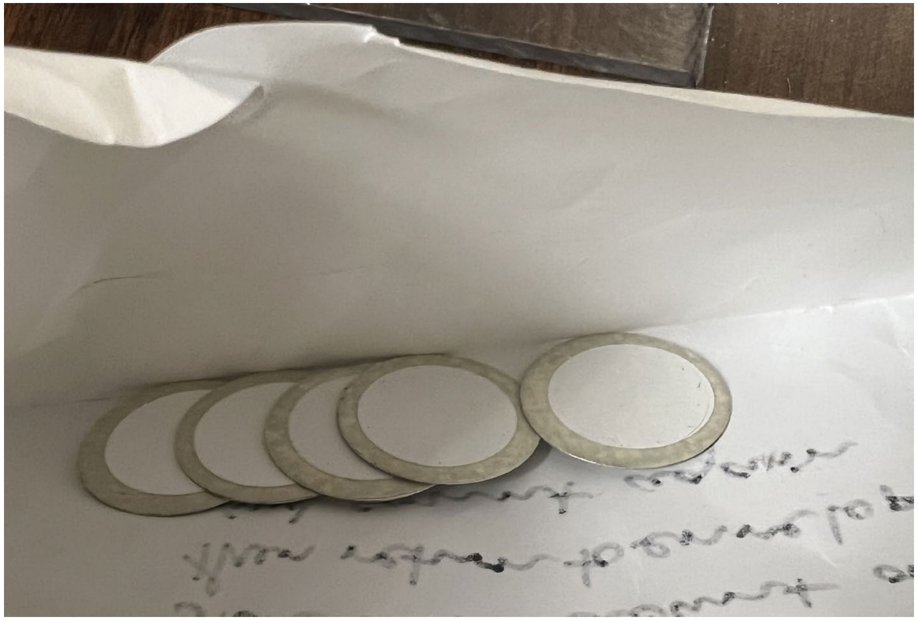
Durability/Storage Testing

Experimental Conditions:

- 200 dpm ^{243}Am, 100 dpm ^{241}Am, and ^{244}Cm
- 10 mL 1 M HCl
- 50 ug Ce
- 1 mL conc. HF
- 20 min precipitation time







Durability/Storage Testing

Percent Change in Activity for PP Samples

Sample	^{243}Am	^{241}Am	^{244}Cm
Initial Activity (dpm)	348	144	138
Two Weeks	+0.9%	-0.2%	-0.4%
Two Weeks – shaken	-3.9%	-2.7%	-4.0%
Three Months	-5.3%	-5.8%	-6.9%
Six Months	-5.2%	-5.9%	-6.1%
Eight Months	-1.0%	-1.9%	-2.2%

Percent Change in Activity for PE Samples

Sample	^{243}Am	^{241}Am	^{244}Cm
Initial Activity (dpm)	242	141	137
Two Weeks	+0.7%	-1.2%	-0.7%
Two Weeks – shaken	+0.0%	-0.7%	-1.4%
Three Months	+0.5%	-2.7%	-2.9%
Six Months	-1.1%	-2.7%	-2.3%
Eight Months	-1.0%	-2.7%	-2.3%

Durability/Storage Testing

Percent Change in FWHM for PP Samples

Sample	^{243}Am	^{241}Am	^{244}Cm
Initial FWHM (keV)	37.9	39.7	29.9
Two Weeks	+5.4%	+3.4%	+16%
Two Weeks – shaken	+4.8%	+3.0%	+13%
Three Months	+38%	+34%	+82%
Six Months	+16%	+11%	+55%
Eight Months	+16%	+12%	+50%

Percent Change in FWHM for PE Samples

Sample	^{243}Am	^{241}Am	^{244}Cm
Initial FWHM (keV)	39.4	41.1	34.9
Two Weeks	+6.9%	+8.2%	+17%
Two Weeks – shaken	+6.0%	+6.1%	+9.5%
Three Months	+32%	+33%	+67%
Six Months	+14%	+16%	+48%
Eight Months	+15%	+13%	+50%

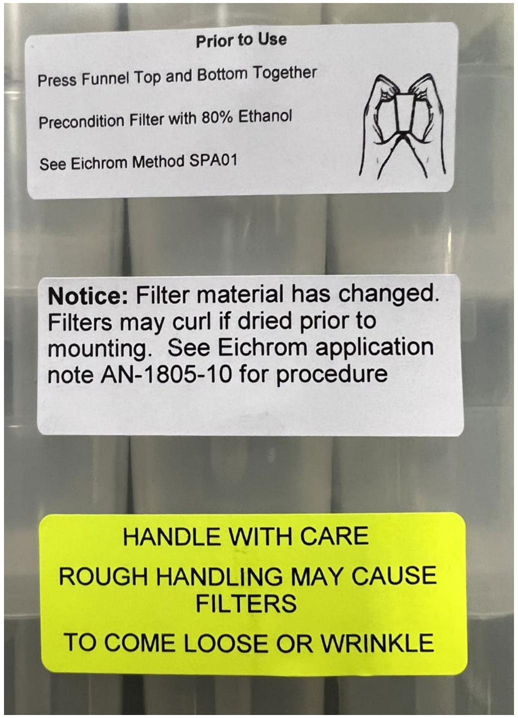
Physical Handling – Conclusions and Process Changes

Conclusions

- Minimal change in % recovery unless gaps in filter
  - – Can identify gaps easily during wetting by "whooshing" of EtOH
- Pall Gelman funnels larger active area – more important to fully center filters
- Minimal activity loss but significant increase in FWHM with long-term storage

Process Changes

- Warning labels
- Less compression to funnel stacks
- Adjust dye cutting pressure/method to reduce jagged edges

