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A BRAND OF
EICHROM TECHNOLOGIES



Resolve® Filters

Madeleine Eddy, PhD.
65th RRMC, Atlanta GA
1 November 2022

Outline

1. New Filter Material

- Identification of alternative membrane
- Analysis of new material performance

2. Review of RE Precipitate Method

- Identification of key parameters for sample prep
- Recommendations for sample drying

3. Conclusions and Future Work

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- Identification of alternative membrane
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2. Review of RE Precipitate Method

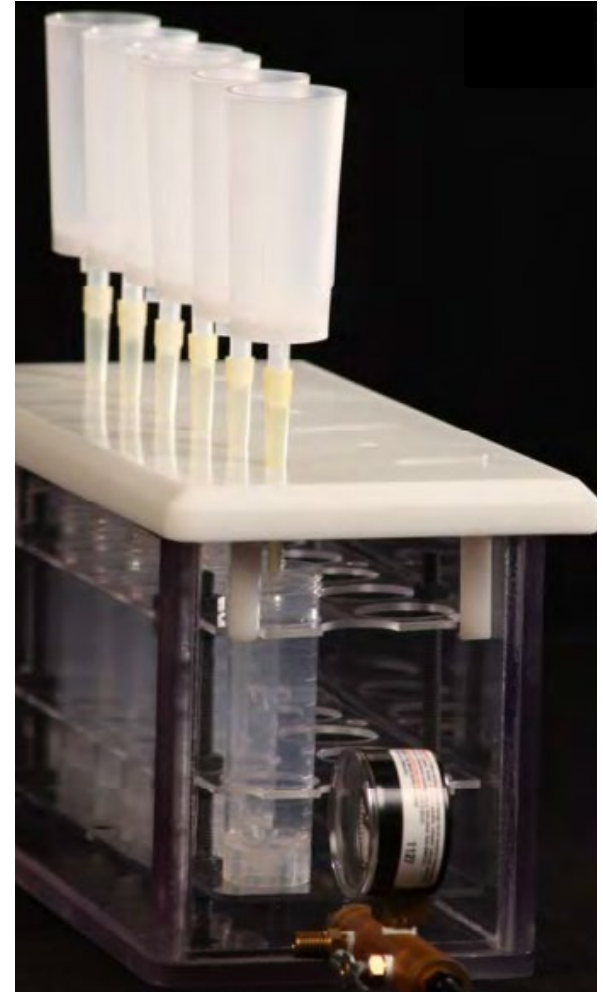
- Identification of key parameters for sample prep
- Recommendations for sample drying

3. Conclusions and Future Work

Identifying an Alternative Material

Criteria:

- Physically similar to polypropylene
- Uniform material quality
- Consistent Supply
- Maintain high spectral resolution
- Compatible with existing methods

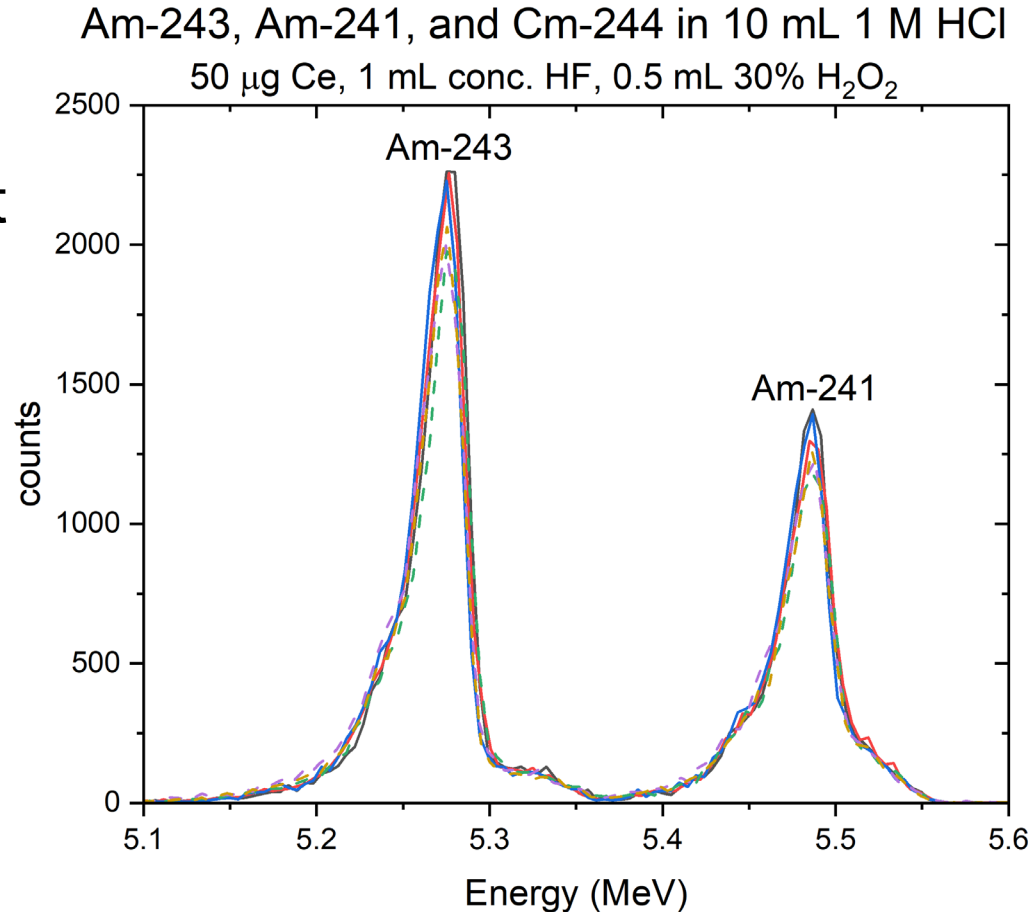


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Phase One

- Identified nine different materials/sources of interest
- Performed standard QC procedure to determine if the materials work with our methods
- Used QC test to identify physical differences between lots



Phase One Results

Material	Pore Size (µm)	Am-241 FWHM	Am-243 FWHM	Both Sides?	Curling?
Current PP	0.1	38.6	35.3	Yes	No
PES	0.1	40.2	37.6	Yes	Severe
PES	0.1	40.8	36.4	Yes	Severe
PP	0.22	non-resolution	non-resolution	-	-
PP	0.45	non-resolution	non-resolution	-	-
PES	0.1	36.6	40.1	Yes	Yes
PP w/PTFE laminated top	0.1	poor recoveries and spotty resolution	poor recoveries and spotty resolution	-	-
PP	0.1	clogged filter	clogged filter	-	-
PE	0.1	40	38	Yes	Minor
PE	0.2	45	42	Yes	Minor



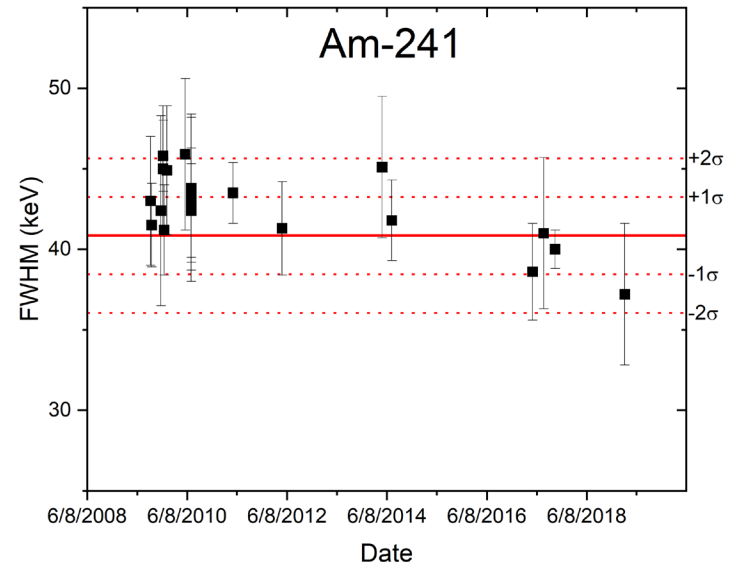
Phase Two

- Purchase full lot of top candidate from Phase One
- Sample all over membrane roll to determine uniformity

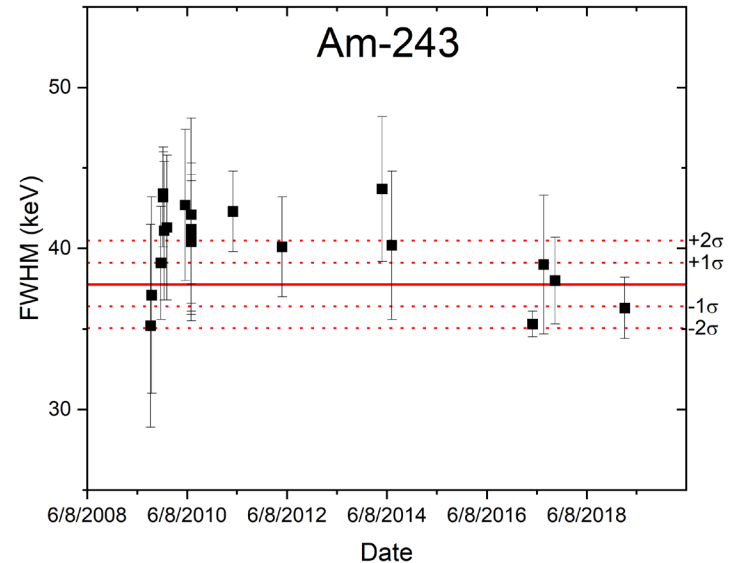


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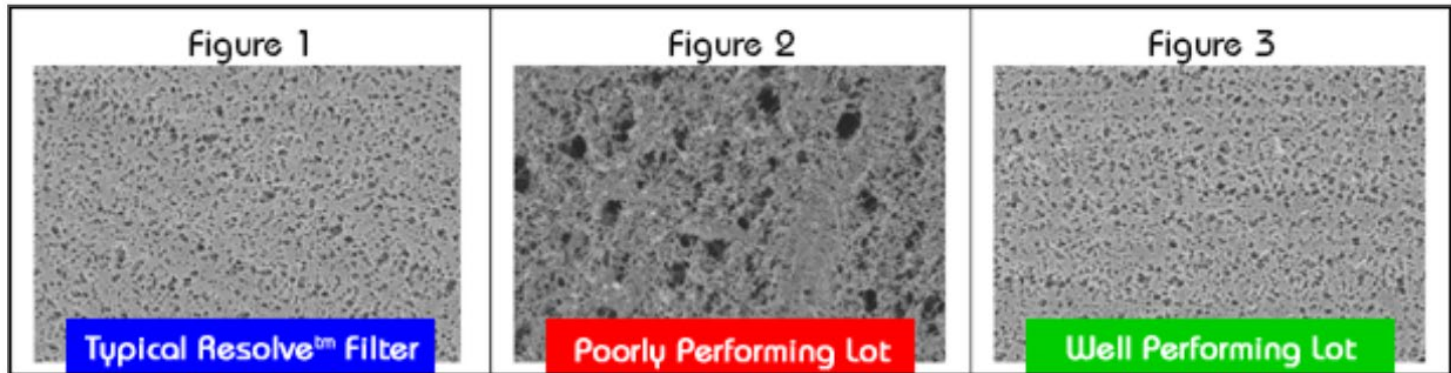
Comparison of PE material to historic PP lots



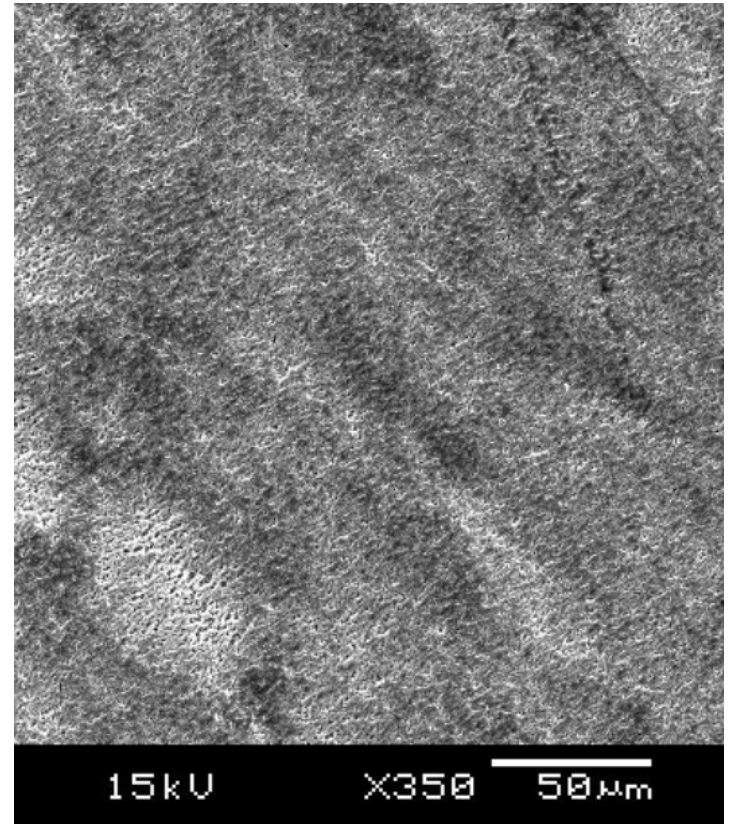
Comparison of PE material to historic PP lots



Phase Three - SEM



- Alpha sources prone to self-attenuation
- Smooth, even precipitate required for high quality spectra
- Defects in filter surface would lead to poor resolution



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Phase Three – PP vs. PE Testing

Element	Elution Solution	$\mu\text{g Ln carrier}$	mL conc. HF	Redox Agent*
Th	15 mL 9 M HCl Dilute to 40 mL	40	3	N/A
U	15 mL 1 M HCl	100	1	TiCl ₃
Np/Pu	20 mL 0.1 M HCl + 0.05 M HF + 0.01 M TiCl ₃	50	1	0.5 mL 30% H ₂ O ₂
Am/Cm	10 mL 1.0 M HCl	50	1	0.5 mL 30% H ₂ O ₂