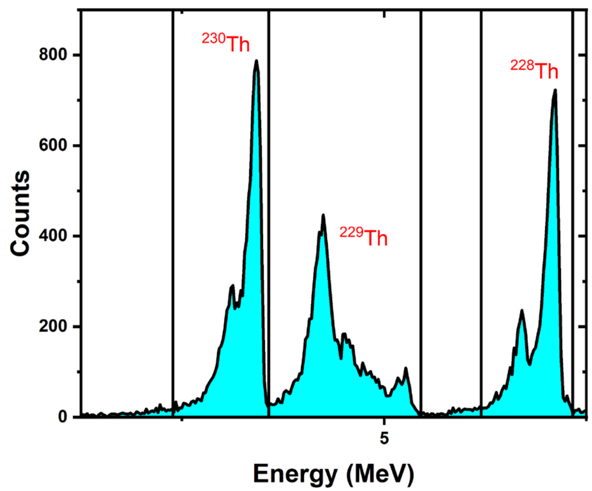


Clean Acid vs. Column Eluent

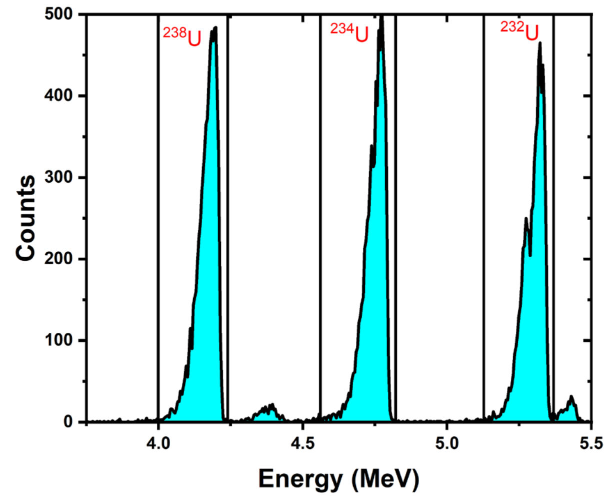
TEVA	UTEVA	TRU	DGA
PC/L/R 30 mL 3 M HNO_3	PC/L/R 30 mL 3 M HNO_3	PC/L/R 30 mL 3 M HNO_3	PC/L/R 30 mL 3 M HNO_3
Th – 15 mL 9 M HCl	Rinse – 20 mL 5 M HCl/0.05 M oxalic acid	**Am – 15 mL 4 M HCl**	Rinse – 20 mL 0.25 M HNO_3
Pu/Np – 15 mL 0.1 M HCl/0.05 M HF/0.01 M $TiCl_3$	**U – 15 mL 1 M HCl**	Rinse – 4 M HCl/0.25 M HF	**Am – 0.1 M HCl**
		U or Pu/Np – 15 mL 0.1 M ammonium bioxalate	

Clean Acid vs. Column Eluent

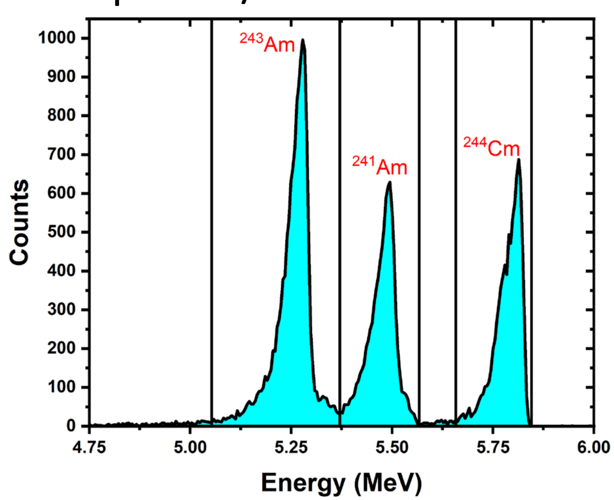
50 dpm Th: TEVA



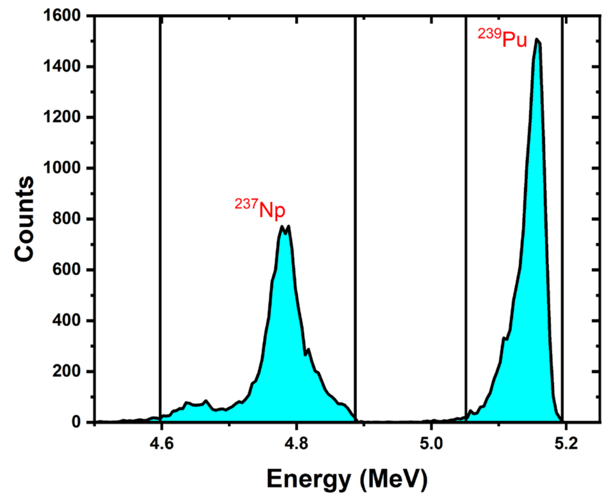
20 dpm U: UTEVA and TRU



100 dpmAm/Cm: TRU and DGA



100 dpmNp/Pu: TEVA and TRU



Clean Acid vs. Column Eluent

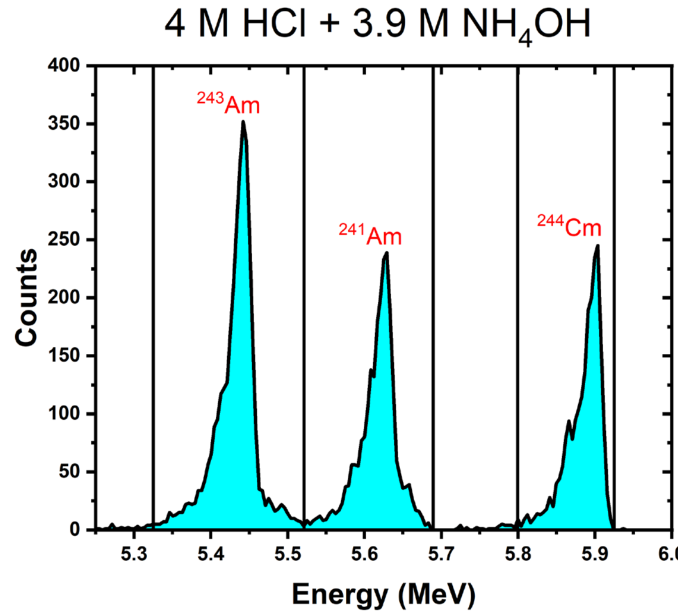
TEVA		UTEVA		TRU		DGA	
Th: 15 mL 9 M HCl diluted to 40 mL		**U:** 15 mL 1 M HCl		**Am:** 15 mL 4 M HCl diluted to 30 mL		**Am:** 15 mL 0.1 M HCl	
dpm -6.5%	*FWHM +4.1%*	*dpm -0.4%*	*FWHM -8.5%*	*dpm -25%*	*FWHM -2.2%*	*dpm -33%*	*FWHM -5.1%*
Pu/Np: 15 mL 0.1 M HCl/0.05 M HF/ 0.01 M $TiCl_3$				**U:** 15 mL 0.1 M ammonium bioxalate			
dpm -8.4%	*FWHM -2.8%*			*dpm -4.7%*	*FWHM -11%*		
				Pu/Np: 15 mL 0.1 M ammonium bioxalate			
				dpm +3.1%	*FWHM +12%*		

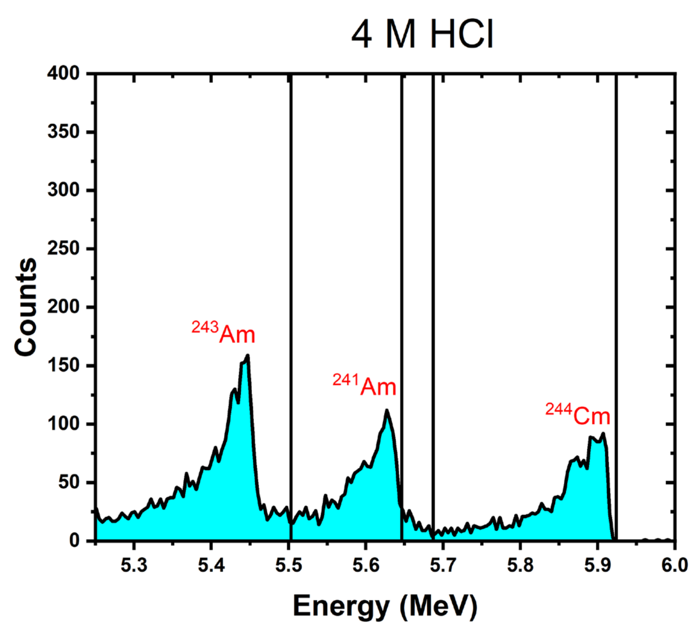
Am/Cm Acid Dependence

Experimental Conditions:

- 20 dpm ^{243}Am, 10 dpm ^{241}Am, and ^{244}Cm
- 50 ug Ce
- 1 mL conc. HF
- 20 min precipitation time

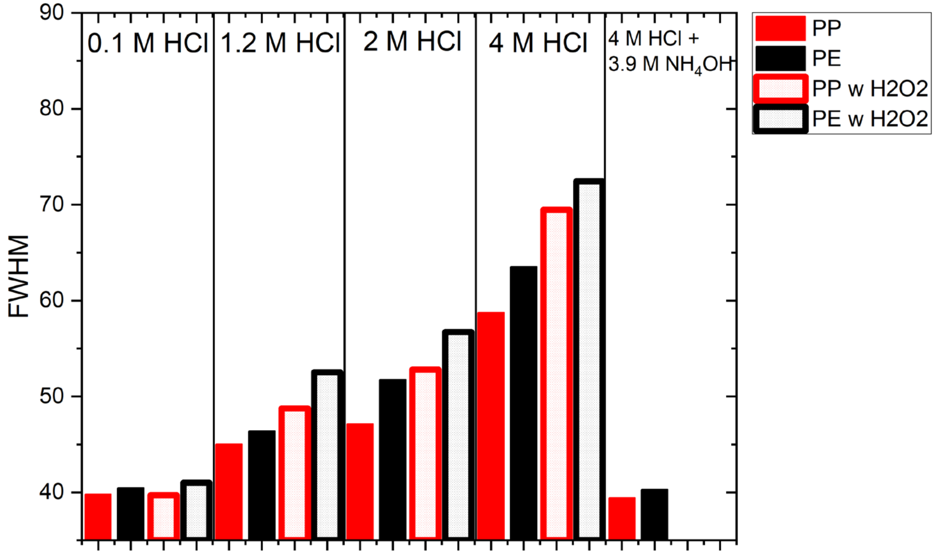
Volume (mL)	[HCl] (M)
15	0.1
50	1.2
30	2.0
15	4.0
15 + 7.6 mL conc NH_4OH	4.0 + 3.9 M NH_4OH



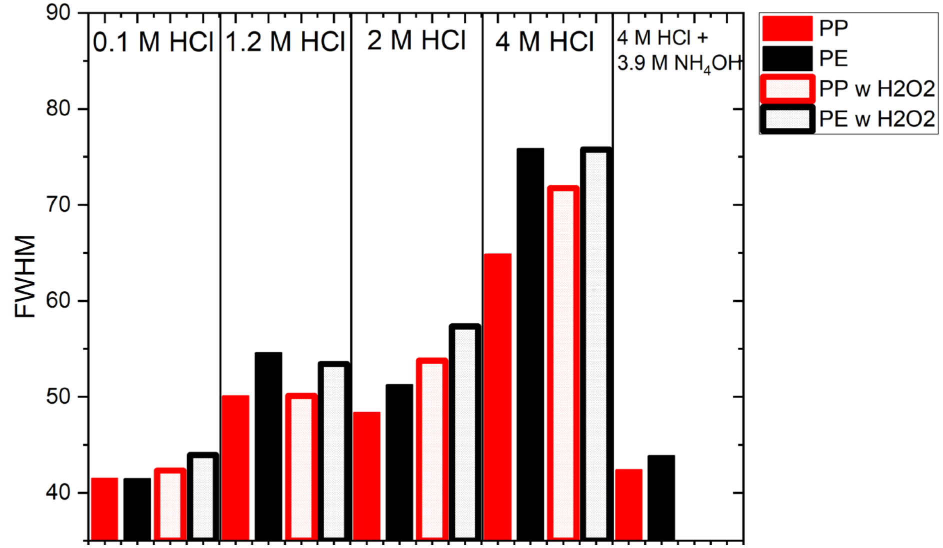


Am/Cm Acid Dependence

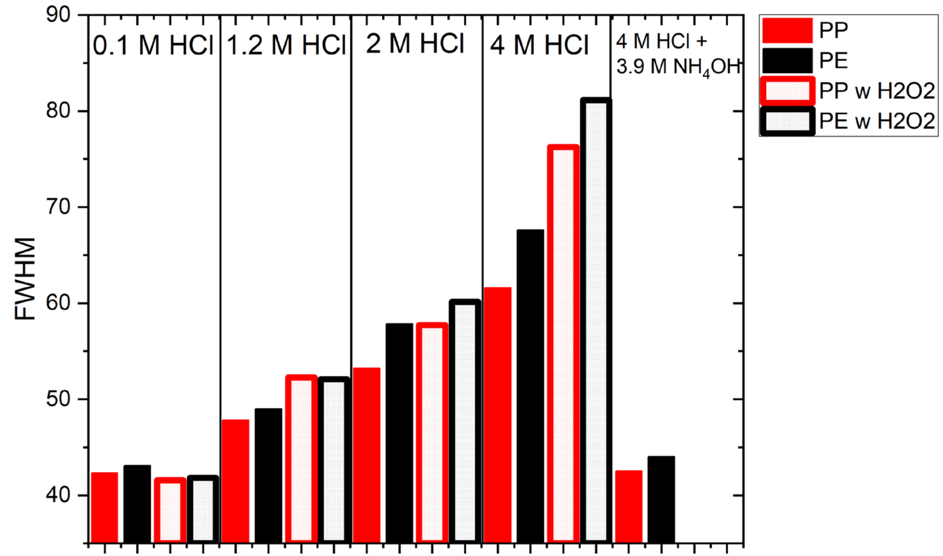
Am-243 FWHM for PP v PE with varied [HCl]



Am-241 FWHM for PP v PE with varied [HCl]



Cm-244 FWHM for PP v PE with varied [HCl]