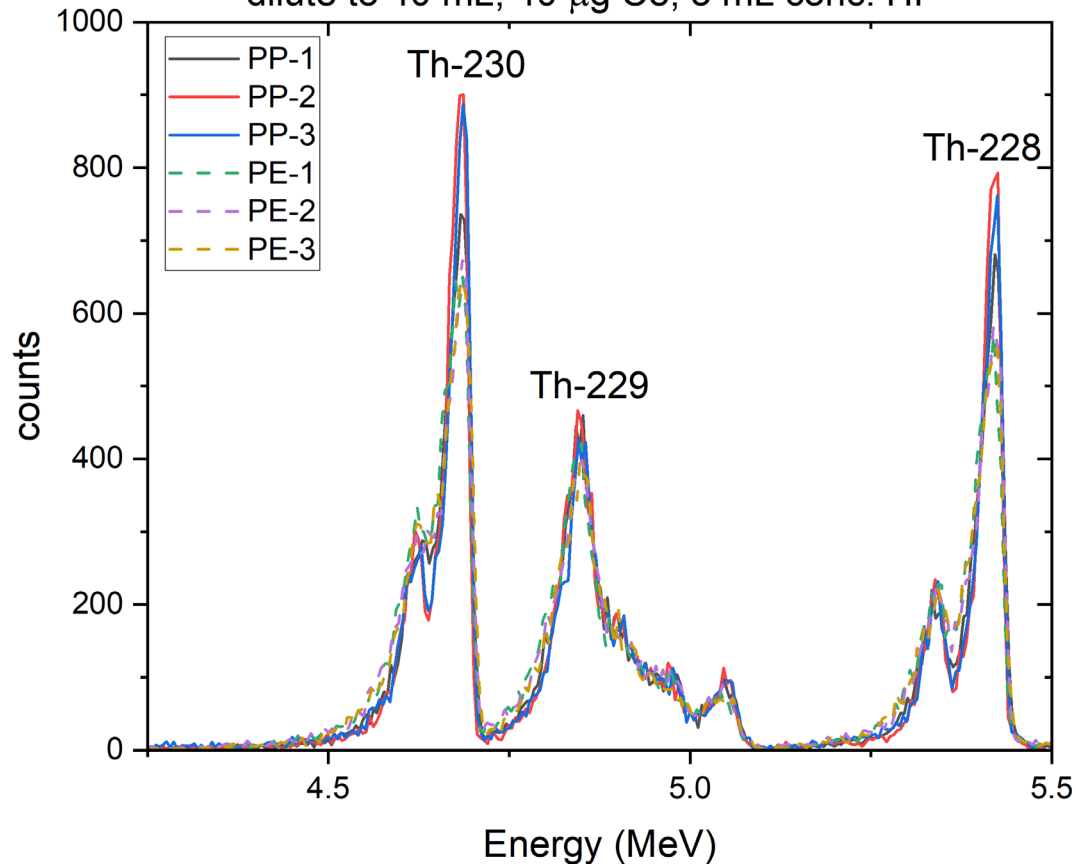
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*Redox Agents used because method only applies to +3/+4 actinides

Standard Th Sample

Th-230, Th-229, and Th-228 in 15 mL 9 M HCl
dilute to 40 mL, 40 µg Ce, 3 mL conc. HF

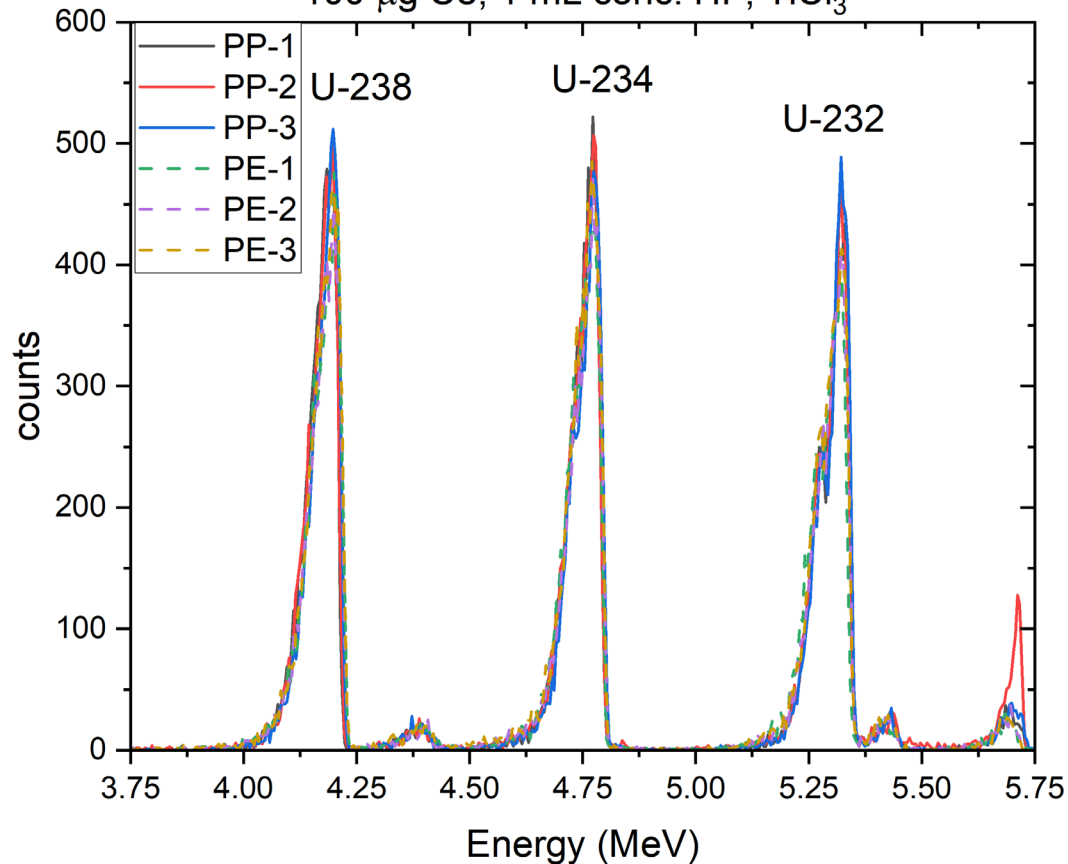


	Th-230	Th-229	Th-228
FWHM	+18.39%	+9.81%	+21.27%
Activity	+1.51%	+0.54%	+1.97%

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Standard U Sample

U-238, U-234, and U-232 in 15 mL 1 M HCl
100 µg Ce, 1 mL conc. HF, TiCl₃



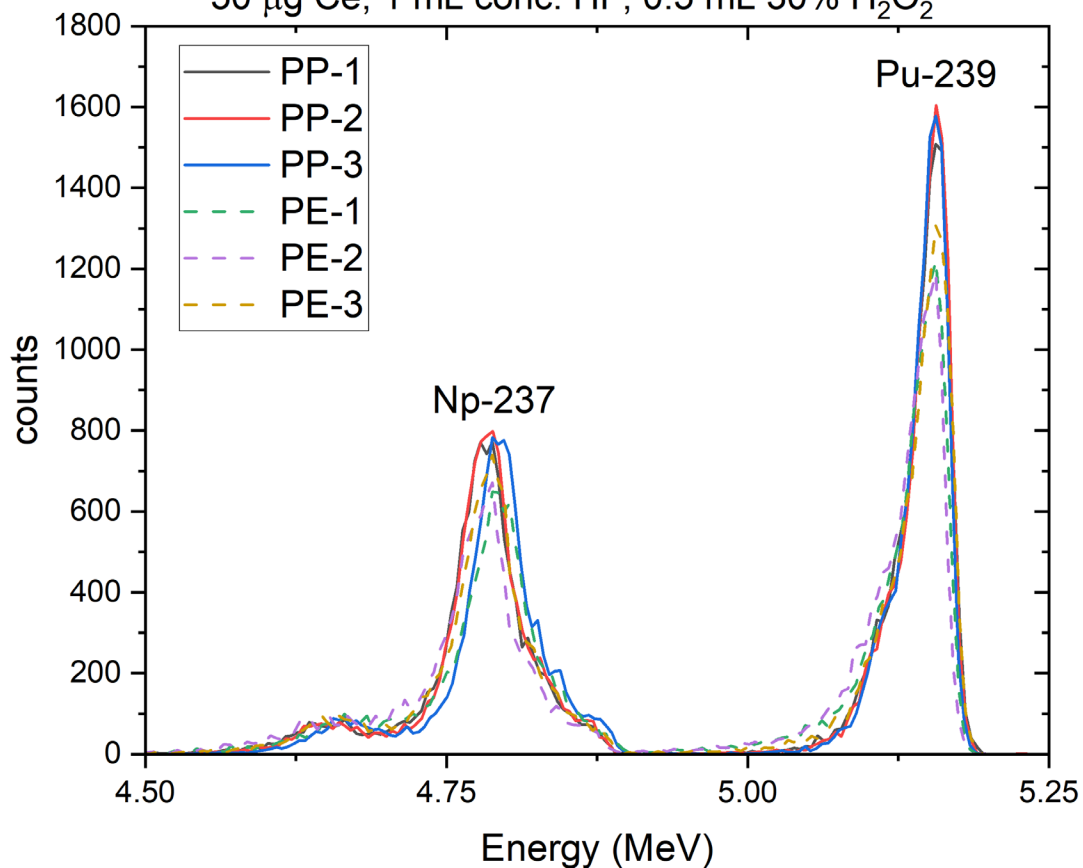
	U-238	U-234	U-232
FWHM	-6.47%	+4.51%	-1.22%
Activity	+0.09%	+0.28%	-0.39%

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Standard Pu/Np Sample

Np-237 and Pu-239 in 20 mL 0.1 M HCl + 0.05 M HF + 0.01 M TiCl₃

50 µg Ce, 1 mL conc. HF, 0.5 mL 30% H₂O₂



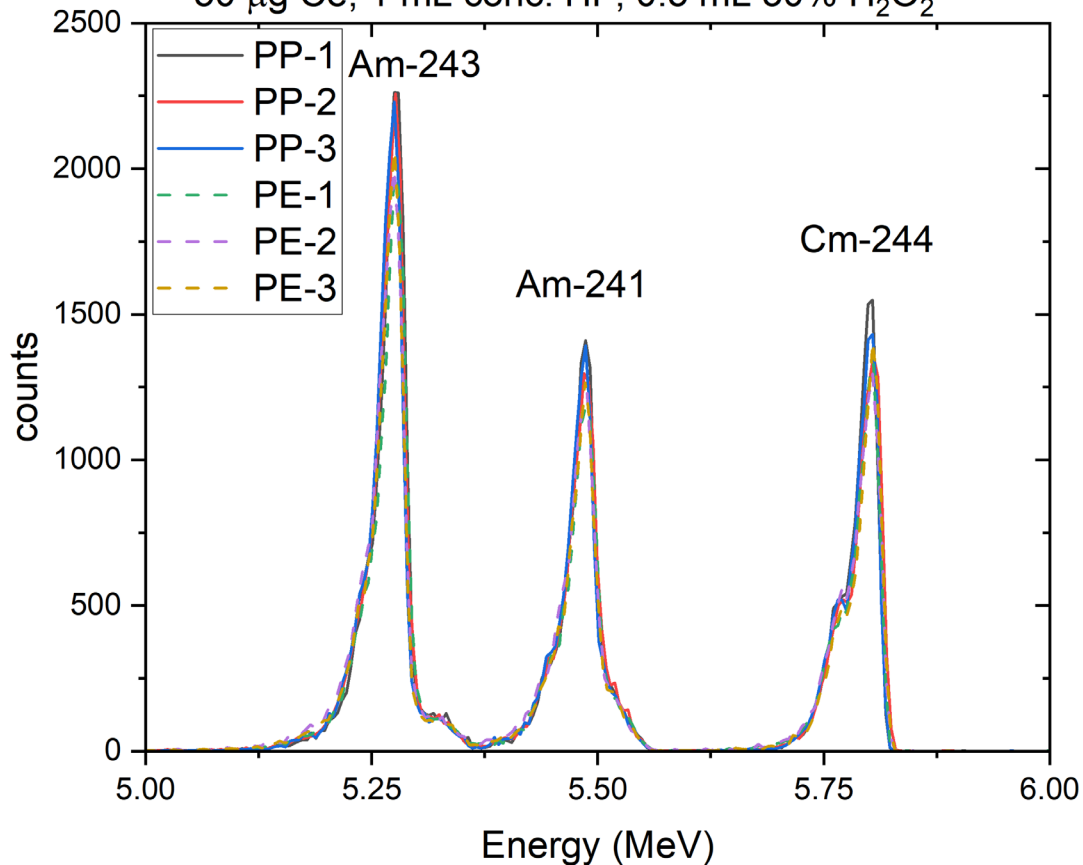
	Np-237	Pu-239
FWHM	+8.02%	+14.03%
Activity	-4.84%	-5.48%