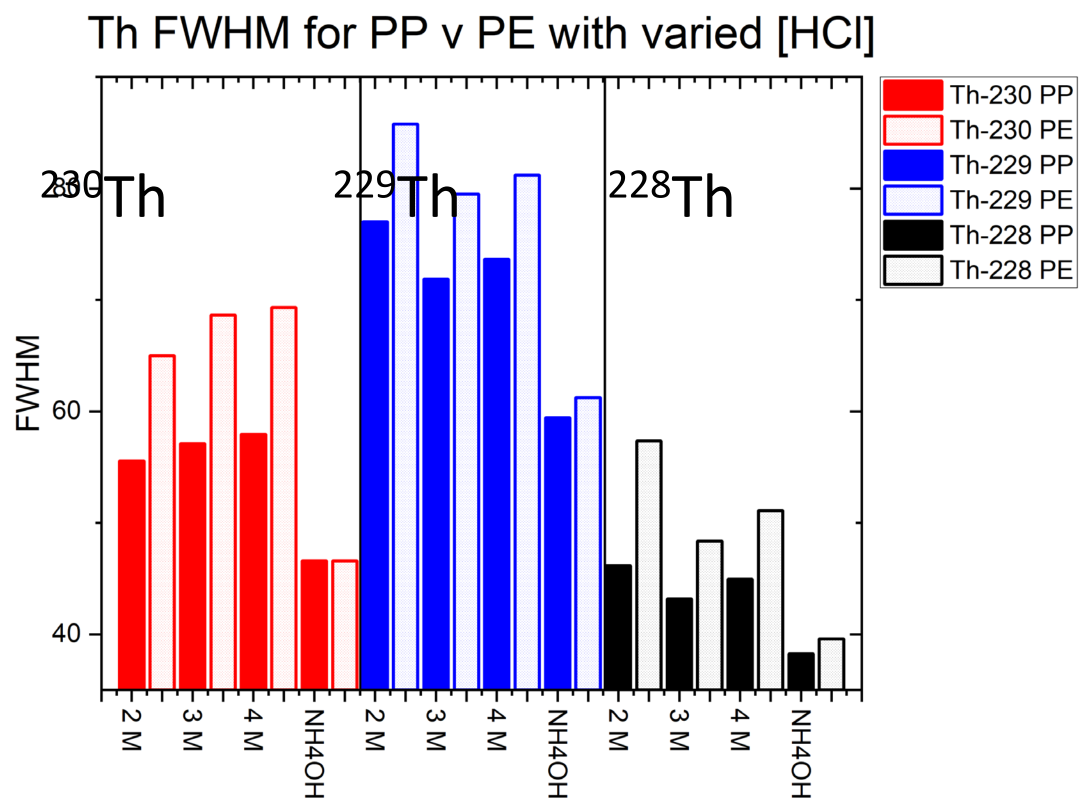


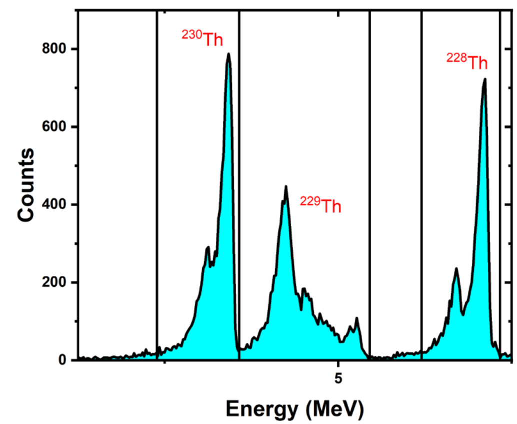
Th Acid Dependence

Experimental Conditions:

- 200 dpm ^{230}Th ^{229}Th, and ^{228}Th
- 40 ug Ce
- 3 mL conc. HF
- 20 min precipitation time

Volume (mL)	[HCl] (M)
30	2
45	3
15	4
15 + 16.9 mL conc NH_4OH	9.0 + 8.9 M NH_4OH





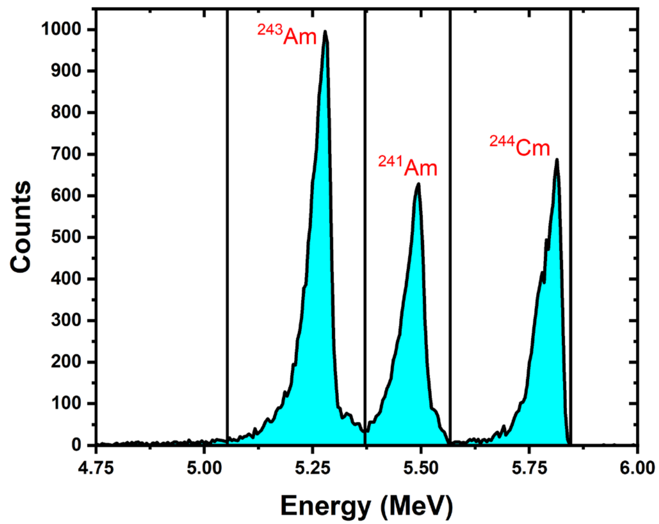
Aqueous Phase Chemistry Conclusions

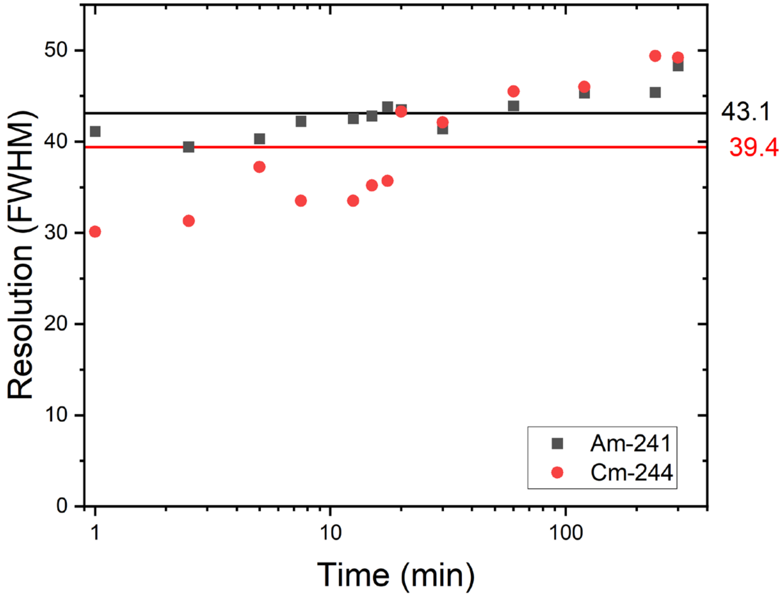
- Acid vs eluent
  - No significant difference in yield for acid vs eluent
  - Some increase in FWHM for Np/Pu-TRU sample
- Am/Cm vs [HCl]
  - FWHM PE > FWHM PP
  - Significant increase in FWHM with increasing [HCl]
  - Neutralizing HCl restores original spectral quality
- Th vs [HCl]
  - FWHM PE > FWHM PP
  - No significant dependence on [HCl] but PE > PE for all conditions
  - Neutralizing HCl greatly improves spectral quality

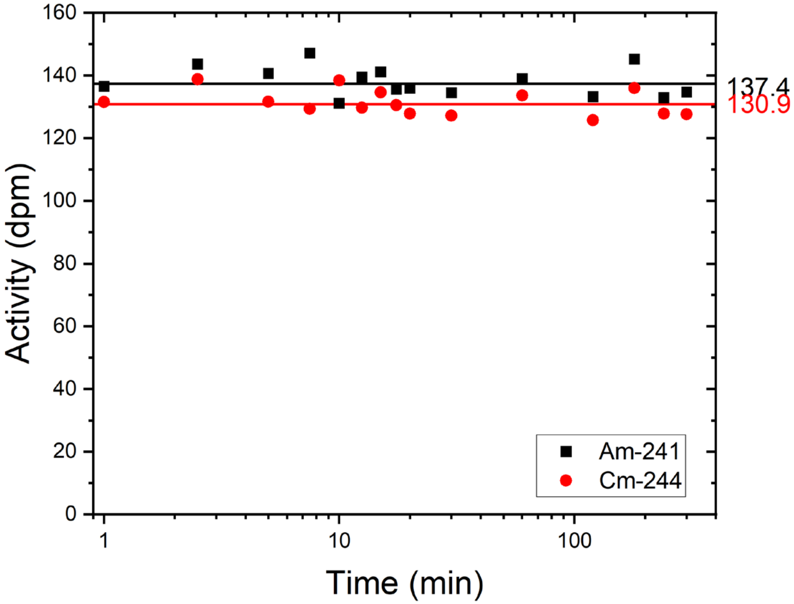
Short-term Delayed Filtration Test for Am/Cm

Experimental Conditions:

- 200 dpm ^{243}Am, 100 dpm ^{241}Am, and ^{244}Cm
- 10 mL 1 M HCl
- 50 ug Ce
- 1 mL conc. HF
- **VARIED** precipitation time







eichrom TECHNOLOGIES
A GCI COMPANY
npo eichrom

Current Work: Investigation of Thicker PE Membrane

- ## Motivation
  - Determine if thicker filters are viable alternative to new PE materials
  - Hope that the thicker membrane may have fewer physical issues related to curling and shifting in funnels
- ## Experiments
  - Perform standard QC to determine product quality
  - Test filter curling
  - Test Am/Cm sample acid dependence
- ## Long-term Objectives
  - If we proceed with this new material, we will monitor customer comments related to filters shifting and ballooning in funnels to determine if we see a decrease in frequency. We hope the thicker material will be sturdier and create a tighter fit in the funnels which will prevent it from slipping during shipping and handling.

Questions?

For more information on alpha spectrometry please join Eichrom at ORTEC's Alpha Spectrometry Training Course from October 14-18, 2024, at GEL Laboratories in Charleston, SC

https://www.ortec-online.com/service-and-support/training/alpha-spectrometry

eichrom® TECHNOLOGIES
A GCI COMPANY

npo eichrom

ORTEC® AMETEK®

GEL Laboratories

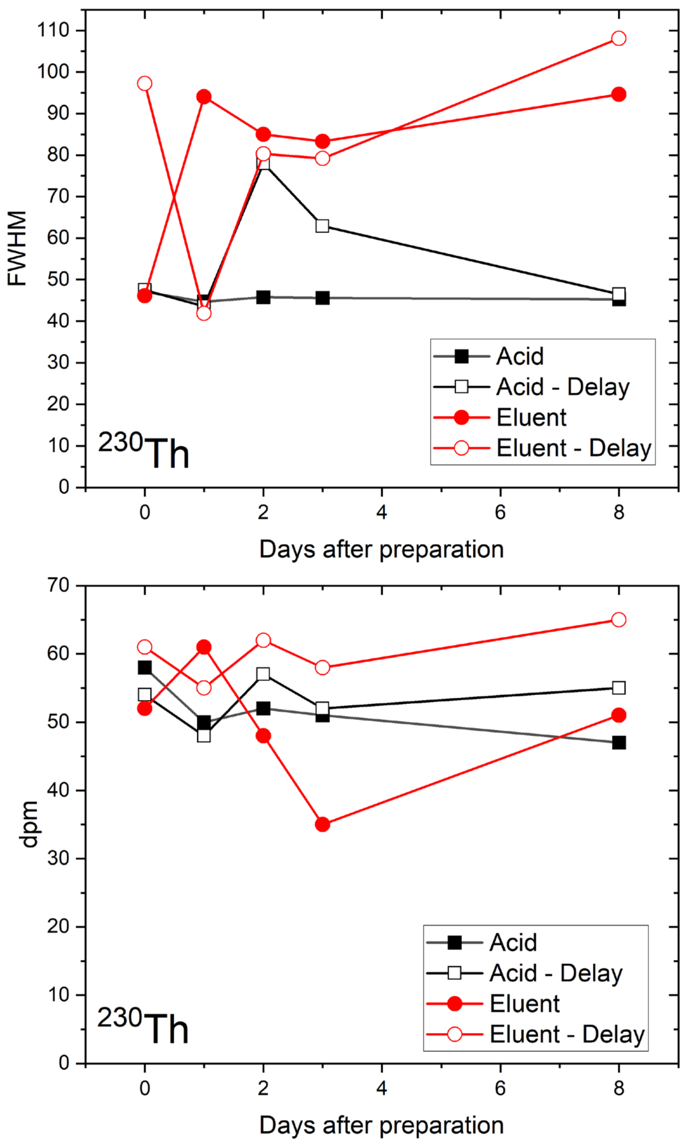
Acid vs Elution Multi-day Test

- Conduct mock columns to collect “eluent” samples
- Prepare 10x samples for each acid and eluent solution with tracers
- Add Ce and HF to 5x samples
- Delay precipitation for other 5x samples
- Each day prepare one delayed filtration and delayed precipitation sample for each solution
- Monitor how DPM and FWHM are affected over time

TEVA	UTEVA	TRU	DGA
PC/L/R 30 mL 3 M HNO_3	PC/L/R 30 mL 3 M HNO_3	PC/L/R 30 mL 3 M HNO_3	PC/L/R 30 mL 3 M HNO_3
Th – 15 mL 9 M HCl	Rinse – 20 mL 5 M HCl/0.05 M oxalic acid	**Am – 15 mL 4 M HCl**	Rinse – 20 mL 0.25 M HNO_3
Pu/Np – 15 mL 0.1 M HCl/0.05 M HF/0.01 M $TiCl_3$	**U – 15 mL 1 M HCl**	Rinse – 4 M HCl/0.25 M HF	**Am – 0.1 M HCl**
		U or Pu/Np – 15 mL 0.1 M ammonium bioxalate	