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Standard Am/Cm Sample

Am-243, Am-241, and Cm-244 in 10 mL 1 M HCl

50 μg Ce, 1 mL conc. HF, 0.5 mL 30% H_2O_2



	Am-241	Am-243	Cm-244
FWHM	+1.64%	+1.80%	+1.57%
Activity	-2.89%	-3.38%	-3.00%

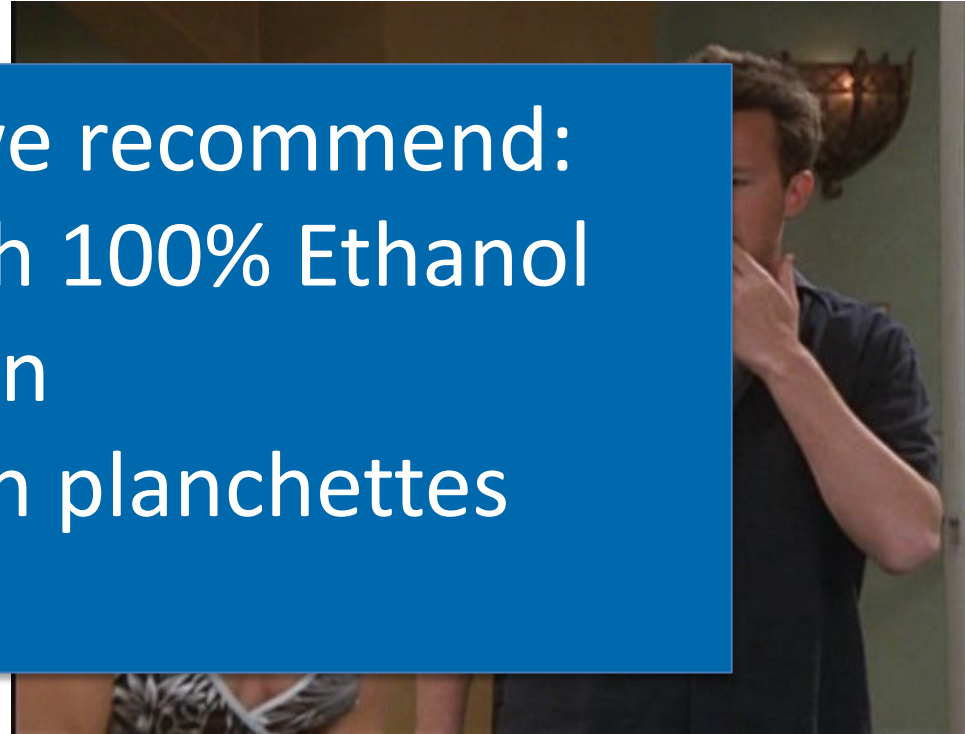
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Polyethylene Material

- Material looks and feels similar to polypropylene
- Uniform performance
- Standard FWH data
- Complete earth

To avoid curling we recommend:
1) Rinse filters with 100% Ethanol after preparation
2) Mount Filters on planchettes before drying

CONCEPT
conditions the filter may curl up when dried



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General Procedure

1. Prepare sample microprecipitate
 - Add Ln carrier
 - Add conc. HF
 - Add oxidizing or reducing agent (if necessary)
 - Mix and wait 30 min for precipitate to fully form
2. Wet Resolve® Filters with 80% Ethanol
3. Wet Resolve® Filters water
4. Add samples to filter/funnel
5. Rinse filter/funnel with water
6. Finish Resolve® Filters with 100% ethanol
7. Mount Filters on planchettes with adhesive/glue
8. Dry under heat lamp

General Procedure

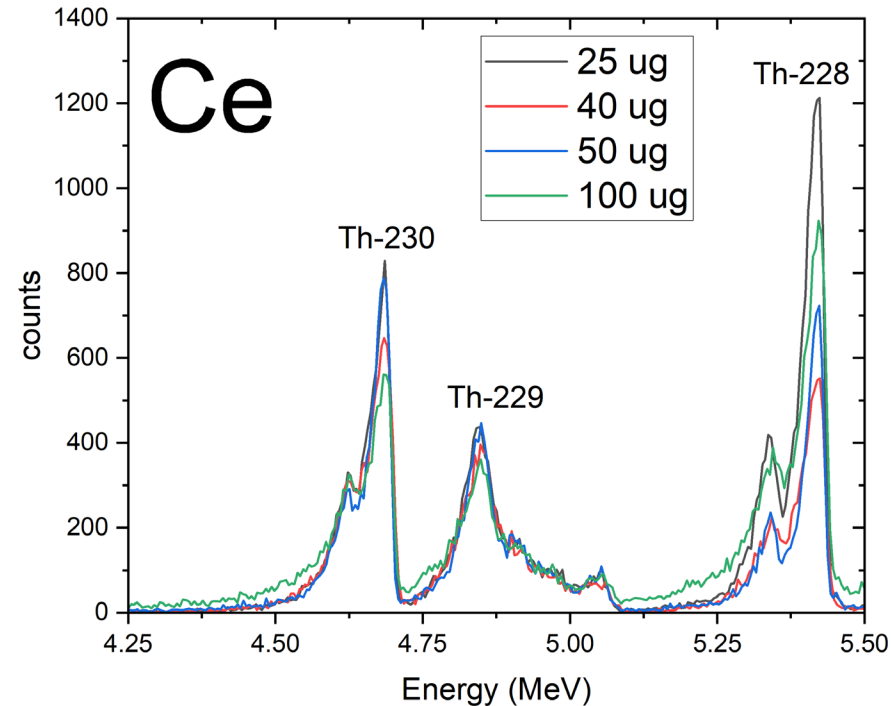
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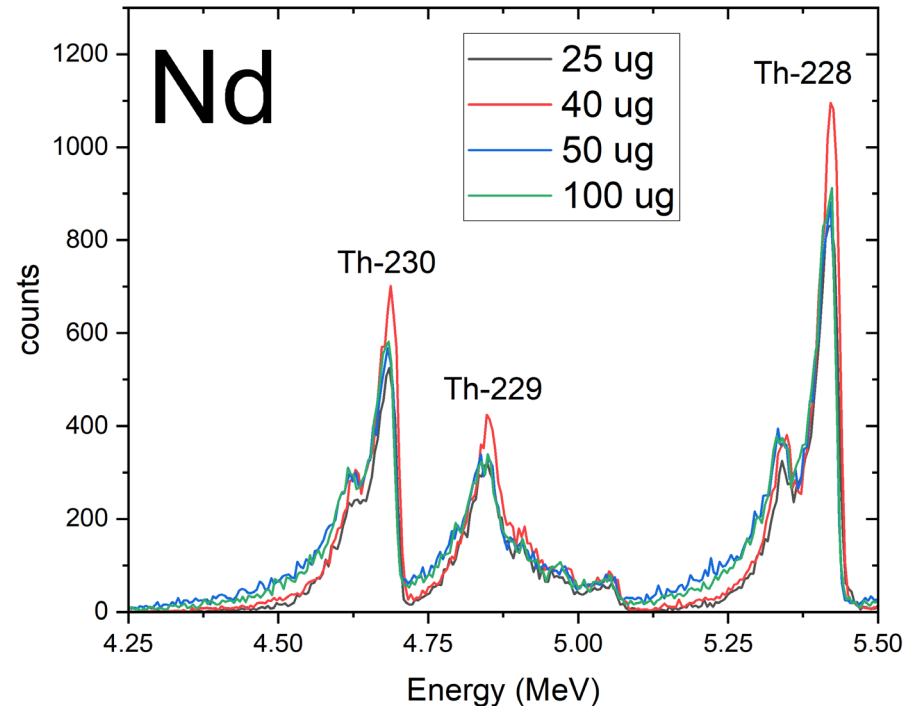
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Sample Prep – Ln Carrier

For Th samples in 9 M HCl the standard procedure is to add **40 μg** rare earth carrier to form the precipitate. But...



Slight peak broadening at 100 μg



Little difference between Ce/Nd

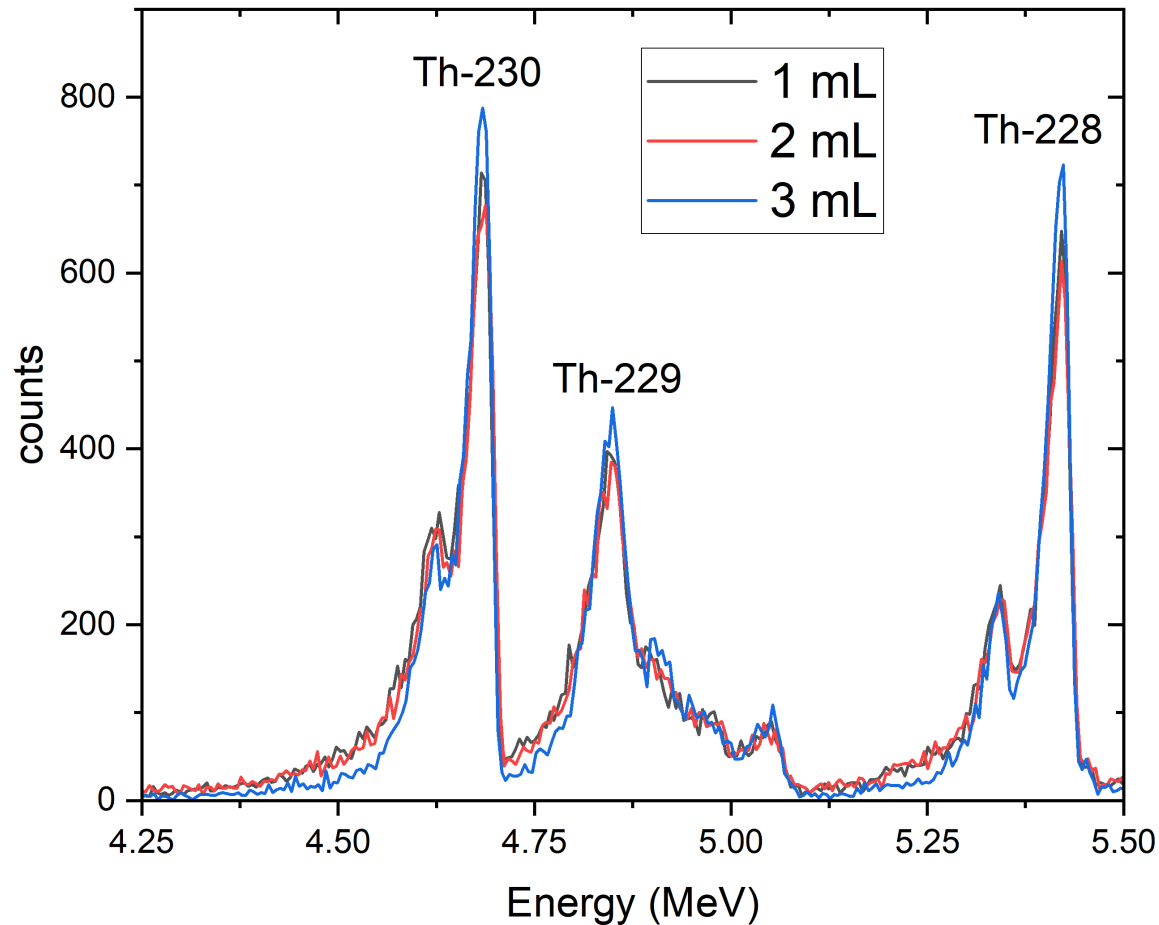


Sample Prep - HF

For Th samples in 9 M HCl the standard procedure is to add **3 mL** conc HF to form the precipitate. But...

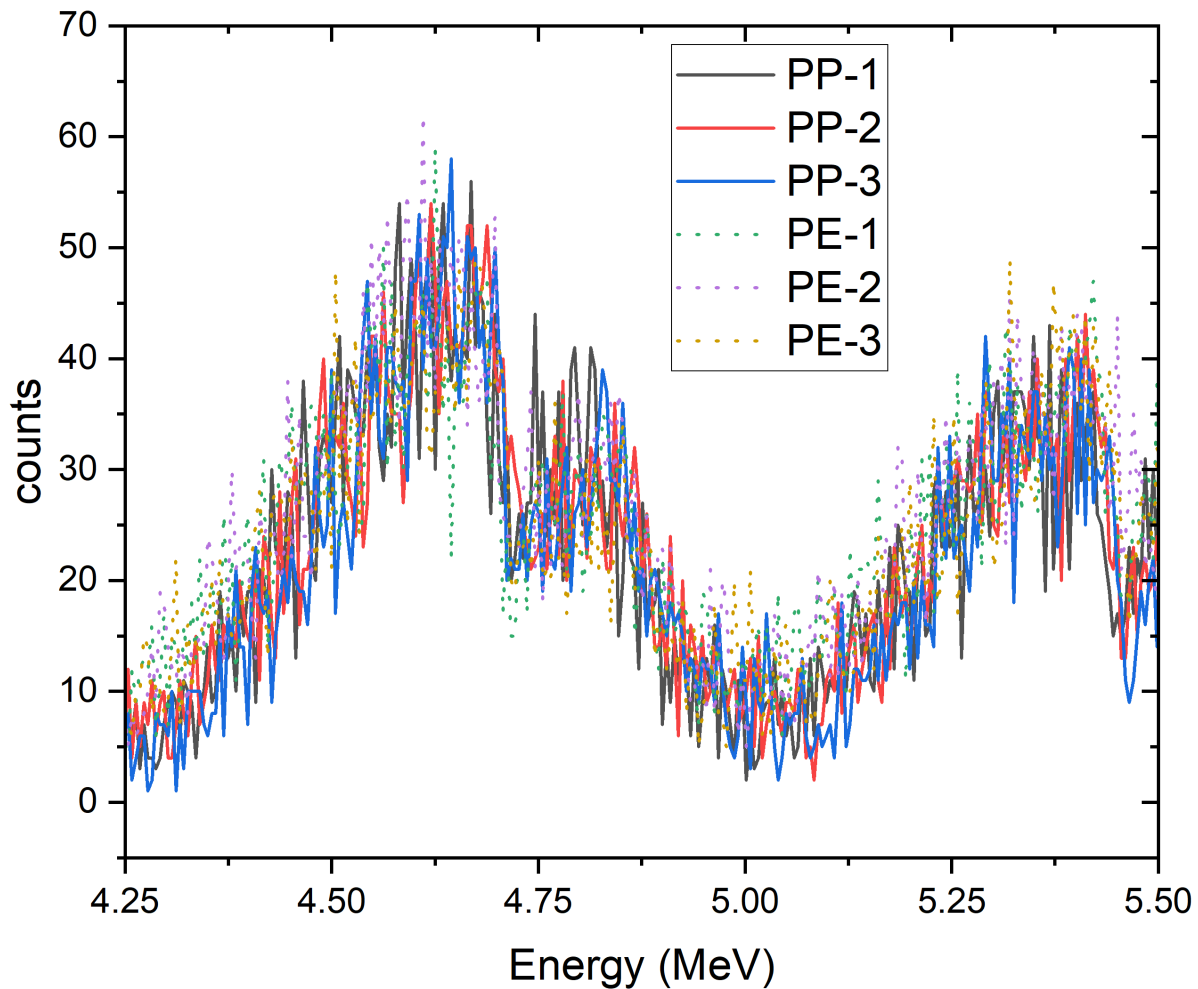
Between 1-3 mL HF
does not really matter