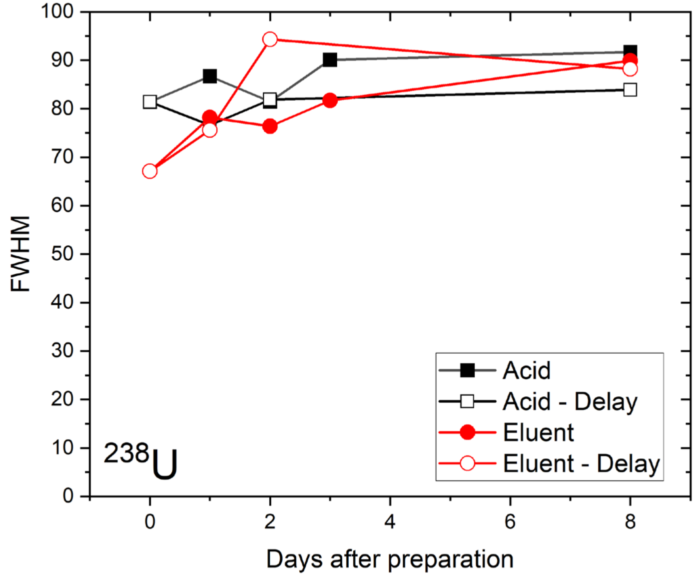
Thorium

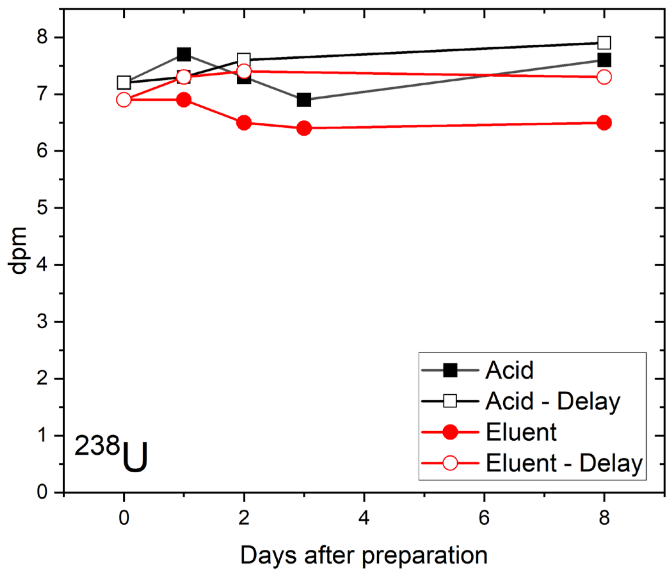
TEVA



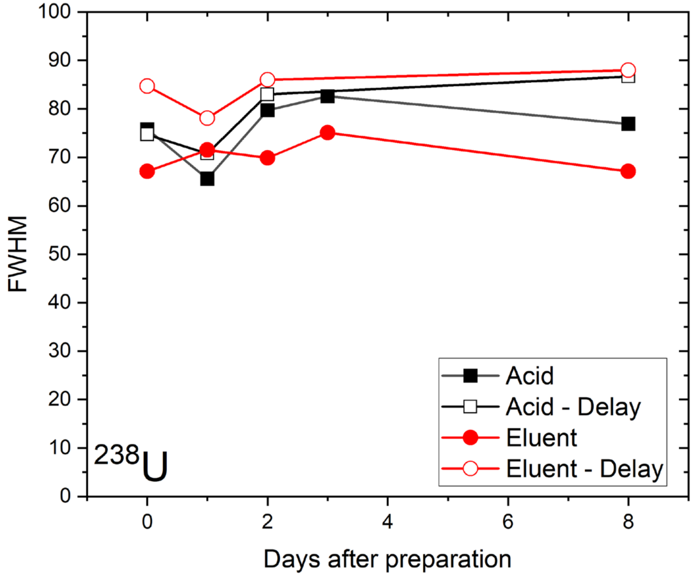
Uranium

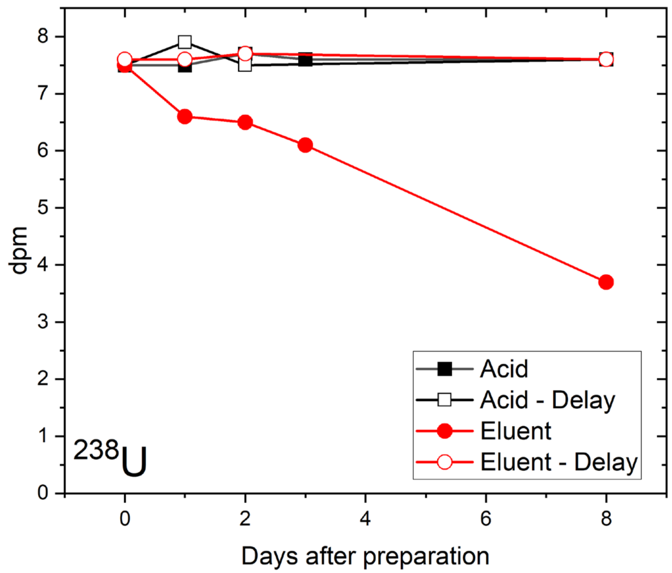
TRU





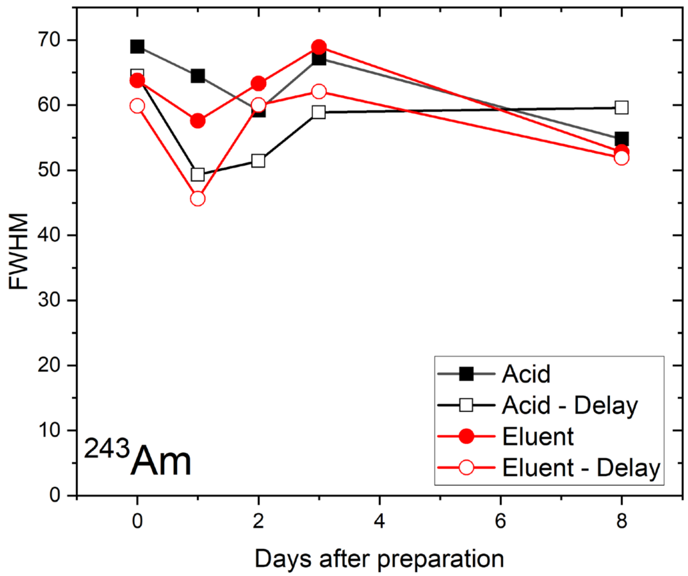
UTEVA

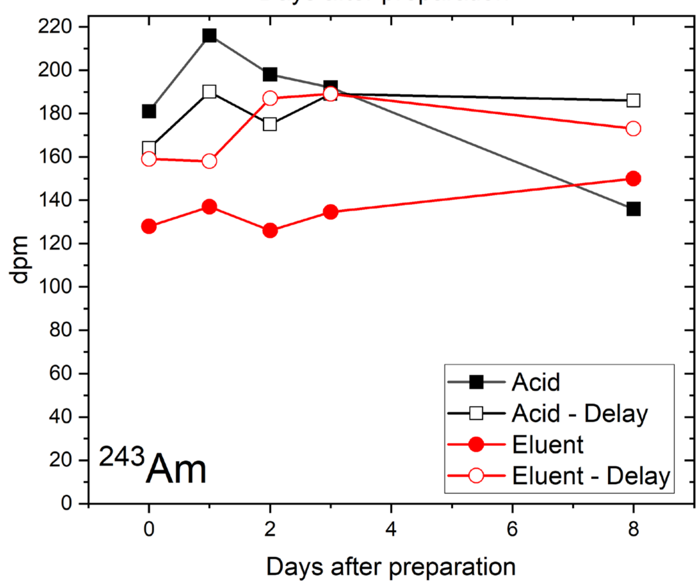




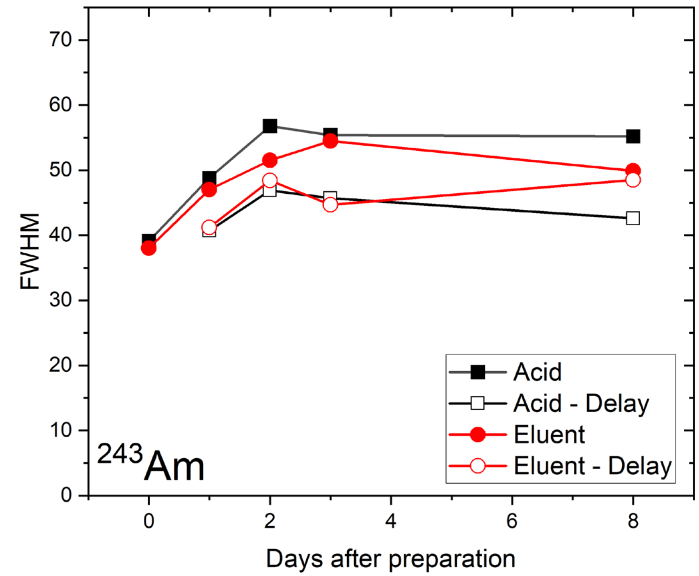
Americium

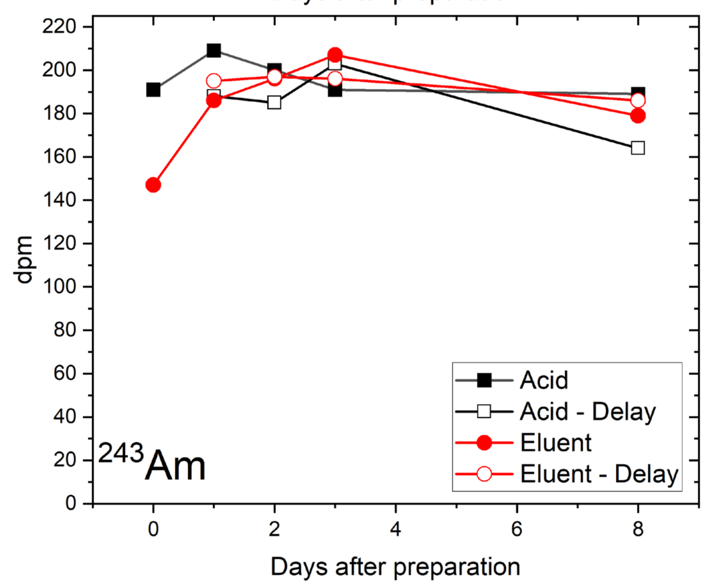
TRU





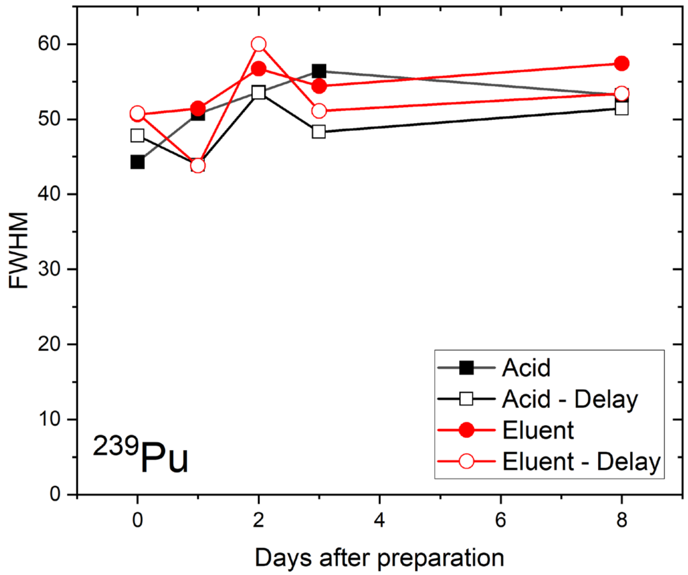
DGA

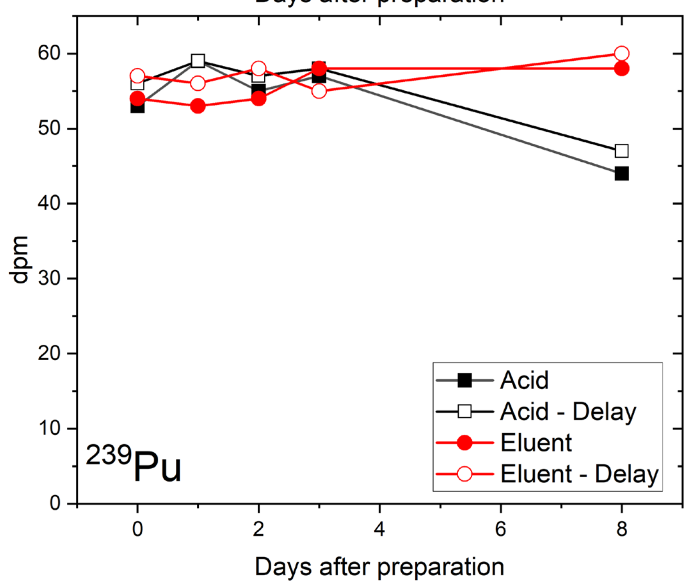




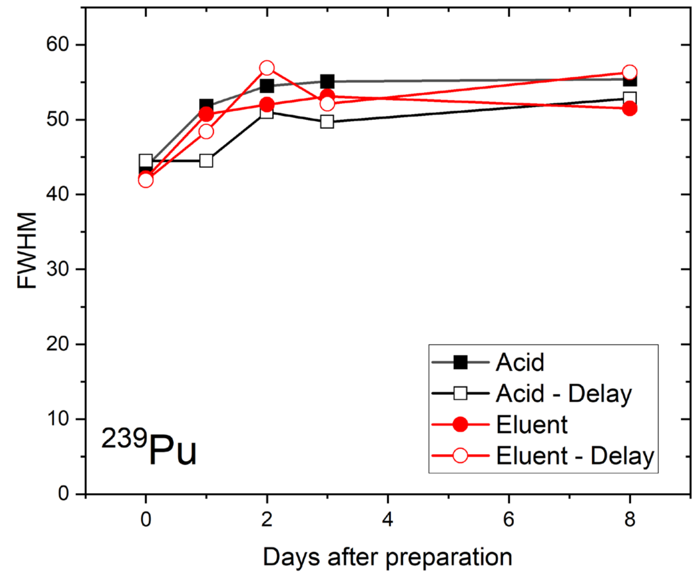
Neptunium/Plutonium

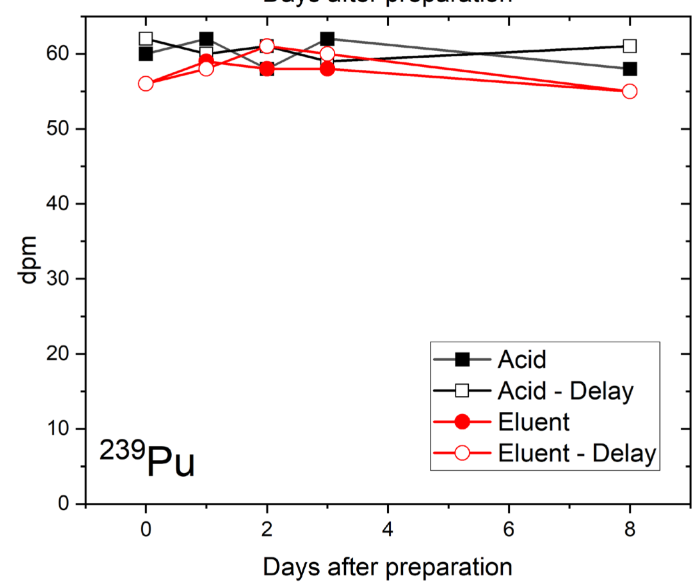
TRU





TEVA

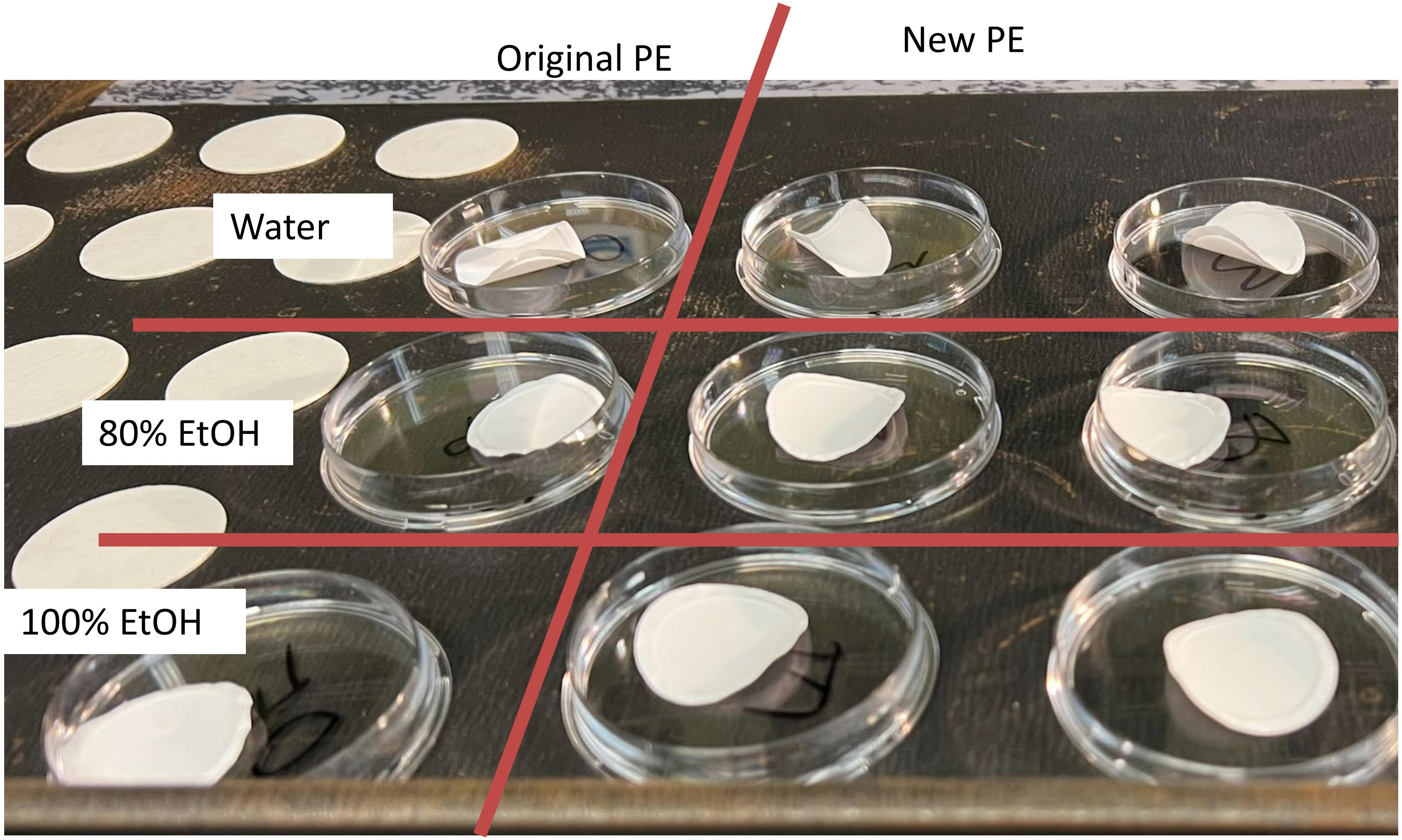




Kinetics Conclusions

- Short-term
  - Activity unaffected, but slight increase in FWHM over time
- Multi-day
  - No relationship between FWHM and day of filtration/precipitation
  - Lower FWHM for Th-TEVA acid samples
  - Decrease in activity/yield for U-UTEVA eluent sample over time
  - Am-DGA samples lower FWHM than Am-TRU
    - Related to [HCl]

Current Work: Investigation of Thicker PE Membrane



Investigations of Thicker PE Membrane