No significant loss of
activity observed



Sample Prep – Redox Agent

- TiCl_3
-
- H_2O_2
- Am/1
-



ipitation

Pu/Np or

interferes



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General Procedure

1. Prepare sample microprobe

- Add Ln carrier
- Add conc. HF
- Add oxidizing
- Mix and

2. Wet

3. We

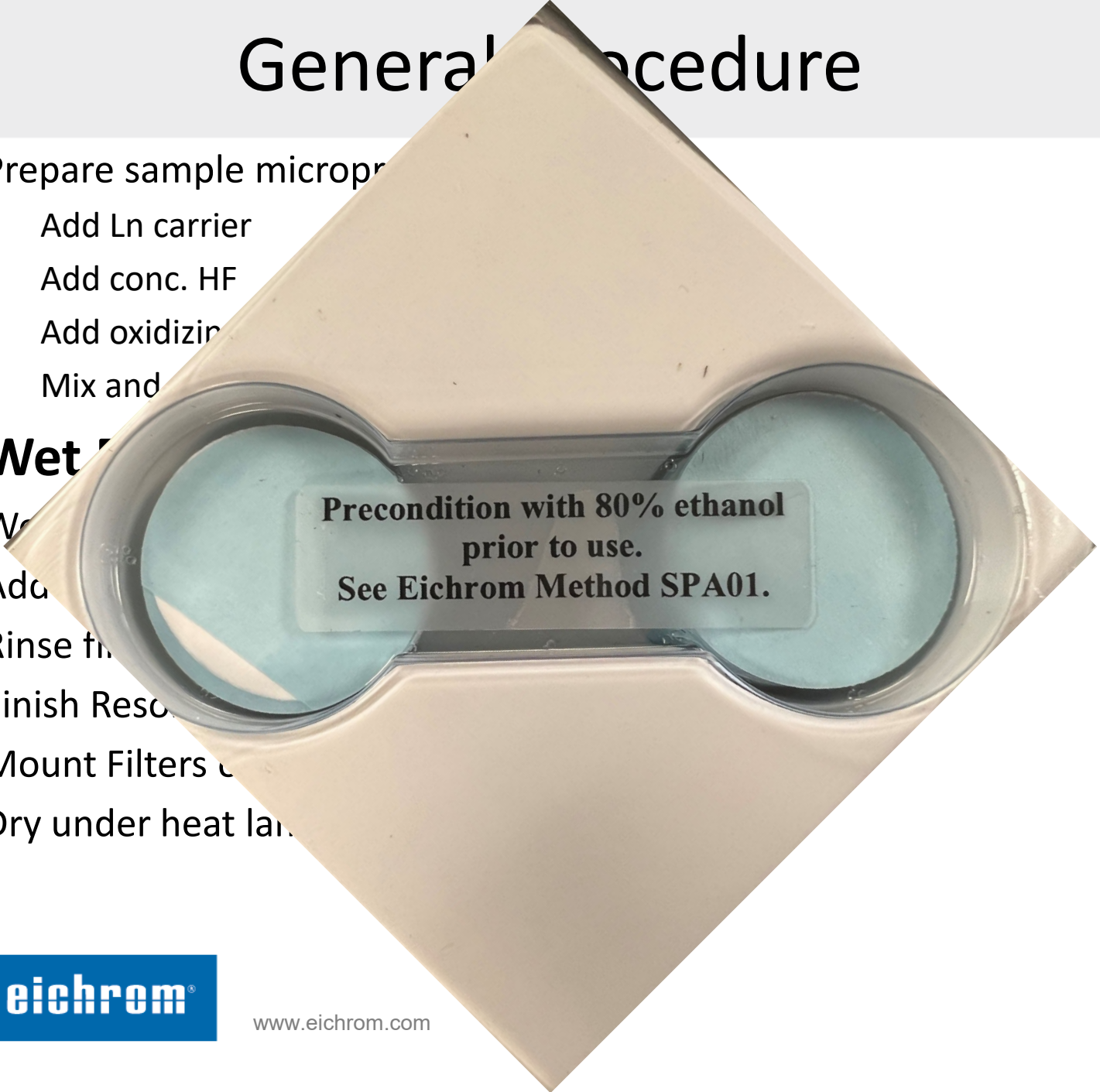
4. Add

5. Rinse f

6. Finish Reso

7. Mount Filters c

8. Dry under heat lab



**Precondition with 80% ethanol
prior to use.
See Eichrom Method SPA01.**

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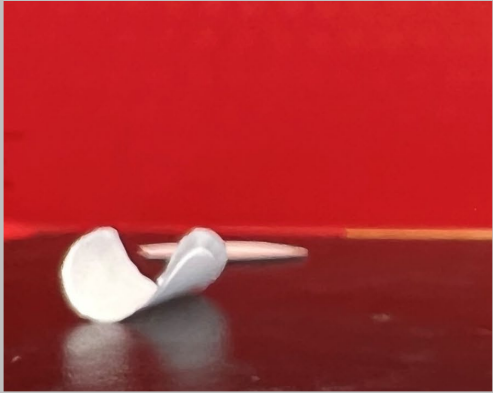
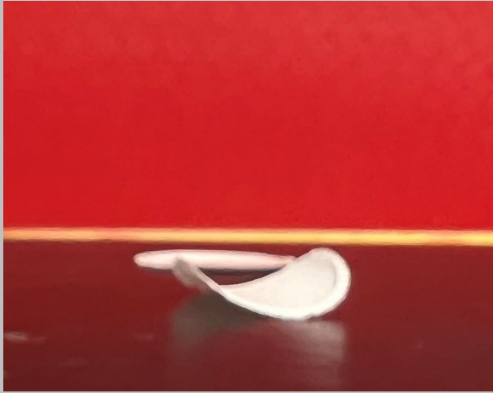

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Finishing with 100% Ethanol

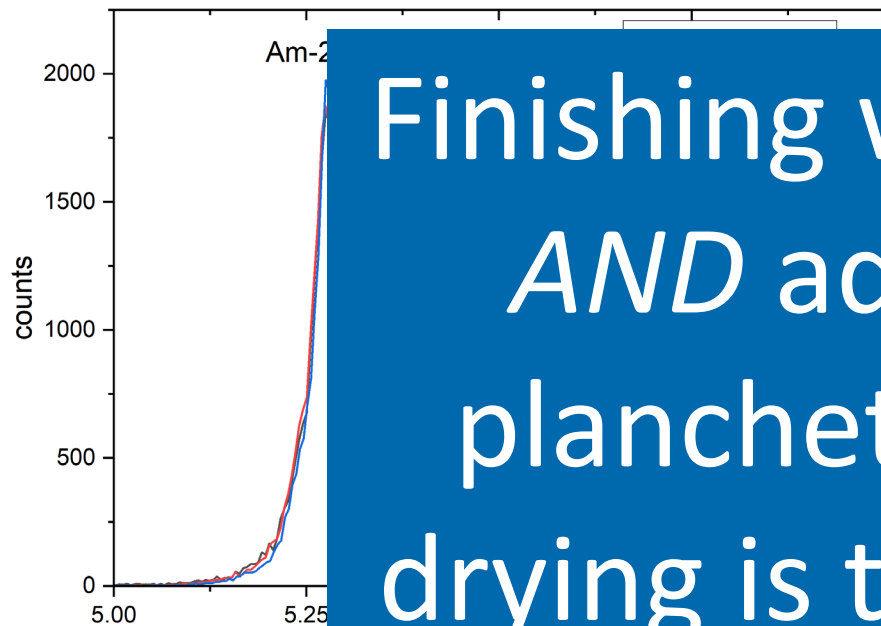
	Water	80% Ethanol	100% Ethanol
Polypropylene	 A white circular plate is shown on a dark, textured surface. The surface is wet with water, and the plate is clean and bright.	 A white circular plate is shown on a dark, textured surface. The surface is wet with 80% ethanol, and the plate is clean and bright.	 A white circular plate is shown on a dark, textured surface. The surface is wet with 100% ethanol, and the plate is clean and bright.
Polyethylene	 A white, curved piece of polyethylene is shown on a dark surface against a red background. The surface is wet with water, and the piece is clean and bright.	 A white, curved piece of polyethylene is shown on a dark surface against a red background. The surface is wet with 80% ethanol, and the piece is clean and bright.	 A white, curved piece of polyethylene is shown on a dark surface against a red background. The surface is wet with 100% ethanol, and the piece is clean and bright.



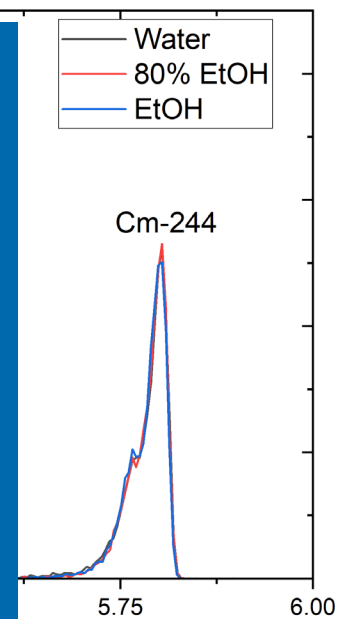
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Wet vs. Dry Filter Mounting

Filter Mounted First



Filter Dried First



Finishing with ethanol
AND adhering to
planchettes before
drying is the best way
to guarantee success

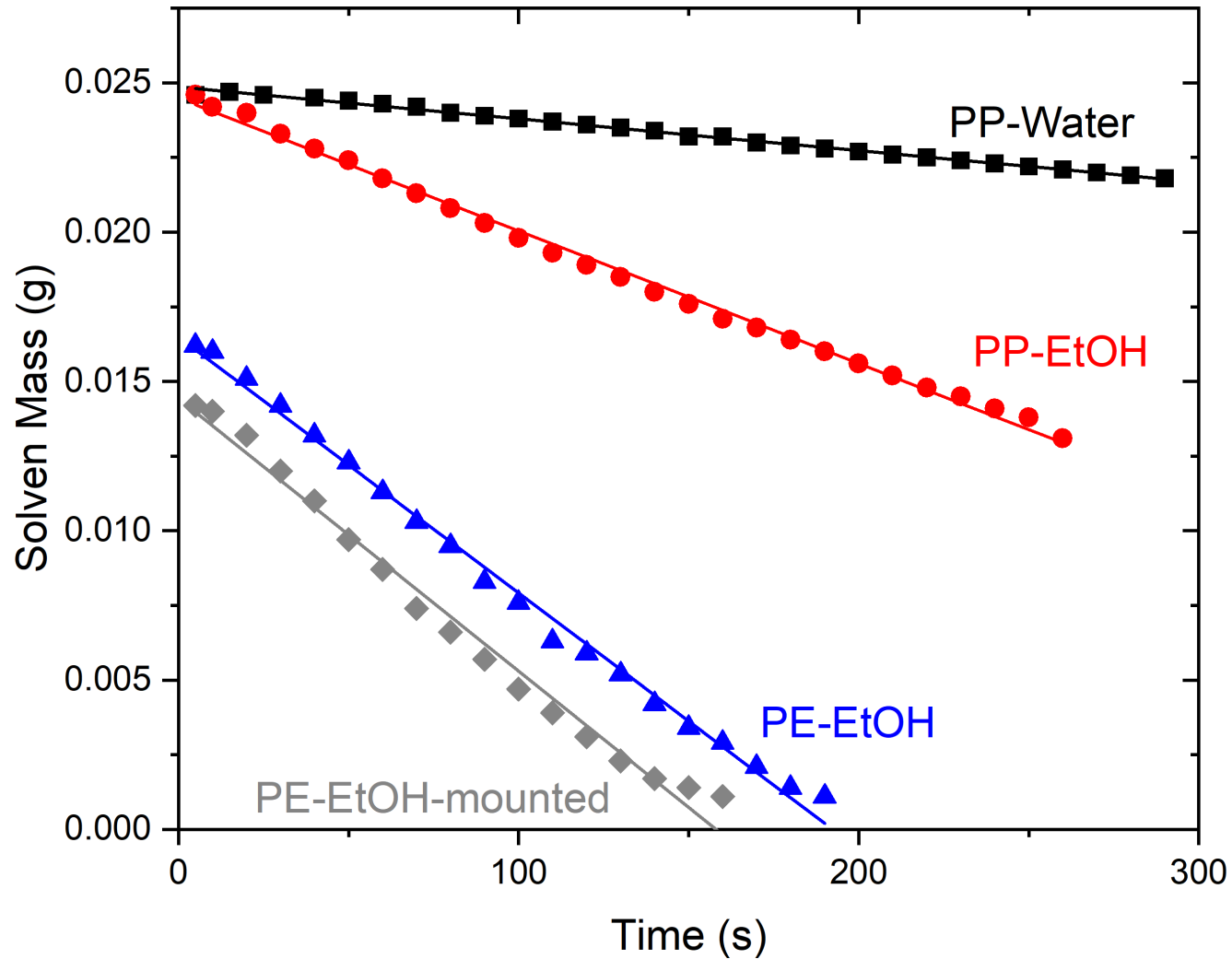
Spectral resolution
not significantly different for different
finishing conditions

if finished with
water were OK

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Filter Drying Time



Conclusions

1. 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

2. 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

Future Work

Preventing filter curling

- More in-depth study of drying time for different solvent finishing conditions

HF alternatives

- Hydroxide methods
 - Actinide samples under common elution conditions
 - More in-depth testing of complexant interferences
- Other F⁻ sources
 - Validate conditions using NH₄F and NH₄FHF instead of HF for common actinide samples

Questions?

For Eichrom Application Notes please visit:

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

AN-1805 for Rare Earth Fluoride Method

AN-1807 for Cerium Hydroxide Method

For Alpha Spectrometry Training please visit:

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

The logo for Eichrom, featuring the word "eichrom" in a white, lowercase, sans-serif font with a registered trademark symbol (®) to its upper right. The text is set against a dark blue rectangular background.

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